

Mechanical Properties, Water-Swelling Behavior, and Morphology of Water-Swellable Rubber Prepared Using Crosslinked Sodium Polyacrylate

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ABSTRACT: A novel water-swellable rubber (WSR) has been prepared by blending chlorobutadiene rubber (CR), reactive clay and other additives with crosslinked sodium polyacrylate (CSP), which was modified by interpenetrating polymer networks (IPNs) technology with crosslinked P(AA-co-BA). The structure of WSR was characterized by scanning electron microscopy (SEM). The mechanical properties, water-swelling ratio by mass, and the percentage loss of CSP in the WSR were investigated. The results showed that the modified CSP grains can be dispersed well in the CR, and that it resulted in increase of mechanical properties and water-swelling ratio and in decrease of percentage loss of CSP, compared with the unmodified one. When the per-

centage content of crosslinked P(AA-co-BA) used to modify CSP reached 30%, the tensile strength, elongation at break, and water-swelling ratio of WSR exhibited maximum value, and percentage loss of CSP exhibited minimum value. When the content of CSP in WSR was 30 phr, the tensile strength, elongation at break, and water-swelling ratio and percentage loss of CSP of the WSR containing CSP modified were 7.7 MPa, 1530, 438, and 2.5%, respectively. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1489–1496, 2006

Key words: crosslinked sodium polyacrylate; compatibilization; chlorobutadiene rubber; water-swellable rubber

INTRODUCTION

Usually a high interfacial tension between the phases of an immiscible polymer blend leads to coarse and unstable morphologies. In addition, a high interfacial tension results in a narrow interface, poor physical and chemical interactions across the phase boundaries, and as a consequence, in a poor adhesion between the phases. For blend systems, the mechanical behavior depends critically on two demanding structural parameters, a proper interfacial tension leading to a phase size small enough to allow the material to be considered as macroscopically homogeneous, and an interface adhesion strong enough to assimilate stresses and strains without disruption for the established morphology. This can be achieved by different ways.^{1,2} One way is to modify one of the phases so as to increase the interfacial area, so that the components will be able to transfer the applied stress effectively, despite having high interfacial tension. Techniques of blending have been used successfully in some blend systems to form interpenetrating cocontinuous net-

work phases. Such systems have improved mechanical properties because of the interpenetrating polymer networks (IPNs), which allows direct load sharing between the components without the need for stress transfer across the phase boundaries.^{3,4}

It is now well established that the phase morphology of immiscible polymer blends can be controlled by IPNs, which act as interfacial agents. The modified polymer added to an immiscible binary blend should (i) reduce the interfacial energy between the phases, (ii) permit finer dispersion during blending, and (iii) result in improved interfacial adhesion. Several experimental investigations have been reported on the compatibilizing action of added block and graft copolymers in heterogeneous polymer blends.

In recent years, more attention has been focused on compatibilization of immiscible polymer blends modified by IPNs technology.^{5–8} Polymer modified by IPNs technology, as compatibilizers reduced the interfacial tension and increased the adhesion between the phases, thereby presented a finer dispersion and a more stable morphology.

As a novel functional material, water-swellable rubber (WSR) can stop water not only by means of elastic sealing but also by means of swelling in water. The general methods used to prepare WSR are mechanical mixing.^{9–16} Hydrophilic super water-absorbent resin cannot disperse well in hydrophobic rubber, so that it

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can break off from rubber network easily. This will weaken water-swelling abilities, mechanical properties, long-term water retention, and repeated usage. These problems of polymer–polymer incompatibility can often be mitigated by modification of one phase by IPNs technology. The compatibility of hydrophilic dispersion phase and hydrophobic continuous phase is expected to be improved greatly, and the loss of super water-absorbent resin from the rubber matrix to be reduced. In such a way, the water-swelling properties may be enhanced, but work using this method has seldom been reported.

The rubbers used are often conventional rubbers or their copolymers. Compared with other general rubber, however, chlorobutadiene rubber has better elasticity, crystallizability, and cohesion. Moreover, it is a synthetic rubber with good oil resistance, solvent resistance, heated aging resistance, and fired resistance properties. Crosslinked sodium polyacrylate (CSP) used as a super water-absorbent resin has high water-retention abilities, stable structure, and a low price. It does not decompose and denaturalize below 300°C.

In this research, chlorobutadiene rubber and CSP were chosen as chief materials and the technique of IPNs was used to modify CSP. Modified CSP was obtained first by swelling CSP network with acrylic acid, and then it was copolymerized with butyl acrylate. The effects of the content of modified polymer on mechanical properties and water-swelling behavior were investigated. The effect of CSP content on water-swelling behavior was studied. The morphological structure of the fractured surface of WSR was characterized by scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

Chlorobutadiene rubber (M_w , 2.0×10^5) was obtained from Changshou Synthetic Rubber Factory, Sichuan Province, China. Crosslinked sodium polyacrylate (CSP) was prepared by our laboratory (water-swelling ratio, 400 g/g). Acrylic acid, butyl acrylate, benzoyl peroxide and dimethylaniline were obtained from Bodi, Tianjin, China. Potassium persulfite and a crosslinking agent (*N,N'*-methylene-bisacrylamide) were obtained from Shanghai Chemical Reagent Factory, China. Reactive clay (particle size, below 200 mesh) was obtained from Xiangfan Chemical, Hubei province, China. Zinc oxide, and magnesium oxide were all chemical grade.

Preparation of CSP modified with P(AA-co-BA)

In this experiment, an amount of CSP was swelled by acrylic acid and an amount of crosslinking agent, which was dissolved completely in acrylic acid at

ambient temperature. After 30 min, butyl acrylate (the compositions of butyl acrylate and acrylic acid were 85:15) and benzoyl peroxide and dimethylaniline initiators (1.5% by mass to monomer, respectively) were added. The mixture was dispersed well under stirring, and then the reaction was carried out at 30°C for 4 h. The product was dried in a vacuum oven at 60°C.

Preparation of WSR

At room temperature, chlorobutadiene rubber was masticated on an open mill (the 160 mm of Φ) for 2 min and then modified or unmodified CSP, stearic acid, zinc oxide, NA-22, reactive clay, and magnesium oxide were fed into the mill in proper order, and mixed continuously until the mixture reached apparent homogeneity. After blending, the batch was laid up on a dry and clean place at room temperature for more than 2 h and then remilled and passed through the open mill seven times. A mixed compound with better dispersion was obtained.

The mixed compound was put in an $160 \times 120 \times 2$ mm³ mold, preheated for 3 min, and cured for 15 min at 150°C in a 16-ton press for vulcanization. After that, the mold was taken out and cooled to room temperature.

Characterization

IR spectra of the pure CSP and the modified CSP coated on KBr pellet were recorded on a Nicolet Magna-IR750 FTIR spectrometer.

The accurately weighed CSP (about 1 g) was immersed in 800 g of distilled water and allowed to soak for 1 day. The swollen gel was then separated from unabsorbed water by screening through a 300-mesh sieve, and the sieve was then weighed to determine the weight of the water-swollen gel. The water-swelling ratio (Q) was calculated using the following equation:

$$Q = (W_2 - W_1) / W_1 \quad (1)$$

where W_2 and W_1 are the weights of the water-swollen gel and the dry resin, respectively. Q was calculated as grams of water per gram of resin.

Tensile strength and elongation at break testing of dumbbell-shaped specimens was performed by using a WDW-20 electronic universal tester (Shenzhen Kaiqiangli Testing Instruments) at room temperature at crosshead speeds of 500 mm/min.

The dried samples were cut about 3 g, weighted accurately, and soaked in distilled water or solution with different pH value at room temperature. At regular intervals, the swollen sample was removed from the distilled water or different pH-values bath; superficial moisture was removed; the weight of the sample

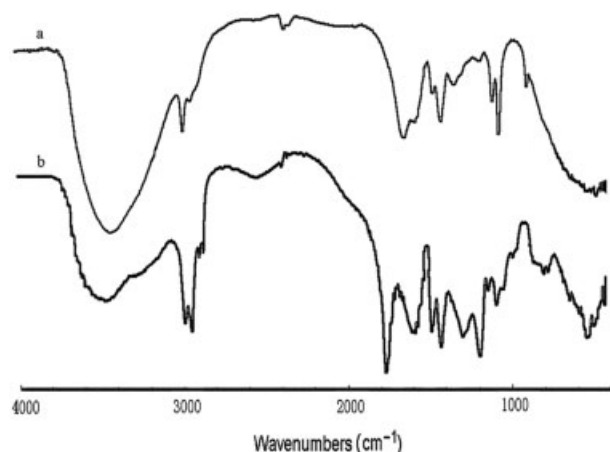


Figure 1 FTIR spectra of CSP (a) and CSP modified with P(AA-co-BA) (b).

was measured immediately, and the sample was placed in the same bath. After the swelling test, the samples were dried at 50°C until a constant weight was reached. The first water-swelling ratio by mass and the first percentage loss by mass were calculated as follows:

$$\text{Water-swelling ratio} = \frac{W_4 - W_3}{W_3} \times 100\% \quad (2)$$

$$\text{Percentage loss} = \frac{W_3 - W_5}{W_3} \times 100\% \quad (3)$$

where W_4 and W_3 were the weights of a sample swelled and unswelled with water, respectively; W_5 was the mass of the dried sample, which had been swelled with water.

The secondary water-swelling ratio was measured by the same method used for the first one, by using the dried sample, which was swelled with water in the measurement similar to the first water-swelling ratio by mass.

Scanning electron microscopy of the WSR was performed on a HitachiS-530 scanning electron microscope. Samples were first cryogenically fractured in liquid nitrogen, mounted on a sample holder, gold-sputtered, and finally observed under the microscope.

RESULTS AND DISCUSSION

FTIR spectra of CSP modified and unmodified

The FTIR spectra of CSP [Fig. 1(a)] and CSP modified with P(AA-co-BA) [Fig. 1(b)], which is purified by IPN technology are given in Figure 1. The characteristic bands of C=O group at 1730 cm^{-1} in the FTIR spectra of modified CSP confirmed that there was a PBA chain in the modified CSP.

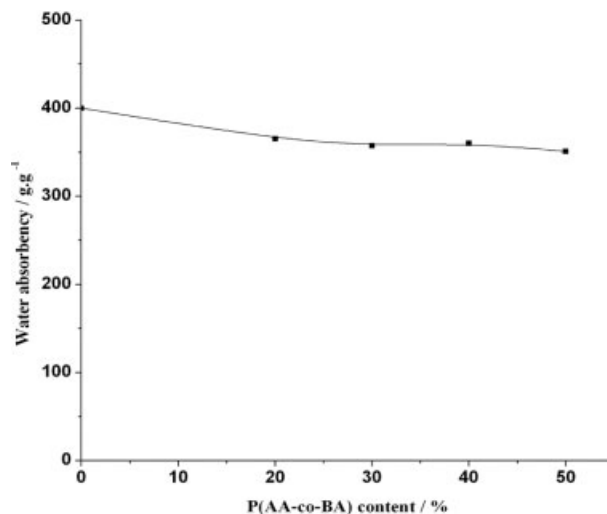


Figure 2 Effect of crosslinked P(AA-co-BA) content on water-swelling ratio of CSP.

Effect of crosslinked P(AA-co-BA) content on water-swelling ratio of CSP

Effect of crosslinked P(AA-co-BA) content on water-swelling ratio of CSP is shown in Figure 2. Compared with that of unmodified CSP, water-swelling ratio of CSP modified by crosslinked P(AA-co-BA) decreased slightly. This indicated that the effect of the introduction of crosslinked P(AA-co-BA) on the water-swelling ratio was smaller.

Effect of crosslinked P(AA-co-BA) content used to modify CSP on mechanical properties of WSR

Effect of crosslinked P(AA-co-BA) content used to modify CSP on the mechanical properties of WSR is shown in Table I. From Table I, we can see that the tensile strength and elongation at break of WSR, unswelled and swelled with water, changed with crosslinked P(AA-co-BA) content. The maximum values occurred when mass ratio of P(AA-co-BA) and

TABLE I
Effect of Crosslinked P(AA-co-BA) Content Used to Modify CSP on Mechanical Properties of Water-Swellable Rubber

P(AA-co-BA)/CSP (wt %)	Tensile strength ^a (MPa)	Elongation at break ^a (%)	Tensile strength ^b (MPa)	Elongation at break ^b (%)
0/100	6.5	1320	1.1	1450
20/100	7.4	1520	2.2	1620
30/100	7.7	1530	2.8	1730
40/100	6.2	1410	2.5	1590
50/100	6.1	1170	2.4	1250

CR, 100 phr; CSP, 30 phr; reactive clay, 30 phr.

^a WSR unswelled with water.

^b WSR swelled with water.

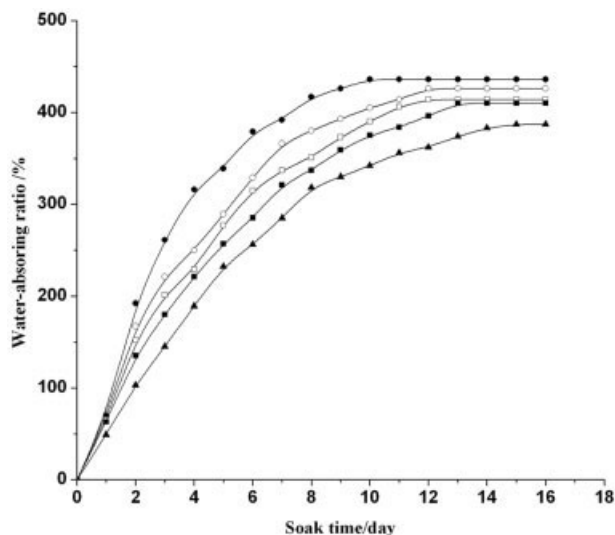


Figure 3 Effect of soak time on water-swelling ratio of water-swelling rubber in which the CSP was modified with different contents of P(AA-co-BA). P(AA-co-BA) contents used to modify CSP were 0%, \blacktriangle , 20%, \circ , 30%, \bullet , 40%, \square , and 50%, \blacksquare .

CSP was 30%. And they increased with an increase in P(AA-co-BA) content, when P(AA-co-BA) content is less than 30%. But when crosslinked P(AA-co-BA) content is more than 30% they decreased. And the tensile strength of WSR unswelled with water was better than that of WSR swelled with water, whereas elongation at break of the former was lesser than the latter.

Hydrophilic super water-absorbent resin cannot disperse well in hydrophobic rubber, so that it results in the decrease of mechanical properties. The compatibility of hydrophilic dispersion phase and hydrophobic continuous phase is expected to be improved greatly, when hydrophilic CSP was modified by interpenetrating polymer networks (IPNs) technology with hydrophobic P(AA-co-BA). CSP dispersed well in the chlorobutadiene rubber (CR), and resulted in the increase of tensile strength and elongation at break of WSR, unswelled and swelled with water; but when the P(AA-co-BA) content used for modifying CSP was too much, the tensile strength and elongation at break of WSR, unswelled and swelled with water, decreased and that could be because the mechanical properties of P(AA-co-BA) was smaller than CR. Its tensile strength decreased and its elongation at break increased because of the plastication of water, when WSR was swelled with water.

Effect of crosslinked P(AA-co-BA) content used to modify CSP on water-swelling ratio of WSR

Figure 3 shows the effect of soak time on the water-swelling ratio of WSR in which the CSP was modified with different contents of P(AA-co-BA). From Figure 3,

it can be seen that the water-swelling ratio of WSR increased with the soak time increase, but the speed of water-absorption for the WSR in which the CSP was modified with different contents of P(AA-co-BA) was different. There is a relationship between the amount of P(AA-co-BA) used for modification and the speed of water-absorption and water-swelling ratio in the equipment of WSR. The maximum speed of water-absorption and water-swelling ratio in the equilibrium of WSR occurred when mass ratio of P(AA-co-BA) and CSP was 30%. The speed of water-absorption and water-swelling ratio in the equilibrium of WSR increased with an increase of P(AA-co-BA) content, when the amount of P(AA-co-BA) used is less than 30%. But when the amount of P(AA-co-BA) used is more than 30%, they decrease with an increase in P(AA-co-BA) content.

Water-swelling ratio in the equilibrium and the speed of water-absorption of WSR are dependent on the amount and distribution of CSP in WSR. The water-swelling ratio in the equilibrium and the speed of water-absorption of WSR increase with the amount of the CSP, when the distribution of CSP in WSR is certain. In other words, when there was a certain amount of CSP in the WSR, water-absorbent resin dispersed well in rubber and also results in the increase of the water-swelling ratio in the equilibrium and the speed of water-absorption of WSR. Figure 3 shows that when hydrophilic super CSP was modified by IPNs technology with P(AA-co-BA) up to 30%, the CSP dispersed well in the CR, and resulted in maximum water-swelling ratio in the equilibrium and the speed of water-absorption of WSR. When the P(AA-co-BA) content used for modifying CSP was too much, the water-swelling ratio in the equilibrium and the speed of water-absorption of WSR decreased because of the hindrance of PBA shell, which hinders water from a thin layer of continuous paste passing well through and reaching the CSP.

To investigate secondary water-swelling properties of WSR, the WSR, which has absorbed a large amount of water and reached water-absorbing equilibrium, was dried and then put into the water, and the changes of water-swelling ratio with soak time were recorded in Figure 4. From Figure 4, it can be seen that the water-swelling ratio of WSR increased also with the soak time increase. It is similar to Figure 3. The maximum speed of water-swelling and water-swelling ratio in the equilibrium of WSR occurred also when mass ratio of P(AA-co-BA) and CSP was 30%. The change of the speed of water-swelling and water-swelling ratio in the equilibrium of WSR with an increase of P(AA-co-BA) content were also similar to Figure 3; but when compared with Figure 3, the speed of water-swelling and water-swelling ratio in the equilibrium of WSR, which was swelled with water and then dried, decreased. And the change of first and

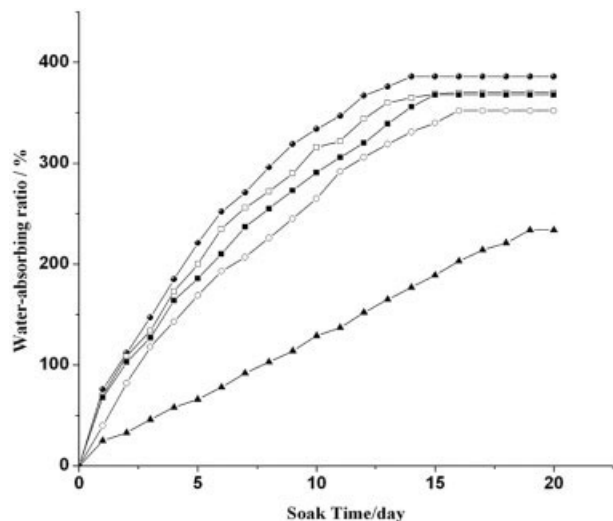


Figure 4 Effect of soak time on the secondary water-swelling ratio of water-swellaible rubber in which the CSP was modified with different contents of P(AA-co-BA). P(AA-co-BA) contents used to modify CSP were 0% ▲, 20%, ○, 30%, ●, 40%, □, and 50%, ■. The other materials content were CR, 100 phr, CSP, 30 phr, and reactive clay, 30 phr.

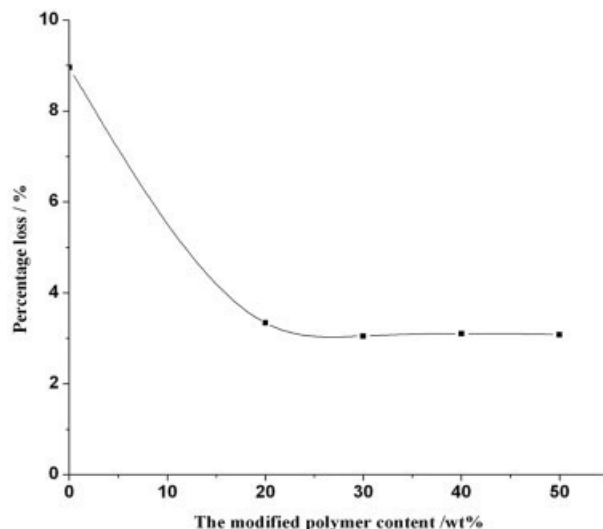


Figure 5 Effect of P(AA-co-BA) content, used to modify CSP, on percentage loss by mass of CSP in water-swellaible rubber; CR, 100 phr; CSP, 30 phr; reactive clay, 30 phr.

secondary water-swelling ratio in the equilibrium of WSR and their difference with P(AA-co-BA) content are shown in Table II. From Table II, it can be seen that the difference of first and secondary water-swelling ratio in the equilibrium exhibited minimum value, when mass ratio of P(AA-co-BA) and water absorbent resin was 30%, and it was only 52%; but it was 153% when CSP was not modified. In WSR consisting of unmodified CSP, the loss of CSP was the main reason for decrease of the secondary water-swelling ratio in the equilibrium. But in the WSR consisting of CSP modified with 50% P(AA-co-BA) for CSP, decrease of the secondary water-swelling ratio in the equilibrium was due to the little decrease of CSP.

Effect of P(AA-co-BA) content used to modify CSP on percentage loss by mass of CSP in WSR

Effect of P(AA-co-BA) content, used to modify water absorbent resin, on percentage loss by mass of water absorbent resin in WSR is shown in Figure 5. From this figure, it can be seen that in WSR, consisting of unmodified CSP, loss of CSP reached to 8.96 wt %; but in WSR, consisting of modified CSP, loss of CSP decreased obviously, and it reached 3.05 wt %. This is because the modified polymer is located at CSP interface and a thin shell of polymer is formed around the dispersed phase. This leads to a broad, stable, and less mobile interface that can resist coalescence. CSP grains could not be dropped out of crosslinked network as a whole from CR.

TABLE II
Effect of Crosslinked P(AA-co-BA) Content Used to Modify CSP on Water-Swelling Ratio at the Equilibrium of Water-Swellaible Rubber

CSP/crosslinked P(AA-co-BA) (wt %)	First water-swelling ratio at the equilibrium (wt %)	Second water-swelling ratio at the equilibrium (wt %)	Δ (wt %)
100/0	387	234	153
100/20	436	352	84
100/30	438	386	52
100/40	414	370	45
100/50	410	368	42

CR, 100 phr; CSP, 30 phr; reactive clay, 30 phr. Δ is the difference of first and secondary water-swelling ratio at the equilibrium.

Effect of pH value on water-swelling ratio by mass

Figure 6 shows the effect of value of pH on water-swelling ratio by mass of WSR. From Figure 6, it is can be seen that when pH value was less than 7, the water-swelling ratio by mass increased with its increase. And when the pH value was equal to 7, it reached the maximum value. But when the pH value further increased water-swelling ratio by mass decreased. When the pH value was less than 7, the carboxylate in water absorbing resin becomes carboxylic acid as it reacts with acid; ionization of carboxylic acid was more weak than that of carboxylate in water solution; and the water-swelling ability of water absorbing resin depends on the amount of carboxylate ion in CSP net, and repulsion between chains in water-absorbing resin decreased with the decreased carboxylate ion amount of chains. When the pH value was

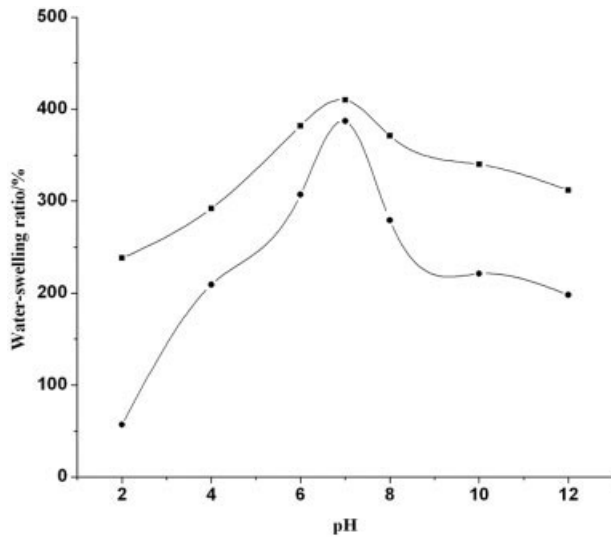


Figure 6 The effect of value of pH on water-swelling ratio by mass of water-swollable rubber. CR, 100 phr; reactive clay, 30 phr; CSP, 30 phr; CSP/P(AA-co-BA): ●, 0 wt%; ■, 30 wt%.

more than 7, the cation formed from the alkali ionization in the water solution hindered the ionization of carboxylate.

From Figure 6, it can be seen that the water-swelling ratio of WSR containing modified CSP was more than that containing unmodified CSP in different pH value, and their difference increased with decrease ($\text{pH} < 7$) or increase ($\text{pH} > 7$) of pH value in the system. When hydrophilic CSP was unmodified, the CSP grains could not disperse well in the CR. As a result, some CSP grains in WSR were not used well to absorb water and swell the rubber, because the resin grains were apart from each other. The smaller (or larger) the pH value in water solution, the smaller the water-swelling ratio of the resin grains. When the water-swelling ratio of the resin grains is less, the movement of water from one water-absorbent resin grain to the other is difficult, because they are apart from each other.

Effect of the amount of CSP on water-swelling ratio of WSR

Figure 7 shows the effect of the amount of modified CSP on the water-swelling ratio of WSR. From this figure, it can be seen, for all WSR, that the water-swelling ratio increased with the increase of soaking time, and that it stopped to increase after reaching the maximum value; but the speed of water-swelling increased with the increase of CSP amount. That is because, in the WSR, the distance between CSP grains decreased with the increase of CSP amount; and the time, which was needed for the water to be carried from one CSP grain to the other, decreased with decrease of distance between the grains.

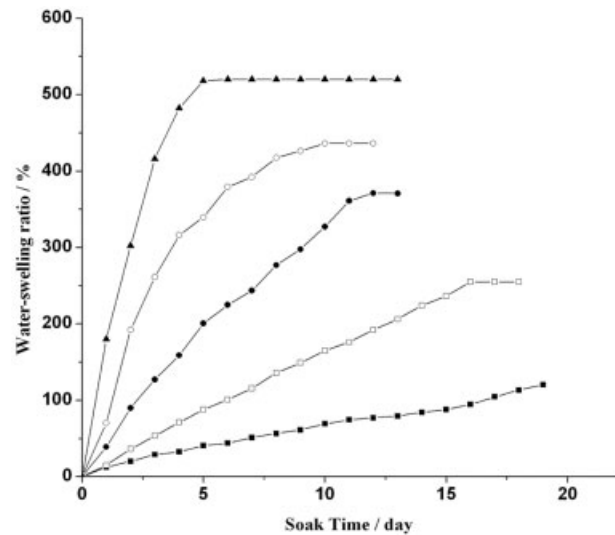


Figure 7 Effect of the amount of CSP modified on water-swelling ratio of water-swollable rubber; CSP content: 15 phr, ■; 20 phr, □; 25 phr, ●; 30 phr, ○; 35 phr, ▲. The other materials content were CR, 100 phr, reactive clay, 30 phr, and CSP/P(AA-co-BA), 30 wt %.

Figure 8 shows the effect of the amount of modified CSP on secondary water-swelling ratio of WSR. From this figure, it can be seen that, for all WSR, change of the secondary water-swelling ratio with the increase of soaking time is similar to that of the first shown in Figure 7. And the difference of first and secondary water-swelling ratio at the equilibrium, shown in Table III, was very small. Compared with the first water-swelling ratio, the secondary water-swelling ratio at

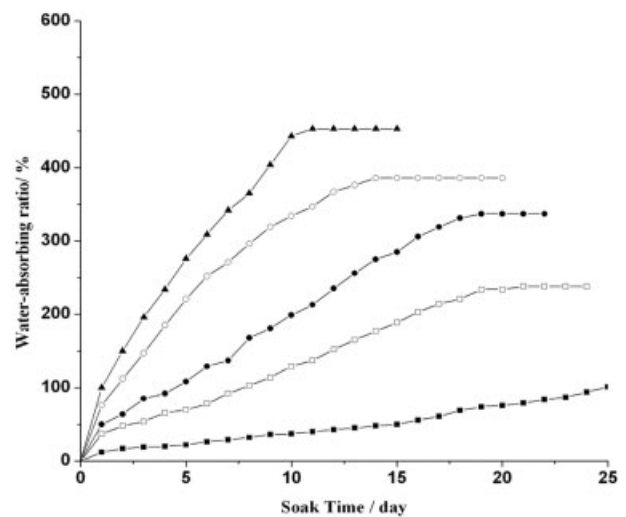


Figure 8 Effect of the amount of CSP on secondary water-swelling ratio of water-swollable rubber; CSP content: 15 phr, ■; 20 phr, □; 25 phr, ●; 30 phr, ○; 35 phr, ▲. The other materials content were CR, 100 phr, reactive clay, 30 phr, and CSP/P(AA-co-BA), 30 wt %.

TABLE III
Effect of the Amount of CSP Modified on Water-Swelling Ratio at the Equilibrium of Water-Swellable Rubber

The amount of CSP (phr)	First water-swelling ratio at the equilibrium (wt %)	Secondary water-swelling ratio at the equilibrium (wt %)	Δ (wt %)
15	120	101	19
20	254	238	16
25	370	337	33
30	438	386	52
35	520	453	67

CR, 100 phr; reactive clay, 30 phr; CSP/crosslinked P(AA-co-BA), 30 wt %. Δ is the difference of first and secondary water-swelling ratio at the equilibrium.

the equilibrium of the WSR declined slightly (Table III), which is due to the loss of CSP.

Effect of the amount of CSP modified on percentage loss by mass

Effect of the amount of the modified CSP on percentage loss by mass is shown in Figure 9. From this figure, it can be seen that the percentage loss by mass was less than 4% for different content of modified CSP. And the percentage loss by mass reached 2% and secondary water-swelling ratio at equilibrium reached 101%, when the CSP content was 15% (Table III). This result is due to the well-dispersed CSP in the CR. It could have something to do with the modification of water absorbent resin.

SEM photographs of the fracture surface for the WSR

Figure 10 shows the SEM photographs of the fracture surface for the WSR. Figure 10(a) shows that the frac-

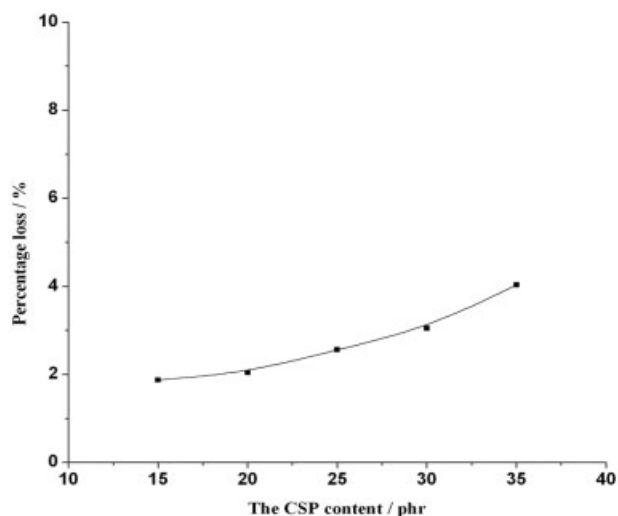
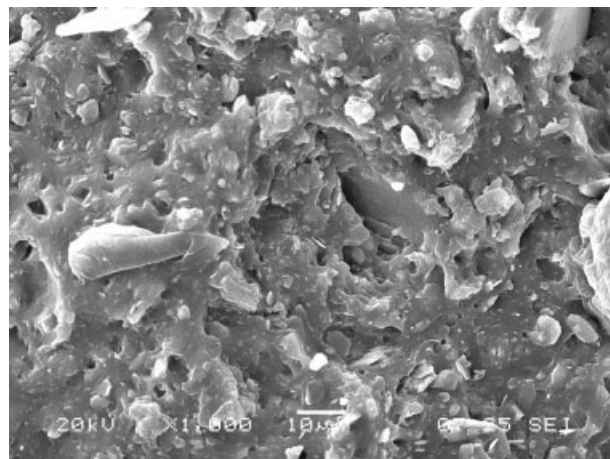
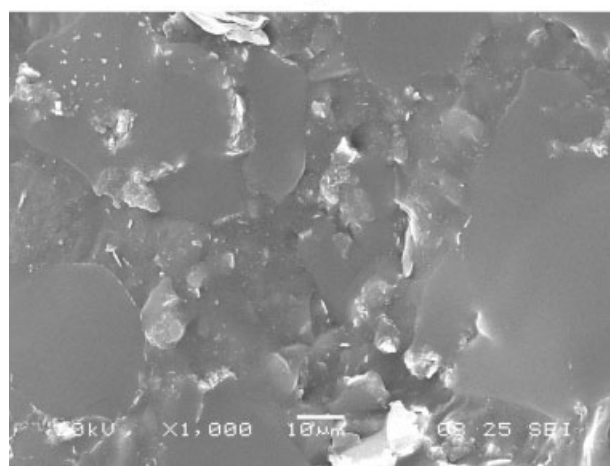


Figure 9 Effect of the amount of water absorbent resin modified on percentage loss by weight; CR, 100 phr; reactive clay, 30 phr; CSP/P(AA-co-BA), 30 wt %.



(a)



(b)

Figure 10 SEM photographs of the water-swellable rubber; CR, 100 phr; CSP, 30 phr; reactive clay, 30 phr; (a) CSP was not modified in the water-swellable rubber; (b) CSP was modified with 30% poly(AA-co-BA).

ture surfaces are distinct. But in Figure 10(b), fracture surfaces are very blurry. It indicates that compatibility of rubber and modified CSP is better than that of rubber and unmodified CSP.

CONCLUSION

A novel WSR has been prepared by blending CR, reactive clay, and others additives with CSP, which was modified by IPNs technology with crosslinked P(AA-co-BA). The compatibility of CSP and chlorobutadiene rubber was improved greatly, when CSP was modified by IPNs technology with crosslinked P(AA-co-BA). And it resulted in better properties of WSR. When the content of CSP in modified WSR was 30 phr, and the content of crosslinked P(AA-co-BA) in the modified CSP was 30%, tensile strength, elongation at break, water-swelling ratio, and percentage loss of the WSR were 7.7 MPa, 1530, 438, and 2.5%, respectively.

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