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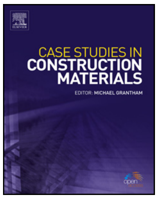

Short communication

Influence of superplasticizer on the rheology of fresh cement asphalt paste

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ABSTRACT

Cement asphalt (CA) paste is an organic–inorganic composite material of cement and asphalt emulsion. Its complicated rheological behavior affects its site application in high speed railway. Superplasticizers (SPs) are usually used to improve the construction properties of fresh CA mortar. However, the principle of SPs acting on the rheology of CA paste is seldom studied. In this paper, the effects of polycarboxylate (PCA) and naphthalenesulfonate (PNS) on the rheological properties of CA pastes, asphalt emulsions (both anionic and cationic) and cement pastes were studied, respectively from the viewpoint of adsorption and zeta potential. Centrifugation method was used to determine the adsorption of asphalt onto cement particle, electroacoustic method was employed to study the zeta potential of cement particles of concentrated paste, and optical microscopy was used to observe the dispersion of particles. The results suggest that both PCA and PNS can decrease the yield stress and apparent viscosity of CA pastes. The effect of SPs on the rheology of CA paste can be explained by two reasons. First, PNS can adsorb on both asphalt and cement surface, change the zeta potential and then decrease their yield stress and viscosity, while PCA only adsorb on cement surface. Second, the competitive adsorption of SPs and asphalt prevents asphalt from adsorbing on cement surface and then more asphalt droplets are released into aqueous solution, thereby enhancing the particle dispersion.

1. Introduction

Slab track systems of types CRTS I and CRTS II have been developed in China, which have been widely used in high speed railway in China. Cement asphalt (CA) mortar is used in the slab track systems as a cushion layer, which fulfills important structural functions (Zhao et al., 2008; Zuo et al., 2005). It consists of cement, asphalt emulsion, aggregate, water and other admixtures. The asphalt emulsion is one of the key composite materials of CA mortar. It is a thermodynamically unstable system with asphalt droplets uniformly dispersed in an aqueous solution of water, emulsifier and some admixtures (Tharwat, 2013; Wang et al., 2012).

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In the authors' previous work (Peng et al., 2014a,b), it is found that the yield stress and viscosity corresponding with practical engineering is very important for its construction quality. CA mortar is characterized by its high fluidity so that it can be placed without vibration, and easily grouted into the chamber between the concrete roadbed and track slab on site. In this respect, it should have a low yield stress, which can increase the spread-ability, however, also increase the probability of aggregate and bubble separation at the same time (Peng et al., 2014a; Feys et al., 2009; Cyr et al., 2000). The static segregation of aggregate and bubble is mainly governed by the yield stress, aggregate density and size. The dynamic segregation of aggregate is mainly governed by viscosity, aggregate density and size. Consequently, the yield stress and viscosity should be in an appropriate range-not too big or too low (Ouyang and Tan, 2015). Consequently, studying the rheological behavior of CA paste, especially the principle controlling its rheological behavior, is urgently needed.

CA mortar can be seen a particle suspension with aggregates dispersed in CA paste (Peng et al., 2014a,b; Zhang et al., 2012). Thus, the rheological behavior of CA mortar is depended on aggregates and particularly, the rheological behavior of CA paste. Only few publications were dealing with the rheological behavior of CA paste. Wang et al. (2008) had studied the rheological behavior of CA paste and the effect of thickener and silica fume, and asphalt to cement ratio (A/C). Zhang et al. (2012) had studied the influencing factors of type of asphalt emulsion, asphalt emulsion content, temperature, and shelf time on the yield stress of CA paste. Hu et al. (2009) had found that both anionic and cationic asphalt droplets exhibited favorable adsorption onto cement grain surface. Ouyang and Tan (2015), Ouyang et al. (2014) and Tan et al. (2014) had studied the rheological model, factors such as polycarboxylate superplasticizer influencing rheological parameters, demulsification process of asphalt emulsion of fresh CA paste. In the previous work, the research group had found that Herschel–Bulkley model is most suitable to character the rheological behavior of CA paste, and analyzed the effects of A/C and solid volume fraction (V_s) on the rheological behavior of CA paste (Peng et al., 2014a). Above all, the rheological behavior of CA paste is mainly determined by cement, asphalt emulsion and their relative proportion. Nevertheless, few chemical admixtures have been designed for CA mortar for the purpose of controlling its rheological behavior, and few studies have been dealing with it.

For fresh cement-based materials, SPs are often adopted to improve their rheology (Mikanovic and Jolicoeur, 2008; Papo and Piani, 2004). SPs are nowadays widely employed in cement technology, since they improve workability at a given water/cement ratio, or, on the other hand, they allow the same workability to be obtained as that of plain cement paste with a great reduction in water content, as well as final products with higher mechanical strengths to be manufactured (Papo and Piani, 2004). The increase in cement paste fluidity by the addition of SPs is connected with the dispersing action exerted by the adsorption of SPs on the cement surface, which modifies the zeta potential of particle or favors their dispersion on account of a phenomenon of steric impediment (Papo and Piani, 2004).

No specialized SP was developed for CA mortar, despite its widespread use in the area of high speed railway in China. More in-depth studies are needed on this field. In the previous work (Peng et al., 2014a), the authors had found that SPs can decrease the apparent viscosity and extent of shear-thinning of CA paste. Ouyang (Tan et al., 2014) had found that polycarboxylate superplasticizer (PCA) can reduce the initial yield stress and plastic viscosity, prevent CA paste from flocculation in order to reduce the yield stress growth rate, and PCA is not related with the growth rate of plastic viscosity. CA mortar used in ballastless track has very high asphalt emulsion content, ranging from 50% to 140% of cement by weight, which is much higher than that of ordinary polymer modified cement materials. Thus, CA mortar could be either cement-based or asphalt-based, whose rheological behavior is very complicated. The effect of SPs on the rheology of asphalt emulsion and cement paste should be clarified respectively. And then the effect of SPs on the rheology and particle dispersion of CA paste is analyzed. In CA paste, asphalt droplets can adsorb on cement surface and increase the cement dispersion (Peng et al., 2014a; Hu et al., 2009). When SPs and asphalt emulsion are added into cement paste at the same time, there may be a competitive adsorption of asphalt and SPs on cement surface, which has an influence on the particle dispersion of CA paste (Shiyun et al., 2010; Beaudoin and Ramachandran, 1989).

A multi-method approach is required to understand different aspects of SP behavior in fresh CA paste. Yield stress and apparent viscosity are the two main rheological parameters which are used to quantify the effect of SP addition to the cement based paste. Considered that CA paste is a composite of asphalt emulsion and cement, the effect of SPs on the rheology of asphalt emulsion and cement paste is first studied respectively in this paper. The zeta potential measurement is adopted to study the adsorption of SP molecules on asphalt and cement surface. An asphalt adsorption measurement is used to analyze the effect of SPs on the particle dispersion of asphalt and cement in CA paste. Optical microscopy observation was made on the paste to study the particle dispersion.

2. Experimental

2.1. Materials and formulation procedure

2.1.1. Cement and asphalt emulsion

CA pastes were prepared with a Portland cement, which physical properties and chemical composition are listed in Tables 1 and 2, respectively, and a cationic and anionic emulsified emulsion which physical properties are listed in Table 3. The particle size distribution of cement and asphalt are shown in Fig. 1.

Table 1
Physical and mechanical properties of cement.

Specific surface ($m^2 kg^{-1}$)	Bending strength (28d) (MPa)	Compressing strength (28d) (MPa)	Particle size (μm)
341	8.1	58.6	16.98

Table 2
Cement chemical and mineral composition (w/%).

Component	SiO_2	CaO	Al_2O_3	Fe_2O_3	SO_3	C_3S	C_2S	C_3A	C_4AF	Ignition loss
Content (%)	21.29	62.33	5.10	2.91	2.18	47.23	25.49	8.6	8.85	2.90

Table 3
Properties of cationic asphalt emulsion.

Type	Material retained on 1.18 mm sieve (%)	Solid content (%)	pH value	Particle size (μm)	Density (g/cm^3)
Cationic asphalt	0.005	59.8	3.44	2.61	1.02
Anionic asphalt	0.003	59.6	9.20	1.97	1.02

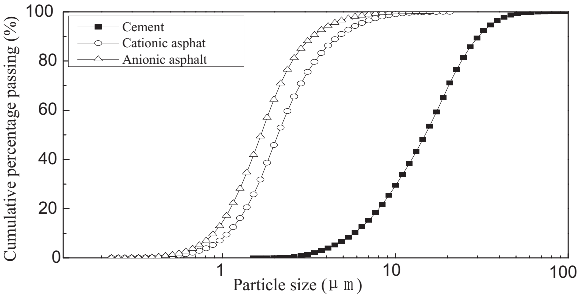


Fig. 1. Particle size distribution of cement and asphalt particles.

2.1.2. *SP*

The SPs are a polycarboxylate (PCA) and naphthalenesulfonate (PNS) superplasticizers with a solid content of 30%. The zeta potential of PCA and PNS solution is 8.1 and −41.1 mV, respectively. The pH of PCA and PNS solution is 6.65 and 7.70, respectively. All the SPs were added with a mass ratio of chemical admixture to cement or asphalt emulsion. And the addition of SPs is according to the ordinary concentration in cement paste and CA mortar.

2.1.3. *Mixing procedure*

All pastes were mixed in a 1 L high-shear mixer rotating at approximately 360 rpm. Such a high-shear mixing regime was used to ensure complete homogeneous suspension. The temperature of materials and mixer were controlled to keep the temperature of pastes at 25 ± 2 °C. The mixing sequence consisted of mixing water, SP and asphalt emulsion at the speed of 60 rpm for 30 s, cement was then introduced gradually within 30 s while the mixer was turned on, and the mixture was mixed at the speed of 120 rpm for a total time of 120 s.

2.2. *Rheology measurement*

Rheology was measured using a “RheoPlus QC” coaxial cylinder rotary rheometer. The type of rotator to test CA and cement paste is ST22-4V-40 and an outer tube of 163 ml volume with 42 mm diameter and 118 mm length, while the type of rotator to test asphalt emulsion is CC39 due to its extremely low viscosity. Considering that the effect of temperature on the rheology of pastes, the temperature of the samples was controlled at 25 ± 2 °C by a water bath. The rheological curves of pastes were tested at 5 min after mixing. The rheological test protocol consists of shearing the paste at a high shear rate of 100 s^{-1} for 60 s to cause structural breakdown of pastes and create uniform conditions before testing. The flow curve was then determined by decreasing the shear rate from 100 s^{-1} to 0.1 s^{-1} during 100 s.

2.3. *Zeta potential measurement*

Zeta potential test was carried out using the zeta potential instrument (as shown in Fig. 2) which worked on the basis of the electroacoustic method. It applies an alternating electric field on the suspension and causes the charged particles to oscillate. The motion of the particles gives a sound wave which is corresponding to the dynamic mobility of the colloidal

Fig. 2. Zeta potential instrument of thick grout.

particles. The zeta potential is calculated from the dynamic mobility which is detected by the zeta potential instrument. About 280 ml of the sample was poured in the sample cell and stirred at a speed of 250 rpm avoiding particles segregation.

2.4. Adsorption of asphalt

Followed by the filtration method to test the adsorption ratio of asphalt onto cement surface (Hu et al., 2009), it is very difficult to separate the cement of 16.98 μm and asphalt particles of about 2 μm. Then the centrifugation method to test the adsorption ratio was adopted, as follows: firstly, the dry centrifuge tube was weighed; secondly, the testing sample was poured in centrifuge tube, and the whole CA paste and centrifuge tube were weighed; thirdly, the CA paste was centrifuged at a speed of 2000 rpm in 5 min, accounting that too small speed and time cannot completely centrifuge the CA paste, excessive speed and time lead to three parts of cement, asphalt and aqueous solution (as shown in Fig. 3); at last, the supernatant part was poured out and dried to measure the asphalt content in supernatant part. The adsorption ratio could be calculated using Eq. (1). In Eq. (1), Π is the asphalt adsorption ratio, m_c is the cement content before centrifugation (g), m_a is the asphalt content before centrifugation (g), and $m_{a\text{-}s}$ is the asphalt content retained in supernatant part (g).

$$\Pi = \frac{m_a - m_{a\text{-}s}}{m_c} \times 100\% \tag{1}$$

2.5. Optical microstructure

Microstructure was observed 5 min after cement adding in water using an optical microscope. To clearly observe the solid particles, the fresh CA pastes were diluted 10 times.

3. Results and discussion

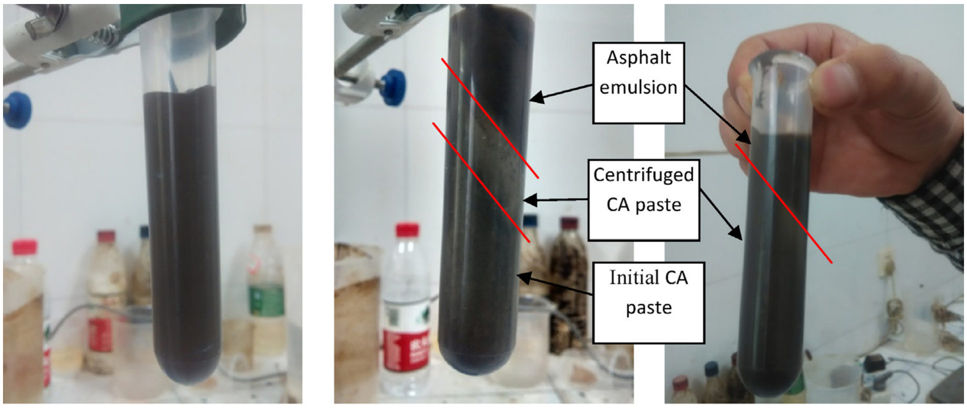
Recently, the authors found that the Herschel and Bulkley (1926) model was quite suitable for describing the rheological behavior of CA paste (Peng et al., 2014b). Consequently, the Herschel–Bulkley model was used to calculate the yield stress in this paper, as given by:

$$\tau = \tau_0 + k\dot{\gamma}^n \tag{2}$$

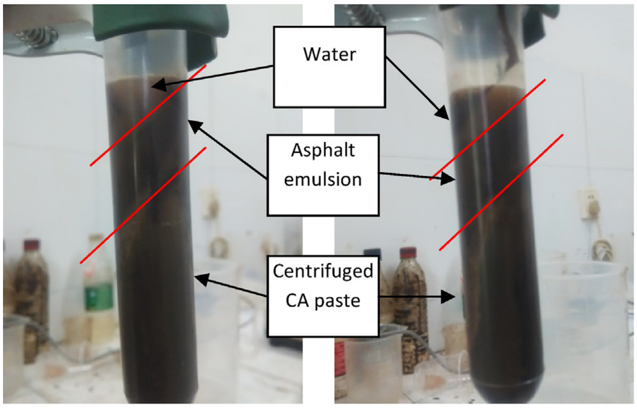
where τ_0 is the yield stress, k is the consistency, and n is the non-Newtonian index. The apparent viscosity of 16 s^{-1} is also adopted to analyze the influences of chemical admixtures on the rheological properties of CA paste, accounting that this shear rate is the maximum shear rate in pouring CA mortar (Peng et al., 2014a). The ‘apparent viscosity’ in the later of this paper is related to apparent viscosity of 16 s^{-1}.

3.1. Effect of SPs on the rheology of asphalt emulsions

SPs are usually used to increase the flowability of cement concrete, owing to their dispersion effect (Feys et al., 2009; Cyr et al., 2000; Papo and Piani, 2004). However, they are seldom used in polymer emulsions. The rheology of asphalt emulsions with various SPs is shown in Fig. 4. Fig. 4 illustrates that the PCA increases the yield stress and apparent viscosity of two asphalt emulsions. And the yield stress and apparent viscosity are increased along with the increase content of PCA. On the contrary, the PNS decrease the yield stress and apparent viscosity of two asphalt emulsions.



Initial status 2000 rpm, 3 min 2000 rpm, 5 min



2000 rpm, 8 min 3000 rpm, 5 min

Fig. 3. The centrifuged picture of CA paste with various rotation rate and time.

Asphalt emulsion is a particle suspension, the stability of which is ensured by the adsorption of emulsifiers. The cationic or anionic emulsifier is a type of amphiphilic molecules with both hydrophilic and hydrophobic groups (Tharwat, 2013). The hydrophobic groups can adsorb on asphalt surface, while the hydrophilic groups project into water. The hydrophilic groups can ionize in water and make the asphalt be positive or negative charged. The pH value of PCA and PNS is 6.65 and 7.70 which is close to electric neutrality, consequently, the pH value of SPs has ignored effect on the pH value and zeta potential of asphalt emulsions.

The PNS molecule has a high density of ions (−41.1 mV), while the PCA molecule has a low density of ions (8.1 mV). When SP molecules are added into asphalt emulsion, they should have a tendency of adsorption on asphalt droplets only seen from the hydrophobic and hydrophilic groups. Considered that the charge density of SPs and asphalt droplets are different, the adsorption of SP molecules onto asphalt surface can be observed by a zeta potential measurement. The zeta potential of

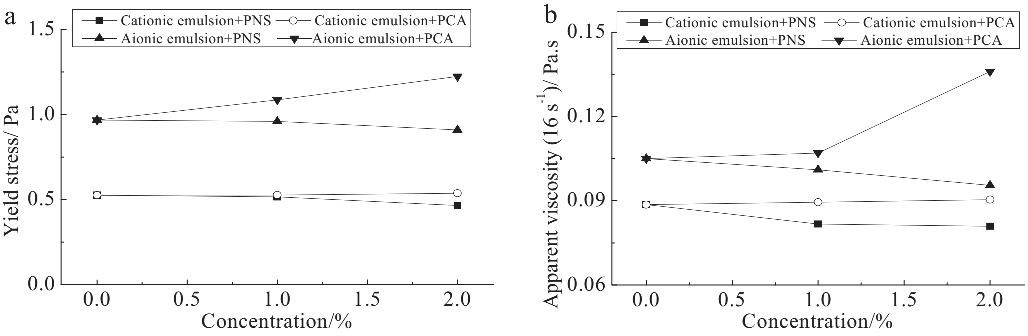


Fig. 4. The rheology of cationic and anionic asphalt emulsion with various SP content.

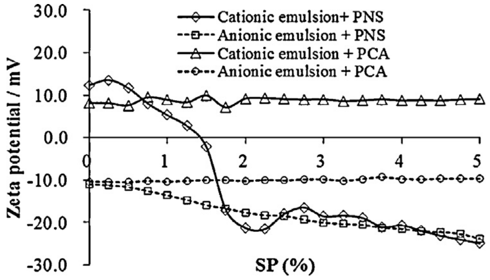


Fig. 5. Zeta potential of asphalt emulsion with various SP content (Peng et al., 2014a).

asphalt emulsions with various SPs is shown in Fig. 5. It can be concluded that the PNS obviously decrease the zeta potential of both cationic and anionic asphalt, which proves that PNS molecules can still adsorb on asphalt droplets or substitute for the emulsifier molecules when the asphalt has had saturated adsorption of emulsifier molecules. Considered that the surface area of asphalt droplet is limited, the substitute for the emulsifier molecules is more credible (Shiyun et al., 2010, 2013; Beaudoin and Ramachandran, 1989). The addition of PCA molecules does not change the zeta potential of asphalt emulsion and increase the particle dispersion. However, PCA can adsorb on solid surface and increase the particle dispersion. Then the fact of PCA molecules not adsorbing on emulsified asphalt surface is more credible.

Generally speaking, asphalt emulsion is a dispersed suspension by its charged asphalt droplets. Then the rheology has a relation with the zeta potential. The PNS increases the absolute zeta potential of asphalt emulsions and increases their dispersion, then decreases the yield stress and viscosity. However, PCA cannot change the zeta potential of asphalt emulsion, resulting in an unchanged electrostatic repulsion. Then it has little effect on the rheology of asphalt emulsion. Nevertheless, as a type of polymer molecule, PCA still can increase the viscosity of aqueous solution, and then increase the viscosity of whole emulsion (Khayat, 1998).

3.2. Comparison of the effects of SPs and asphalt emulsions on the rheology of cement paste

Asphalt emulsion and SPs can improve the flowability of fresh CA mortar (Peng et al., 2014a; Zhang et al., 2012; Mikanovic and Jolicoeur, 2008; Papo and Piani, 2004; Wanga et al., 2015). The comparison of the effects of SPs and asphalt emulsions on the rheology of cement paste is shown in Fig. 6. The water to cement ratio by weight (W/C) is 0.37. It can be seen that all the four polymers can decrease the yield stress and viscosity of cement paste, and the decreasing extent is increased with the increasing of polymers content. The SPs of PCA and PNS are more effective in decreasing the yield stress and viscosity than asphalt emulsions. And the PCA is more effective than PNS regardless of content.

The most widely accepted explanation for the mechanism of rheology is that the SP and asphalt emulsions can adsorb on the surface of cement grains and disperse the flocculated cement (Peng et al., 2014a; Zhang et al., 2012). After being mixed with water, cement grains begin hydrating, consequently developing a heterogeneous charge distribution on the surface of hydrating cement grains. Silicate hydrates exhibit a negative surface charge, whereas aluminate hydrates possess a positively charged surface. Meanwhile, the hydrating cement particles also provide adsorption sites for positively and negatively charged ions, molecules, and particles. This phenomenon in the superplasticizer system has been extensively investigated (Mikanovic and Jolicoeur, 2008; Papo and Piani, 2004). The adsorption of asphalt emulsions and PNS neutralizes the surface charge of the hydrating cement, thereby preventing the flocculation of cement grains by inducing electrostatic and/or steric repulsion interaction among the cement grains.

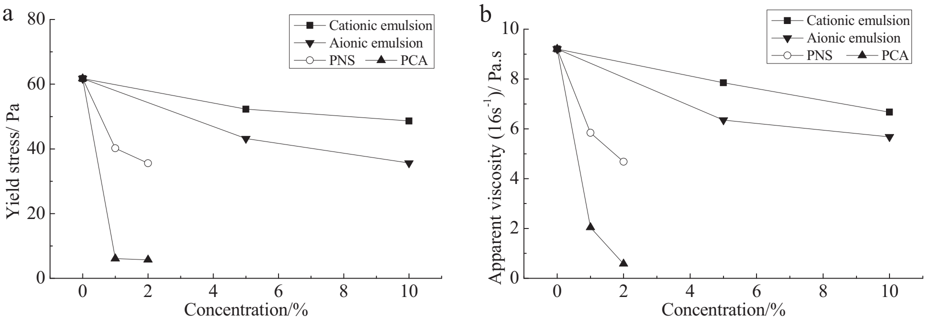


Fig. 6. The rheology of cement particles with various polymers.

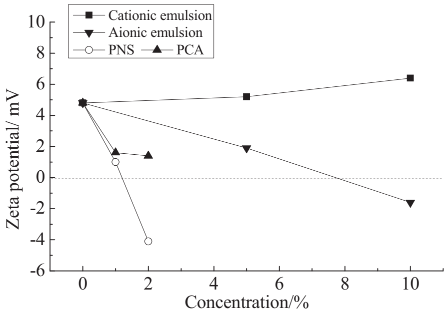


Fig. 7. The zeta potentials of cement particles with various polymers.

In order to analyze the asphalt emulsions and SPs on the rheology of fresh CA paste, the adsorption of asphalt emulsions and SPs on cement surface and its effects on the particle interaction should be measured. Literatures (Hu et al., 2009; Mehta and Monteiro, 2006) show that the concentrations of asphalt emulsions and SPs in the present paper are far below their saturated contents. Then all the added polymers might be adsorbed on cement surface.

The zeta potential of cement particles with various polymers is shown in Fig. 7. The W/C of cement paste is 0.37. Fig. 7 shows that all the polymers can change the zeta potential of cement particles, which proves the adsorption of polymers on cement surface. The zeta potential is all decreased with the increasing of polymer content except cationic emulsion. Only the anionic emulsion and PNS can change the positive zeta potential to negative with high content. The cement particle with PNS has the biggest negative zeta potential. And cement particle with the anionic emulsion has a biggest positive zeta potential. The PCA only change the zeta potential of cement particle a little, accounting that it mostly provides a steric repulsion.

The cement paste with PCA has the lowest yield stress and viscosity, though has a high zeta potential, accounting that its steric hindrance is more effective than other polymers with electrostatic repulsion. The asphalt emulsion has decreased the rheology of cement paste, which is due to its neutralizing the opposite charge of cement surface (Peng et al., 2014a; Zhang et al., 2012; Shiyun et al., 2010). However, the asphalt emulsions also increase the solid volume fraction (V_s) of cement paste, whereas they have V_s of about 60%. Then the dispersion of asphalt emulsion is reduced. Above all, the SPs are more efficient than asphalt emulsion on improving the rheology of cement paste.

3.3. Effect of SPs on the CA paste

The curves in Figs. 8 and 9 illustrate that PNS and PCA has an advantage on decreasing yield stress and viscosity of CA paste with cationic emulsion (PC-CEA) and CA paste with anionic emulsion (PC-AEA) pastes. The asphalt to cement ratio by weight (A/C) and W/C of CA pastes are 0.30 and 0.51, respectively. All the pastes show a decrease in yield stress and viscosity with the increase content of SPs, regardless of the type of CA paste. The yield stress and viscosity of CA pastes with PCA are lower than ones with PNS, regardless of the concentration of SPs. It can be included from the experiment results that the PCA molecules are highly effective on increasing the dispersion of cement particles in CA paste.

Since that both SPs and asphalt droplets can adsorb on cement surface and improve the rheology of CA paste, the contribution of SP or asphalt on the rheology is complex. Then the effect of SPs on the asphalt adsorption ratio on cement surface was studied as shown in Fig. 10. The A/C and W/C of CA pastes are 0.30 and 0.51, respectively. Seen from Fig. 10,

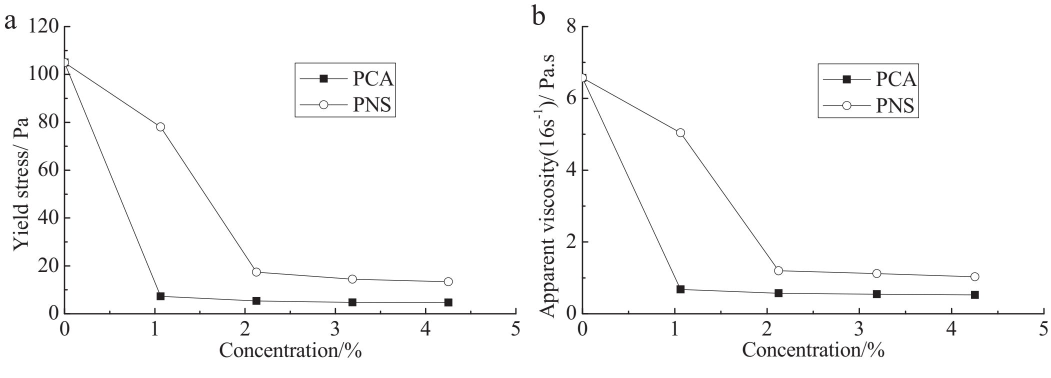


Fig. 8. Rheology of PC-CEA pastes with various SPs.

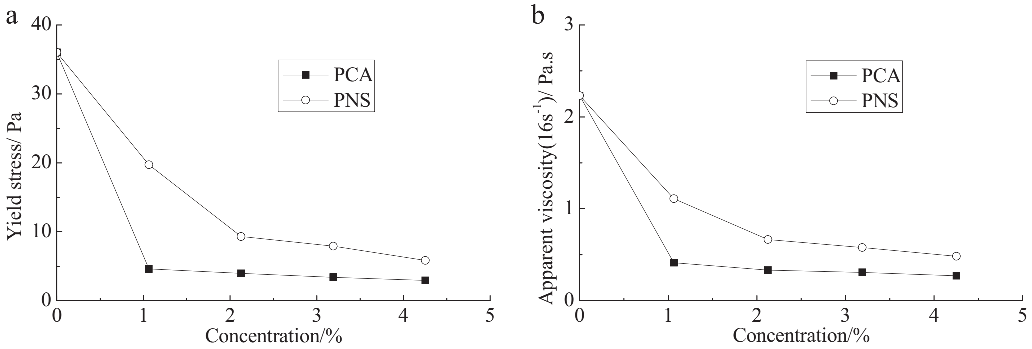


Fig. 9. Rheology of PC-AEA pastes with various SPs.

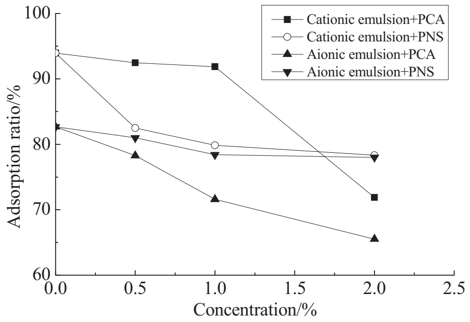


Fig. 10. The effect of SPs on the asphalt adsorption on cement.

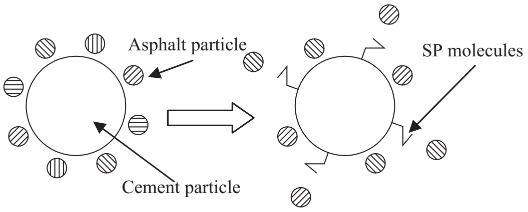


Fig. 11. The procedure of competitive adsorption of SP molecules with asphalt droplets.

the addition of PNS and PCA both decrease the asphalt adsorption ratio. For PC-CEA paste, PCA leads to a higher adsorption ratio than PNS when SP concentration is lower than 2%, however, a lower adsorption ratio when SP concentration is higher than 2%. For PC-AEA paste, PCA leads to a lower adsorption ratio than PNS, regardless of SP content.

The results indicate that there is a competitive adsorption on cement surface between SPs and asphalt droplets. Considered that SPs are more efficient in improving the rheology of cement paste, more SP molecules adsorbing on cement surface indicates an increasing of the cement particle dispersion and decreasing of the yield stress and viscosity. On the other hand, a lower adsorption ratio of asphalt means that more adsorbed asphalt droplets are released into aqueous solution, acting as free droplets. Free asphalt droplets are spherical and can act as a lubricating effect, which is very good at the movement of cement particle (Peng et al., 2014a). The procedure of competitive adsorption of SPs with asphalt droplets can be illustrated in Fig. 11.

The particle dispersion is a very important yardstick to affect the rheology of cement based paste (Ish-Shalom and Greenberg, 1960). The optical microstructure of CA paste with SP is shown in Fig. 12. It shows that the addition of SP decreases the particle size of cement, more asphalt droplets released into aqueous solution, which proves the above analysis from another aspect.

Compared with PNS, PCA can decrease the yield stress and viscosity of cement paste, increase yield stress and viscosity of asphalt emulsion and cause more asphalt released into aqueous solution, as a result, decrease the yield stress and viscosity of CA paste. Above all, improving the dispersion of cement particles paste and promoting of asphalt desorption are the main reasons to decrease rheological parameters of CA paste.

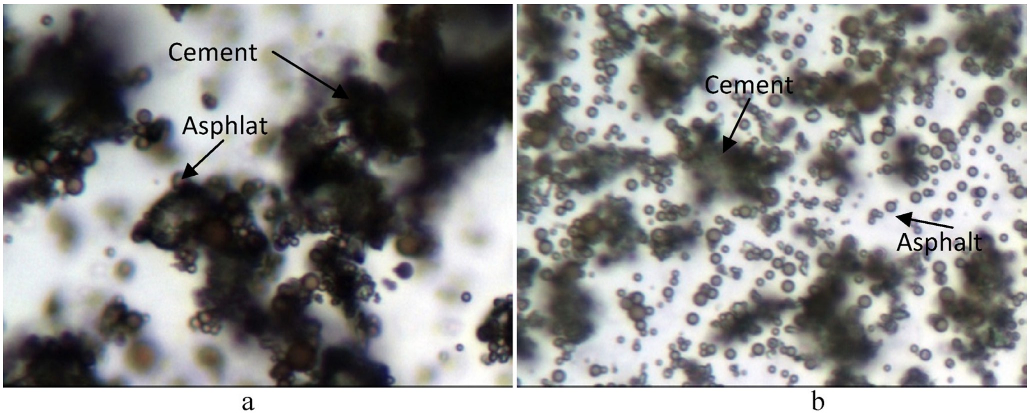


Fig. 12. Optical microstructure for fresh CA paste at 5 min after preparation: (a) Reference; (b) SP of 2% content.

4. Conclusions

Based on the experimental investigation carried out in this paper, the following conclusions can be made:

1. The rheology of fresh CA paste is mainly dominated by the interaction of cement particles which may adsorb SP molecules and asphalt droplets.
2. PNS molecules can adsorb onto asphalt droplet benefiting rheological behavior of both anionic and cationic asphalt emulsions, while PCA molecules don't.
3. PCA and PNS molecules have strong competitive adsorption with asphalt droplet onto cement particles.
4. PCA is more efficient in decreasing the yield stress and apparent viscosity of CA paste than PNS which may be ascribed to the following reasons: (1) PCA and PNS acting on cement particles by steric effect and electrostatic effects, respectively; (2) more asphalt droplets are released which improve the rheological behavior of CA paste.

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