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The Effect of pH on the Properties of a Cationic Bitumen Emulsifier

Surfactants used in road surface treatments have an optimal application pH value which is an important condition for applications, otherwise stable bitumen emulsions with high solid contents are impossible to achieve. Therefore, a wide range of pH values were employed to investigate its effect on the bitumen/water interfacial properties of a cationic bitumen emulsifier. It is shown that interfacial tension and dilatational modulus have correlations with pH value. The lowest value of interfacial tension declined with the decrease of pH value. The strong acid system has the highest dilatational modulus while this modulus of the neutral system is the lowest. Compared with the neutral system, the maximum of the dilatational modulus also appears in the acid or alkaline system at a relatively low concentration. Physical properties of bitumen emulsions, including storage stability and Zeta potential, show the same changing rule as the interfacial rheology.

Key words: pH effect, bitumen emulsifier, interfacial activity, bitumen/water interface

Der Einfluss des pH-Wertes auf die Eigenschaften eines kationischen Bitumen-Emulgators. Tenside, die im Straßenbau für die Behandlung der Straßendecke verwendet werden, haben einen optimalen pH-Wert, der für die Anwendung eine wichtige Voraussetzung ist, da, ansonsten stabile Bitumenemulsionen mit hohen Feststoffgehalten unmöglich eingestellt werden können. Daher wurde der pH-Einfluss auf die Grenzflächeneigenschaften eines kationischen Bitumen-Emulgators in einem Bitumen/Wasser-System in einem breiten pH-Bereich untersucht. Es wird gezeigt, dass die Grenzflächenspannung und das Dilatationsmodul Korrelationen mit dem pH-Wert aufweisen. Der niedrigste Wert der Grenzflächenspannung nahm noch mit der Abnahme des pH-Wertes ab. Das Starke-Säure-System hat das höchste Dilatationsmodul, während das neutrale System das niedrigste hat. Im Vergleich zum neutralen System tritt das Maximum des Dilatationsmoduls auch bei einer relativ geringen Konzentration im Säure- oder Alkalisystem auf. Die physikalischen Eigenschaften von Bitumenemulsionen, einschließlich der Lagerstabilität und des Zetapotentials, zeigen dieselbe sich ändernde Regel wie die Grenzflächenrheologie.

Stichwörter: pH-Einfluss, Bitumenemulgator, Grenzflächenaktivität, Bitumen-Wasser-Grenzfläche

1 Introduction

Bitumen emulsions which are prepared by asphalt binders and emulsifiers have been used for road surface treatments such as chip seals [1]. As the asphalt binders have various components, bitumen emulsions become a complex system and it is difficult to understand the physical behavior of emulsified bitumen. However, this issue is the key point to enhance the performance of bitumen emulsions in applications. Therefore, understanding and predicting the behavior and performance characteristics of emulsified bitumen is an important topic for researchers.

Interfacial studies are effective ways to research the fundamental mechanisms, including kinetics of film formation, surfactant adsorption and film rupture, which indicate emulsion behaviors. Interfacial film compressibility and elasticity can be quantified via Langmuir film balance techniques [2, 3], shear viscometry [4, 5] and oscillatory drop measurements [6, 7]. Among these methods, interfacial dilatational rheology is a useful technique to understand the interfacial adsorption behavior of surfactants, as well as their elasticity at air-liquid and liquid-liquid interfaces [8–10]. This technique has also been used to explore film elasticity at different interfaces, such as crude oil-water interface [11], model oil-water interface [12] and model oil-air interface [13]. It has been reported that there is a correlation between the emulsion stability and rheological properties of the water-oil interface that high elasticity is corresponding to high stability. Ortiz et al. studied the effect of additives on asphaltene interfacial films and emulsion stability via the observation of the change in film properties. There were two opposing effects of additives on film properties and emulsion stability [14]. Kang et al. analyzed the stability mechanism of a w/o crude oil emulsion stabilized by polymer and surfactant. The two stabilizers presented different effects, i.e. the surfactant decreased the interfacial tension and the polymer increased the interfacial elasticity [15].

Cationic bitumen emulsions are widely used in surface treatment. They deposit more rapidly than anionic and nonionic emulsions on aggregate surfaces, bonded to the aggregates by the electrostatic interaction at the interface of bitumen and aggregate material. When cationic surfactants have been applied for the preparation of bitumen emulsions, they should be used at an optimal value of pH. Otherwise, the interfacial activities would dramatically decrease and it is impossible to obtain stable emulsions with high solid content of bitumen. The cationic soap solutions are normally prepared by dissolving the cationic surfactant in water to which a sufficient amount of a suitable acid, for instance, hydrochloric, sulfuric and phosphoric acid, is added until the desired pH value from 1 to 7 is reached, and a clear emulsifier solution is obtained [16]. Commonly, a narrow range of pH is essential for the cationic bitumen emulsion to present a favorable pavement performance. Although cationic bitumen emulsifiers have been widely used in road maintenance, less work has been made to study their characteristics in this field. Herein, the bitumen/water interfacial activities of a bitumen emulsifier commonly used were investigated in a wide range of pH values to reveal the relation between interfacial properties and pH, which may provide a guidance for selecting optimal conditions in the preparation of bitumen emulsions used in road surface treatments.

2 Experimental Procedure

2.1 Materials

The cationic slow-set emulsifier INDULIN W-5 was employed to prepare bitumen emulsions, which is purchased from MeadWestvaco Co. and used as received. It is designed to perform in a wide variety of applications including slow set slurry surfacing, tack coat, fog seal, and solventless cold mix applications such as recycling and base stabilization. Emulsifiers of the INDULIN series have been widely used in road surface treatment and they have similar molecular structures [17]. The optimal pH for its acidic soap solution is between 2 and 3, which is well-known in applications and adopted accordingly. In this paper, aqueous solutions of W-5 were acidified by adding hydrochloric acid to obtain a pH value ranging from 3 to 9.

2.2 Interfacial tension measurements

Interfacial tension was measured using a TECLIS TRACKER drop shape analyzer. The oil phase was loaded into a syringe and injected through a U-shaped needle into a quartz cuvette containing the water phase. A droplet was formed at the tip of the needle and illuminated. The profile of the droplet was captured using a CCD camera and analyzed by a video image profile digitizer board connected to a personal computer. The dynamic interfacial tension was also investigated using the pendant drop technique. Before the droplet was formed, the image capture software was triggered, collecting images at 2 frames/s for the first 10 min and 1 frame/min thereafter. Experimental runs of 10,000 s were chosen according to the literature reported [18]. The samples at the concentration of 1000 mg/L with different pH were measured to obtain their dynamic interfacial tensions. Replicate measurements of each sample studied in this article have been carried out. Replications of one target sample have similar resultant values, and the standard deviation value for the experimental data of each sample is <0.2.

2.3 Interfacial elasticity measurements

To obtain the interfacial dilatational rheology of a liquid-liquid interface, a droplet is formed to provide an interface and the interfacial tension (γ) is tracked as a function of time. Controlled oscillatory strain deformations of interfacial area (A) are applied to recover rheological information of monolayers or third phase films at the interface. The interfacial dilatational modulus (ε) is defined by the following expression [19]:

$$\varepsilon = \frac{d\gamma}{d\ln A} = A\frac{d\gamma}{dA} \tag{1}$$

In an oscillating system, the interfacial dilatational modulus is a complex quantity and has both a real and an imaginary component defined as follows:

$$\varepsilon = \varepsilon' + \varepsilon'' \tag{2}$$

where ε' is the real component (elastic modulus), and ε'' is the imaginary part (viscous modulus). Depending on the system of interest, the $\gamma(t)$ response may lag behind the imposed $A(t)$. This lag is described by a phase shift, ϕ, which is defined as phase angle. Purely elastic interfaces present the $\gamma(t)$ behavior completely in-phase with $A(t)$ and the phase angle equal to 0°, whereas purely viscous interfaces are completely out-of-phase, with a phase angle of 90°. The elastic and viscous moduli can then be expressed as functions of the magnitude of the dilatational modulus, $|\varepsilon| = (\varepsilon'^2 + \varepsilon''^2)^{1/2}$, and the phase shift can be expressed by the following equations:

$$\varepsilon' = \varepsilon_d = |\varepsilon|\cos\varphi \tag{3}$$

$$\varepsilon'' = \omega\eta_d = |\varepsilon|\sin\varphi \tag{4}$$

All dilatational rheology experiments were run on an oscillating pendent drop tensiometer TRACKER. Finer control of drop volume was afforded by using a 500 µL microsyringe and higher-gauge curved needle. A personal computer analyzes images of the droplet shape to solve for γ from the force balance between Laplace and head pressure on the droplet.

The oscillation amplitude has an effect on the elastic and viscous moduli [12]. For amplitudes up to 45 % of the initial area, a Laplacian drop was maintained and the total modulus did not vary significantly from that measured when the amplitude was as low as 2 %. In the current work, the amplitude of oscillation was 10 % of the initial area as the other researchers did [19, 20].

Elasticity measurements were taken after 6 h, in which situation surfactants are likely to approach their adsorption equilibrium at the oil/water interface. To obtain an elasticity measurement, the drop size was oscillated periodically rather than continuously. The duration of each set of oscillations was 0.017, 0.033, 0.1, 0.2, and 0.5 Hz (periods of 60, 30, 10, 5, and 2 s, respectively), and the interval between sets of oscillations is one period. The surfactant concentration ranges from 1 to 1000 mg/L. Replicate measurements of each sample studied in this article have been carried out. Replications of one target sample have similar resultant values, and the standard deviation value for the experimental data of each sample is <0.3.

2.4 Preparation of bitumen emulsions

An aqueous phase was prepared by dispersing 5.0 wt.% emulsifier in distilled water. Bitumen emulsion was obtained by homogenizing 60 wt.% asphalt binder (temperature 130 °C) with 40 wt.% aqueous phase (temperature 55 °C) in a high-speed colloid mill.

2.5 Zeta potential measurement

Zeta potentials were measured using a Zeta plus potential analyzer manufactured by Brookhaven Instruments Corporation. For each measurement, one millimeter sample was added in 500 mL distilled water and dispersed with the help of magnetic stirrers for 10 min as reported [21]. To obtain the Zeta potentials, samples have been tested five times and error bars are marked for each data.

2.6 Storage stability measurement

Storage stability of bitumen emulsion was measured according to ASTM D244. This test method determines the difference of the sample residues in percent taken from the top and bottom of material placed in undisturbed simulated storage for 24 h. The result is expressed as the average of the two individual values obtained by determining the difference between the percent residue of the top and bottom samples for each storage cylinder. Low value is related to favorable storage stability. Each sample has been tested twice and error bars are marked for each data.

3 Results and Discussion

3.1 Interfacial activity

Interfacial tensions of INDULIN W-5 at pH values ranging from 3 to 9 were measured and shown in Fig. 1. These results are good evidence for surfactants in bitumen emulsions that they have the ability of migrating to the bitumen/water interface. The interfacial tensions decrease with the increase of surfactant concentration. As the concentration increases, more surfactant molecules adsorb at the bitumen/water interface, resulting in a decrease of interfacial tension. The interfacial tension stops declining until the adsorption equilibrium is achieved. In this regard, the number of W-5 molecules which adsorb at the bitumen/water interface reaches saturation. As shown in Fig. 1, the pH value of the bulk solution has great influence on the interfacial tension of W-5. The critical micelle concentration shows a downward trend with rising pH value. It seems that W-5 molecules form micelles at relatively high concentrations when the pH value is low. The lowest value of interfacial tension declines with the decrease of pH value, which is 8.9, 7.3, 6.4 and 4.7, respectively. It is also clear that the interfacial tension at the pH 3 is much lower than other values at the surfactant concentration of 1 mg · L^{-1}.

When the pH value is low, the amines are neutralized by hydrochloric acid and chlorohydrates that are formed, are stronger water-soluble [22]. Amine chlorohydrates are the effective emulsifying species, thus the ability of W-5 to reduce the interfacial tension improves at low pH value. When hydrochloric acid is added to modify the pH value of W-5 solu-

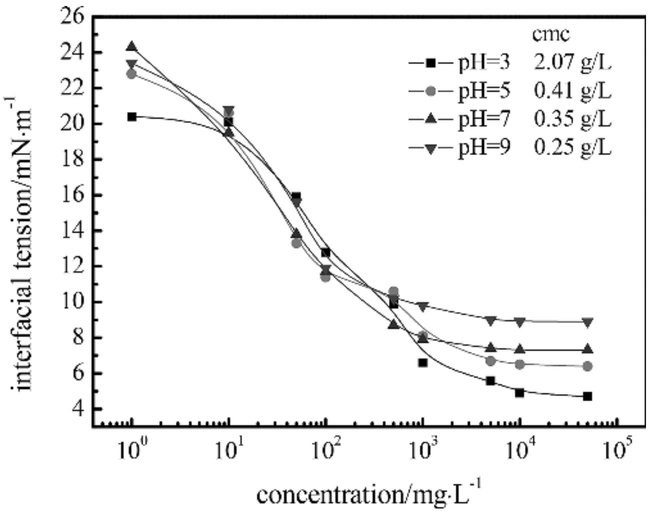

Figure 1 Variation of interfacial tension of INDULIN W-5 at different pH as a function of concentration

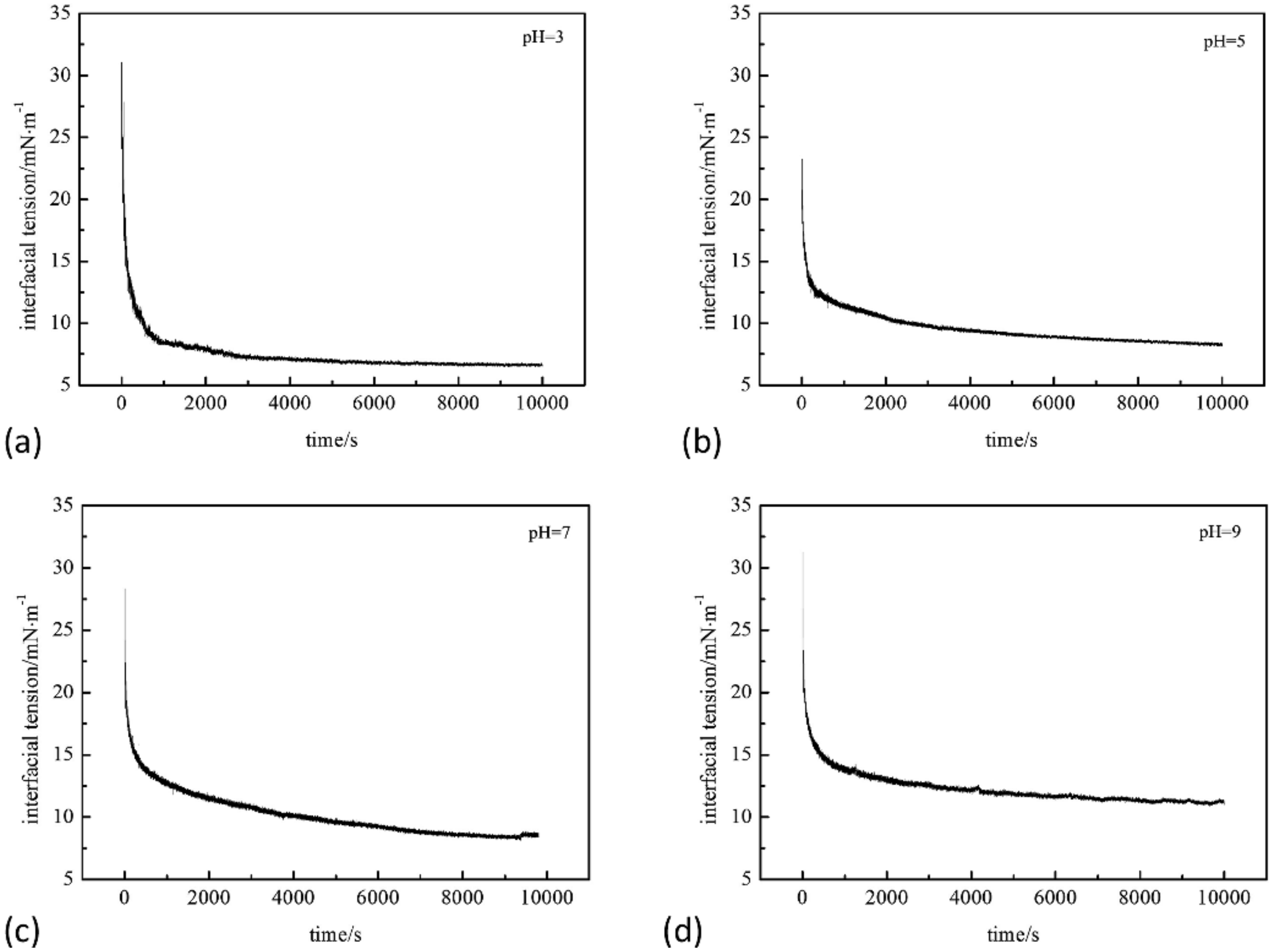

Figure 2 Dynamic interfacial tension at a fixed concentration of 1000 mg/L at different pH values

tion, counterions of Cl^- are dissolved in the bulk solution and they actively migrate to the headgroups of ionic emulsifier and attach there. The counterions have the ability to compress the electrical double layer of the headgroups, which weakens the electrostatic interactions among the headgroups of W-5 [23]. As a result, the adsorption layer has a relatively compact structure and the interfacial tension decreases remarkably. Because more cationic emulsifier molecules adsorb at the bitumen/water interface, micelles form at a relatively high concentration in bulk solution.

The dynamic interfacial tension is shown in Fig. 2. All the target samples show a rapid initial decrease followed by a progressive reduction in the decay rate, and then the interfacial tension appears to approach asymptotically a certain value with time. This value is 6.5, 8.1, 8.5 and 11.1 mN/m as the pH value is 3, 5, 7 and 9, respectively. The dynamic interfacial tension represents an adsorption process of surfactants at the bitumen/water interface. Among four target systems, the one with the pH value of 7 takes more time to reach the equilibrium. It is considered that particles in solution have a relatively slow migration velocity in the neutral system.

As shown in Fig. 3, the dilatational modulus is a function of the frequency in each system, which is caused by relaxation processes at the interface. As a result, the dilatational modulus increases with the oscillation frequency [24, 25]. It is also obvious that both surfactant concentration and pH value have great influence on the dilatational modulus. The dilatational modulus reaches to the maximum value at the concentration of 50 mg · L^{-1} in acid or alkaline system, and at concentration of 100 mg · L^{-1} in neutral system. The sam-

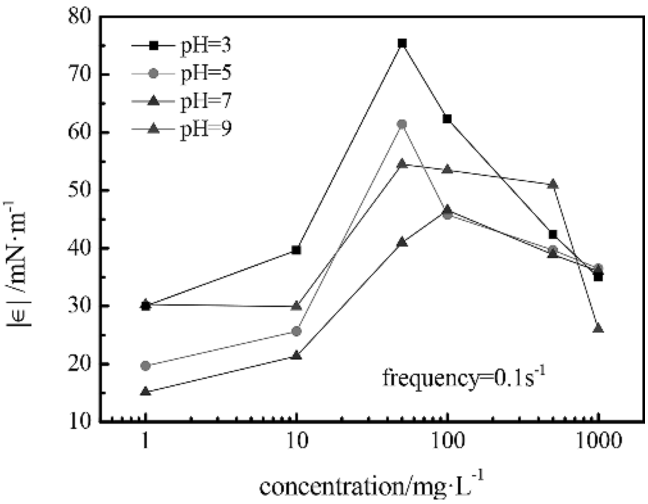

Figure 4 Variation of the dilatational moduli as a function of emulsifier concentration at a fixed frequency of 0.1 s^{-1}

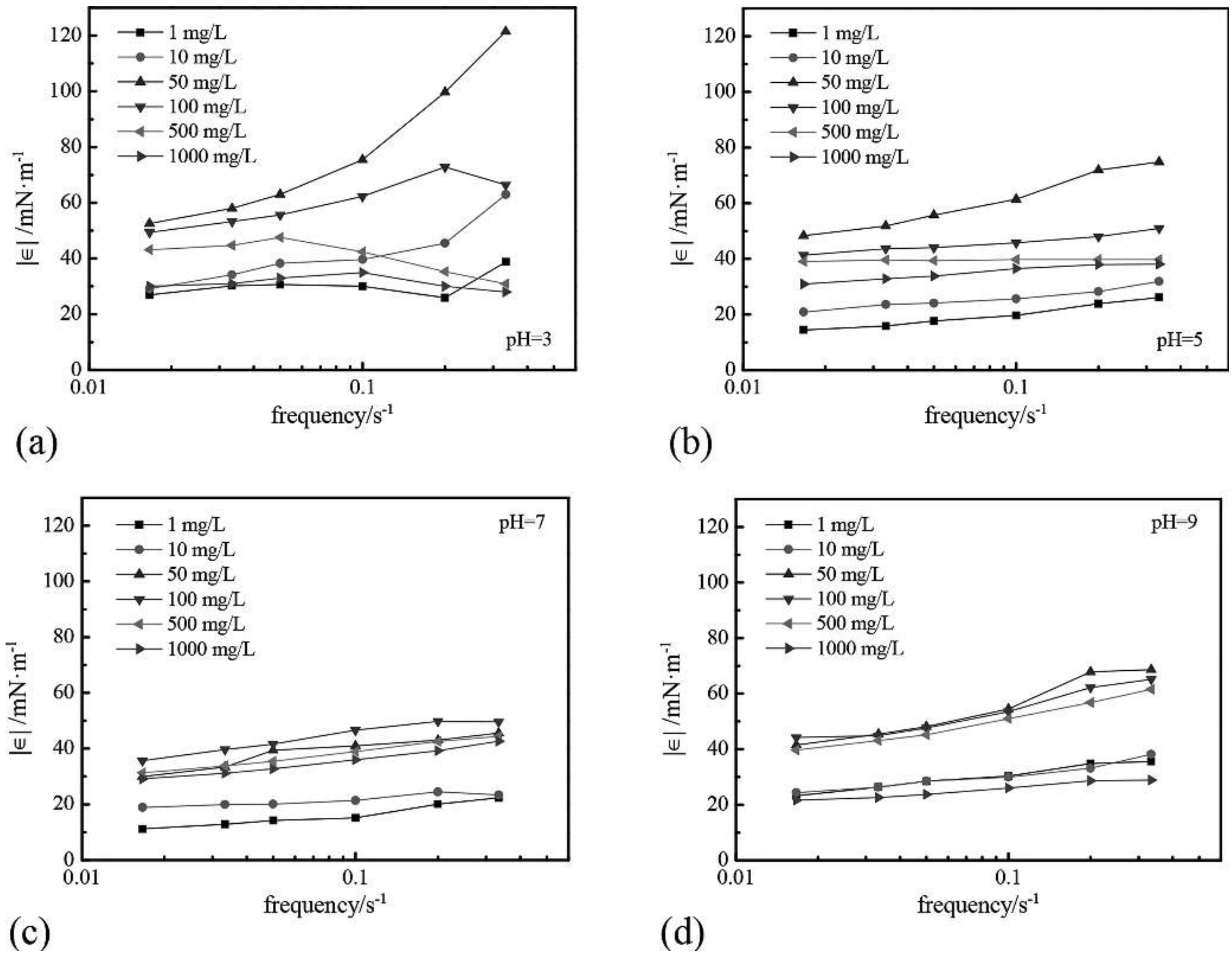

Figure 3 Variation of dilatational modulus as a function of frequency at pH of (a) 3, (b) 5, (c) 7 and (d) 9

ple with 1 mg · L^{-1} surfactant has a relatively small dilatational modulus. As the surfactant concentration keeps constant, pH value has a remarkable influence on the dilatational modulus. To observe well the change of dilatational modulus with concentration and pH value, the variation at a fixed frequency is shown in Fig. 4.

The dilatational modulus increases gradually with the surfactant concentration followed by the maximum and then decreases. Generally speaking, an increase of surfactant concentration in the bulk solution can affect the interfacial dilatational viscoelasticity. Furthermore, a rising concentration in surface layer enhances the ability of the surfactant molecules diffusing from the bulk to a subsurface. The dilatational elasticity becomes strong with an increase of concentration, while the diffusion of surfactant molecules from the bulk to the surface decreases the dilatational elasticity [26, 27]. An equilibrium state is finally achieved, and the modulus reaches the maximum value as the surfactant concentration increases to a certain value.

From Fig. 4, the dilatational modulus is strongly affected by the pH value. Among all these target systems, the one with pH 3 has the highest modulus while the one with pH 7 is the lowest. Compared with the neutral system, the maximum of the dilatational modulus appears at a relatively low concentration (50 mg · L^{-1} in the acid and alkaline systems). Furthermore, the maximum value is highest in acid systems (pH = 3, 5) and lowest in neutral system (pH = 7) which is around 44 mN · m^{-1}. Among all the target systems, this value is relatively high at the pH value of 3, reaching 76 mN · m^{-1}. After adding hydrochloric acid to modify the pH value of the W-5 solution, the amines are neutralized and form chlorohydrates that are more water-soluble. Amine chlorohydrates are the effective emulsifying species. Ionized and polar groups are essential moieties for the adsorption on the bitumen/water interface. As amines have electric charges, they are prone to adsorb at the interface, especially for the ones with positive charges.

The elastic and viscous moduli are also measured, which present a similar tendency in variation as shown in Fig. 5. The moduli are also reliant on to the concentration of emulsifier and frequency. Most of the values increase with the frequency. When the frequency is high, it decreases slowly with increasing concentration, which implies that the surface deformation may play a dominant role in deciding the dilatational viscosity. Obviously, the elastic modulus is much higher than the viscous modulus, close to the value of dilatational modulus, which indicates that the elastic character is dominant at the oil/water interface.

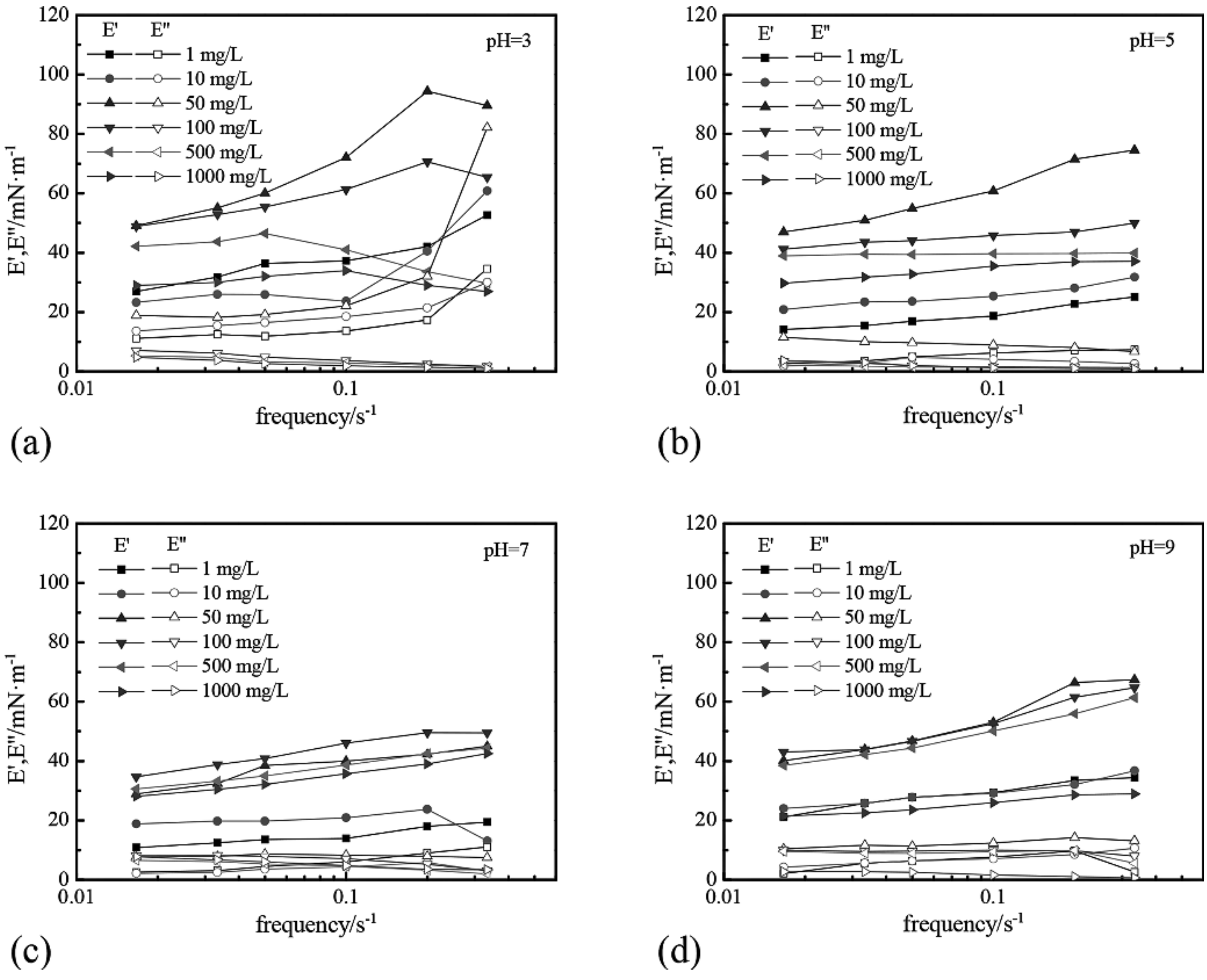

Figure 5 Variation of the elastic (E′) and viscous (E″) moduli as a function of frequency at pH of (a) 3, (b) 5, (c) 7 and (d) 9

As shown in Fig. 6, these curves increase firstly and then decrease with the concentration. Especially for the elastic modulus, it shows the same variation with the concentration as for the dilatational modulus, increasing up to a maximum value at a given frequency. As it has been stated for dilatational modulus, at low and high concentrations the increasing surface concentration and the surfactant molecular exchange play a dominant role in determining the dilatational elasticity.

3.2 Emulsion stability

The stability of bitumen emulsion can also be defined as the resistance of the dispersed droplets to coalescence. In the target bitumen emulsions, a double layer of ions and counter-ions exists in emulsion surrounding each dispersed asphalt particle. This double layer affects the stability of the emulsion system, of which strength can be evaluated by Zeta potential. In most cases, a large Zeta potential indicates a good stability of emulsion. Measurement of the storage stability of bitumen emulsion is a direct way to present the stability of emulsion. Low value is related to a favorable storage stability. Zeta potentials and storage stability are shown in Fig. 7. The value of the storage stability increases with the pH value and reaches a peak at the value of 7, while the change of Zeta potential is in the opposite trend. As the pH value is 3, the bitumen emulsion presents the lowest storage stability and the highest Zeta potential, indicating favorable emulsion stability. The emulsion stability is poor at the pH value of 7. Based on the results of interfacial rheology, the dilatational modulus is the highest at the pH 3 and the lowest at the pH 7, which shows good agreement with above results of emulsion stability.

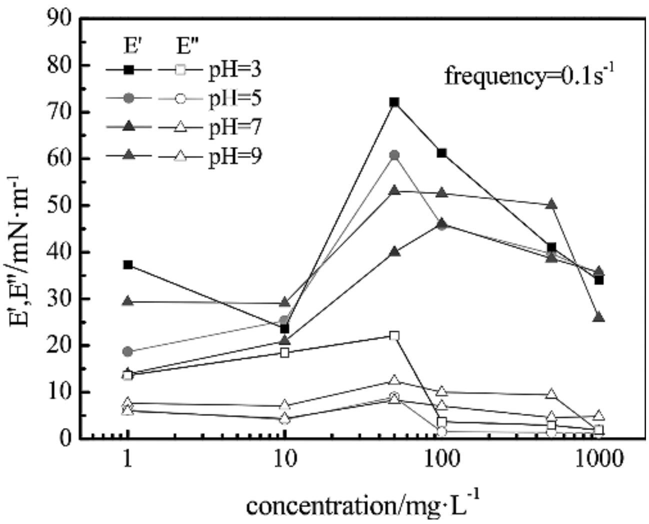

Figure 6 Variation of the elastic (E′) and viscous (E″) moduli as a function of emulsifier concentration at a fixed frequency of 0.1 s^{-1}

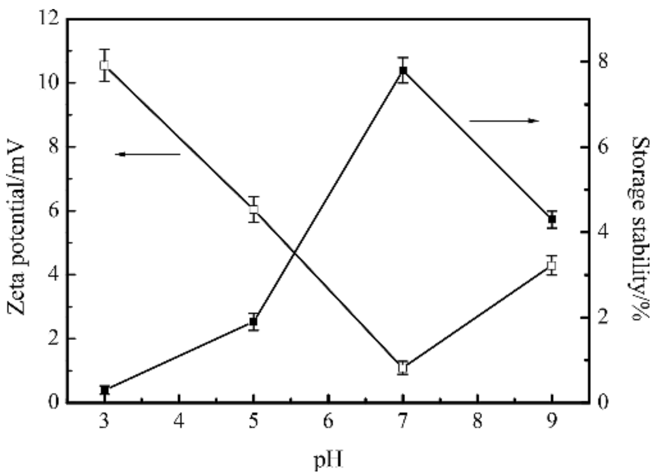

Figure 7 Zeta potentials and storage stability of asphalt emulsions

4 Conclusions

In order to investigate the pH effect on the interfacial properties of a cationic bitumen emulsifier, a wide range of pH values were considered, including acid, neutral and alkaline environment. The results show that the pH has a strong influence on the surface activities of bitumen emulsifiers at the bitumen/water interface. The interfacial tension decreases with the increase of pH value. The dilatational modulus is relatively high in acid and alkaline systems, especially at the pH 3. The amine moiety of bitumen emulsifier forms chlorohydrates as the pH is reduced by hydrochloric acid, thus the emulsifier is more water-soluble and has a high emulsifying ability. As amines have electric charges, it is easy for them to adsorb at the interface, especially for the ones with positive charges. The physical properties of bitumen emulsions are also affected by the pH value, which shows the same changing rule as the interfacial rheology. At the pH 3, storage stability is the lowest and Zeta potential shows a high value, indicating a favorable storage stability of bitumen emulsions.

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