



Effect of salt concentration on the phase separation of bitumen emulsions



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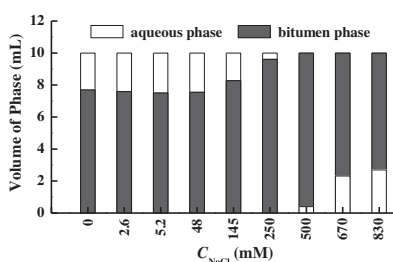
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HIGHLIGHTS

- ▶ Different phase separation situations are observed at various salt concentrations.
- ▶ Salt screening effect is responsible for the increased phase separation.
- ▶ Density changes of aqueous phase are more crucial at high salt amounts.

GRAPHICAL ABSTRACT



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ABSTRACT

This work has studied pronouncedly different phase separation situations for bitumen emulsions at various salt concentrations. With increasing sodium chloride concentration (C_{NaCl}), the added salt promotes the degree of phase separation of bitumen emulsions at C_{NaCl} lower than 5.2 mM, where the separated water is the upper phase and the bitumen is the lower phase. Nevertheless, this kind of phase separation is gradually reduced as C_{NaCl} increases from 5.2 to 250 mM. At $C_{NaCl} = 250$ –500 mM, the bitumen emulsions with very low degree of phase separation are observed to transit from the aqueous phase in the upper position and the bitumen phase in the lower level into the reverse situation. Further increase of salt amount in bitumen emulsions leads to the enhanced phase separation at $C_{NaCl} = 500$ –830 mM. These results of phase separation of bitumen emulsions are explained as the combined influences of the screening effect on the electrostatic repulsion of the bitumen droplets and the density changes of the aqueous phase induced by the added salt. While the salt screening effect is responsible for the increased phase separation at C_{NaCl} smaller than 5.2 mM, other phase separation behaviors at higher salt amounts may be mainly attributed to the increasing density values of the aqueous phase due to the salt addition.

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1. Introduction

Bitumen, as a complex mixture of different molecular weight hydrocarbons and nonmetal derivatives, is widely used for road construction to bind with the mineral aggregates together to make up the layers of pavement [1,2]. Due to its extremely high viscosity, pure bitumen is very difficult to manipulate at normal

temperature [3]. Bitumen has to be heated to high temperature usually above 130 °C to get sufficient fluidity. Instead, bitumen emulsions are prepared as a convenient way of converting bitumen into a workable and effective binder for road maintenance products. But bitumen emulsions are thermodynamically unstable system, which can break down over time through a variety of physicochemical instability mechanisms, such as gravitational separation, coalescence, flocculation, inversion, and Ostwald ripening [4,5]. Thus, one important requirement of bitumen emulsions is very good stability during storage and long distance transportation. Nevertheless, bitumen emulsions are designed to break quickly

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after the application to the mineral aggregates for the establishment of the final pavement performance [6,7]. In order to design and manufacture bitumen emulsions that are capable of exhibiting above opposite properties, it is necessary to deeply understand the factors that determine the phase behaviors of bitumen emulsions.

Because the mineral aggregates in road construction such as cement and sand generally carry negative charges, cationic surfactants like quaternary ammonium compounds are frequently used to emulsify the mixture of bitumen and water [8,9]. The cationic surfactant molecules may adsorb on the surfaces of dispersed bitumen droplets to form a positively charged surfactant film, which is responsible for controlling the stability of emulsion systems due to the electrostatic repulsion of the bitumen droplets. Indeed, the electrostatic factor like salt addition has been demonstrated to have an important effect on the bitumen emulsions. The added salt may partly neutralize the charge of surfactant film at bitumen/water interface and reduce the electrostatic repulsion of bitumen droplets, leading the bitumen emulsions to become unstable. Therefore, it was previously reported that the demulsification rate and separation efficiency of bitumen emulsions were increased with the salt additions [7,10]. As the water content decreases in the breaking of bitumen emulsions mixed with the mineral aggregates, the salt concentration of bitumen emulsions increases and further reduces the electrical repulsion forces. Meanwhile, various kinds of salt elements such as Ca, Si, Al, Fe, Na, K, and Mg are contained in the components of mineral aggregates, which also greatly influence the breaking of bitumen emulsions [11]. Although the salt effects on the physicochemical properties of common emulsion systems have been fully investigated for a long time, the study of effects of salts on the emulsions containing especially high sticky substances like bitumen as the dispersed phase is very little. Thus, the systematic investigations of salt effects on the phase behaviors of bitumen emulsions are of great importance for the control of the stability and break speed of bitumen emulsions to fulfill different requirements.

The objective of this work is to understand the effect of salt concentration on the phase separation of bitumen emulsions. A series of bitumen emulsions are prepared at varying sodium chloride concentrations (C_{NaCl}) and fixed amounts of three components of bitumen as the dispersed phase, water as the continuous phase, and a cationic surfactant octadecyltrimethylammonium chloride (OTAC) as the emulsifier. The phase boundaries of bitumen emulsions with various salt amounts were first determined. The zeta potential, density, viscosity, and micropolarity of bitumen emulsions were then measured for understanding the dramatically different phase separation behaviors of bitumen emulsions observed at various salt concentrations.

2. Experimental

2.1. Materials

SK bitumen was obtained from Korea. Quaternary ammonium surfactant octadecyltrimethylammonium chloride (OTAC) was purchased from Sinopharm Chemical Reagent Co. Sodium chloride (NaCl) of analytical grade was purchased from Lingfeng Chemical Reagent Co and water was deionized water.

2.2. Bitumen emulsion preparation

Prior to the emulsification process, bitumen was heated in an oven at 150 °C for 2 h. The aqueous phase, which is made of mixture of octadecyltrimethylammonium chloride (OTAC) and deionized water, was immersed in a 65 °C water bath for 15 min. The melted bitumen was added gradually into the surfactant solution under the homogenization using a high-speed homogenizer (FA25, FLUKO)

for 10 min. Under magnetic stirring, the homogenized bitumen emulsion was completely mixed with NaCl solutions for desired sodium chloride concentrations (C_{NaCl}). In the final bitumen emulsions, the compositions of bitumen and OTAC were fixed at 60 wt% and 0.5 wt% respectively, but varying salt concentrations.

2.3. Phase boundary determination

The influences of salt concentration on the phase separation of bitumen emulsions were studied at C_{NaCl} ranging from 0 to 830 mM. The prepared bitumen emulsions of various salt concentrations were placed in the test tubes stored in the thermostated water bath at 25 °C for 5 days to reach phase equilibrium. The volumes of the aqueous phase and the bitumen phase were then recorded. The phase separation index of bitumen emulsions was defined as the ratio of the volume of the aqueous phase to the total volume of bitumen emulsion.

2.4. Zeta potential measurement of bitumen emulsions

The zeta potential of bitumen emulsions was determined according to the procedure reported in the literature [1]. As the bitumen emulsions being too concentrated, it was necessary to centrifuge them to measure the zeta potential of the aqueous phase. The bitumen emulsions were centrifuged for 30 min at 12,000 rpm. The zeta potentials of the separated aqueous phase were measured using a Malvern Zetasizer Nano ZS (Malvern Instruments, London, England). The zeta potential was calculated on the basis of the Smoluchowski relation of the ionic mobility with the surface charge.

2.5. Density measurement of the bitumen phase and the aqueous phase

The density of the bitumen phase of bitumen emulsions was estimated as the density of the bitumen used for emulsion preparation. The bitumen with known quality was heated to melt and then poured into the test tube. The ratio of the quality to the volume of the tested bitumen was recorded as the density of the bitumen phase. Because the added NaCl was mainly dissolved in the aqueous phase of bitumen emulsions, it is expected that the density of the bitumen phase remains constant value at different salt concentrations. Meanwhile, the density of the aqueous phase of bitumen emulsions was approximately measured as the density of a series of NaCl solutions using a density meter (DMA-5000M, Anton Paar) at 25 °C.

2.6. Viscosity measurement of bitumen emulsions

The viscosities of freshly prepared bitumen emulsions containing various salt concentrations were measured using a ULTRA Programmable Rheometer (DV-III) provided with rotor type SC4-18. Viscosity data of bitumen emulsions were acquired at 25 °C with circulating water.

2.7. Micropolarity measurement of the aqueous phase

The micropolarity of the aqueous phase of bitumen emulsions was determined from steady-state fluorescence measurement of pyrene polarity index (I_1/I_3) using an Edinburgh FLS900 spectrofluorophotometer equipped with a water-circulating bath at 25 °C. I_1/I_3 is the ratio of the intensities of the first and the third vibronic peaks in the fluorescence emission spectrum due to pyrene. Pyrene was excited at 337 nm and the emission spectra were scanned from 350 to 500 nm.

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