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# THE EFFECT OF PH AND BITUMINOUS DENSITY ON STABILIZATION OF BITUMEN-IN-WATER EMULSION

# S. Hassan Firoozifar<sup>\*</sup>, Sanaz Foroutan, Sara Foroutan

# Research Department, Pasargad Oil Company, Tehran, 1879913111, IRAN, Email: h-firoozifar@pasargadoil.com

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#### Abstract

Understanding the stability mechanism of bitumen-in-water emulsion directly affects its transport, storage and usage. In order to have a bitumen emulsion with high stability which covers all the necessary requirements, we had focused our attention to the methods for stabilizing against settlement and creaming. Therefore, the stability of several emulsion prepared with different amount of solvent in the bituminous phase were investigated at different pH. All the necessary tests were conducted on the samples to achieve the optimum condition in producing complete stable emulsions. Finally, we found that acidity of the water phase and density of the bituminous phase, which can be adjusted by addition of suitable solvents, are so important in emulsion stability.

Key Words: Emulsion; Bitumen; Stabilization; Creaming; Density.

#### 1. Introduction

Stabilized bitumen emulsion which could be kept for a long period of time has been one of investigators interests in the past decade. Unfortunately, it is not so easy to do so, because emulsions are unstable systems and break by many factors such as coalescence, flocculation and so on <sup>[1-3]</sup>. These factors could change the size of droplets. Therefore having an exact formula in producing bitumen emulsion is so important in achieving long-term stability.

In concentrated emulsions dispersed particles of bitumen are so close to each other and there is only a thin film of continuous phase between them <sup>[4, 5]</sup>. These particles are separated by each other by electrostatic repulsion. The presence of surfactants at the surface of these particles generates great repulsion force and improves the stability of the emulsion <sup>[6]</sup>.

One method to increase the stability of bitumen emulsion is to decrease the viscosity of bitumen (converting hard particle to softer one) by adding a suitable solvent to the bitumen phase. Solvent can be used to give a temporary softening to the binder. The bitumen will then have an improved coating ability to the aggregate. There are two things to be considered in choosing a suitable solvent. First of all, the solvent has to be compatible with the bitumen, i.e. the mixture of bitumen and solvent should be homogeneous and stable. Second, the solvent has to be sufficiently volatile not to leave any residue in the bitumen which can give a lower viscosity. White spirit is the most widely used solvent.

Emulsification process is opposed by the internal cohesion and viscosity of the bitumen, and the surface tension which resists the creation of new interface. The interfacial area between the liquid phases is greatly increased in an emulsion. One liter of bitumen emulsion may have an interfacial area of 5000 m<sup>2</sup> <sup>[7]</sup>. Emulsions are thermodynamically unstable systems which, given the opportunity, would phase separate through coalescence of the

dispersed droplets <sup>[8]</sup>. Once the droplets are formed, they must be stabilized against all kinds of destabilizing factors like creaming, phase inversion, flocculation and coalescence.

Therefore, to have a bitumen emulsion with high storage stability, we focused our attention to the methods of stabilizing emulsion against settlement and creaming by changing the water pH and bituminous density.

### 2. Experimental

The bitumen used in this work was supplied Passargad Oil Company (Table 1). Cationic emulsifier is made of ligninamin and alkylamin, which obtained from Iterchimica Srl. (Italy) and used as received without further purification. Other chemicals were obtained from Merck Co (Germany), and also used as received.

SPECIFICATION	Bitumen	TEST METHOD
Specific Gravity @ 25/25°C	1.0102	ASTM D 70
Penetration @ 25°C (dmm)	68	ASTM D 5
Softening Point (°C)	52	ASTM D 36
Ductility@ 25°C (cm)	+ 100	ASTM D 113
Flash Point (°C)	+ 250	ASTM D 92

Table 1: Properties of bitumen

# 2. 1. Emulsions Preparation

Bitumen emulsions were prepared using an Emulsion preparation Laboratory Unit "Emulab" (Breining Co., Germany) with the same amount of emulsifiers at different pH (1,1.3,1.5,1.7,2) and solvent contents (0,0.5,1,1.5,2 %). In a typical procedure, a specific amount of emulsifier was added to a beaker on hot plate equipped with magnetic stirrer which contains 2 liter of mineral water and heated to 45° C. pH was adjusted to a desired amount by addition of 37% HCl solution. On the other hand, bitumen was heated to 140° C in a separate container and the desired amount of solvent was added with care. These two phases were mixed with each other under high shear mixer of the unit to produce emulsion.

# 2. 2. Emulsions Characterization

#### 2. 2. 1. Settlement

500 ml of bitumen emulsion was kept in a glass cylinder at room temperature for 5 days. Then approximately 55 ml of the sample was removed from the top and the bottom of the emulsion by means of a pipette and  $50 \pm 0.1$  g of the emulsions were placed in two beakers. These beakers were put in an oven at 163° C for 3 h. Then they were removed from the oven and allow cooling down to room temperature, and weighted. The storage stability of an emulsion is defined as the differences between the percentage of residue from its top and bottom.

# 2.2.2. Engler viscosity

This test method covers the determination of specific viscosity of emulsion according to ASTM D1665. Engler viscosity is calculated by measuring the time for a specific volume of a liquid material (bitumen emulsion), to flow through a tube in a constant temperature.

# 2.2.3. Sieve Test

This test method measure the degree at which a bitumen emulsion may contain particles retained on 850-micrometer sieve according to ASTM D244. In typical procedure, 1 Kg of bitumen emulsion is poured on the mentioned sieve, wash gently with water and calculate the weight of solid materials which remains on the sieve.

#### 2.2.4. Cement mixing

This test method is used to identify the ability of a slow setting bitumen emulsion to mix with a finely divided, high surface area material (Type III, Portland cement) without breaking the emulsion according to ASTM D244.

#### 3. Results and discussion

Emulsions, particularly those having low bitumen content and low viscosity, are at risk to settlement. At ambient temperatures, the density of bitumen is slightly greater than that of the aqueous phase of the emulsion. Consequently, the bitumen particles tend to fall through the aqueous phase, resulting in a bitumen rich lower layer and a water rich upper layer.

Settlement can be reduced by equalizing the densities of the both phases. Because the coefficient of thermal expansion of bitumen and the aqueous phase are not the same, their densities can be made. As it is shown in Figure 1, increasing the solvent content, decrease the settlement of emulsion, to the less than 1% (ASTM D2397). Also, in all curves there is a minimum around pH=1.5 which is an indication of the highest stability and the least difference between the densities of bitumen and aqueous phase.

It is seen in Figure 2 that the size of emulsion particles and subsequently the residue of emulsion on the sieve, decrease by reducing pH. Also, according to Figure 2, all sieve test results are under 0.1% which is considered as a characterization of a suitable emulsion in ASTM D2397.





Figure 1 Effect of pH and solvent content on settlement

Figure 2 Effect of pH and solvent content on sieve test

If the viscosity of the bitumen is reduced, the particle size of the emulsion will be reduced too, which in turn, tends to increase the viscosity of the emulsion. In the case of cationic emulsions, viscosity can be increased by decreasing the acid content or increasing the emulsifier content. Effect of pH and changing the solvent content on the viscosity of emulsion is shown in Figure 3.

At high bitumen contents, the bitumen particles are more likely to come into contact with each other resulting in an increase in the rate of break. On the other hand, the smaller the size of the bitumen particles, the finer will the dispersion, resulting in a slower breaking rate. The breaking rate of a bitumen emulsion is increased by reducing the acid, and the ratio between acid and emulsifier contents, and increasing the emulsifier content  $^{[9]}$ .





Figure 3 Effect of pH and solvent content on Figure 4 Effect of pH and solvent content on Engler viscosity

breaking index

As illustrated in figure 4, increasing the solvent content, decreases emulsion particle size and subsequent breaking rate. It is seen that addition of solvent causes the breaking rate decreased to less than 2% which is suitable for a standard emulsion, according to ASTM D2397.

#### 4. Conclusion

In conclusion, addition of solvent to the bituminous phase increases the stability of bitumen emulsion by decreasing the difference between densities of two phases. Also, we find that all emulsions were more stable at pH around 1.5. In addition, use of solvent leads to an emulsion with smaller and uniform particle size which cause less flocculation and then more stability. Furthermore, solvent decreases the breaking rate of emulsion on the aggregates which is a considerable property in some applications like prime coat.

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