Effect of Processing Parameters on Properties of Bitumen Emulsions

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Abstract. Bitumen emulsions are special type of paving binder materials used in road construction industry due to its environmentally friendly approach, cost efficiency and less energy consumption compared to other conventional bitumen binders. Despite so many benefits, the use of bitumen emulsions is still limited in many regions due to difficulties faced in its emulsification process. Controlling several process parameters are needed during formulation while achieving the sufficient storage stability and viscosity. Therefore, this study was conducted to investigate the effect of different processing parameters on the properties of bitumen emulsions in terms of storage stability and viscosity. The 80/100 grade bitumen was emulsified through colloid mill, considering different pH values and emulsifier concentrations as processing parameters during emulsification process. The results indicate that the storage stability has improved significantly with increased concentration of emulsifier whereas high acidic system in terms of pH level has also substantially enhanced storage stability at lower level of emulsifier concentration. In addition, the viscosity of bitumen emulsions has also been affected by the emulsifier concentration and higher amount of emulsifier resulted in higher values of viscosity at lower pH level. The data achieved through this investigation can be further used to study the effect of other formulation parameters on properties of bitumen emulsions.

1. Introduction

Bitumen emulsions are prioritized over a conventional asphalt binders to be used in a road maintenance applications due to its cost efficiency, environmental advantages and lower energy consumption [1,2]. The production of bitumen emulsion is a technical task, since several processing parameters need to be controlled and optimized during the emulsification process [3]. The parameters include bitumen/water ratio, emulsifiers concentration, temperature of both soap solution and disperse phase (bitumen), pH value, speed of mixing shear; these processing parameters directly or indirectly influence the physical and rheological properties of bitumen emulsions [4]. The basic physical and rheological properties of bitumen emulsions are its storage stability, viscosity, mean droplet size distribution and breakage behaviour [5]. The properties of bitumen emulsions dictate its suitability of particular types of emulsion in the field applications [6]. In addition, the interfacial tension on bitumen/water interfaces caused by emulsifier activity is affected by acidic or alkaline levels in terms of pH value. Since the interfacial tension among bitumen/water droplets decreases with the increased value of pH. The emulsifier has high emulsifying ability at high pH value and makes the dilation modulus high at particular value of pH

around 2-3, particularly for cationic type of emulsions which results in a more favourable storage stability [7,8].

Despite affecting stability of emulsions, the pH value also affects the rheology of emulsions. The viscosity of bitumen emulsions increases with increased amount of acid and alkaline under extra shear provided [9]. The viscosity of bitumen emulsion is also influenced by mixing speed and time, droplet size & distribution and emulsifier concentration [10]. However, the flow characteristics such as viscosity of emulsion plays significant role in applications of bitumen emulsions since the emulsions viscosity neither should be very low to flow from aggregates nor be more viscous for mixing and workability purposes [11].

The physical properties of bitumen emulsions are also affected by its droplet size and distribution. The size of emulsion droplets increases with increased volume of bitumen fraction, however, the droplets size decreases with increased of rotor speed, since small droplets result in higher viscosity [12]. The influence of mechanical parameters on storage stability also extends to the emulsifier type and penetration grade of bitumen which has significant effect on storage stability of bitumen emulsions [13]. Since it becomes necessary to study the processing parameters during the emulsification of bitumen and its effect on the properties of bitumen emulsions. The influence of different pH values and emulsifier concentration on properties of bitumen emulsions such as stability and viscosity are investigated in this study.

2. Experimental procedure

2.1. Materials

A base bitumen with a penetration grade of 80/100 was used in the emulsification and the physical properties are listed in table 1. The cationic slow & medium set emulsifier Redicote N39L was used in the emulsification of bitumen, used as received. It is designed to produce slow and medium set emulsions based on its concentration. The acid, hydrochloric (HCL) was employed to adjust the pH of soap solution and pH was measured by litmus paper. Since the deionized water was used for preparing soap solution. The colloid mill with shearing speed of 2840rpm was used to formulate the emulsions as shown in the figure 1.

Table 1. Physical properties of bitumen binder.

Penetration @ 25°C,100g, 5 Sec	Softening Point °C	Flash Point °C	Ductility @ 25°C 5 cm / min	Specific Gravity @25°C
80-100	43-54	232	100	1.01-1.06

2.2. Preparation of bitumen emulsions

The soap solution produced at temperature 60± 3°C by introducing different dosage such as 0.5%, 1% and 1.5 % of emulsifier by weight of aqueous phase at different pH 2, 4 and 6 values. The soap solution was poured followed by heated base bitumen up to 150± 3°C in a Colloid Mill, preparing oil in water 60:40 emulsions and mixed for 5 minutes as shown in the Figure 1 (a) & (b). The samples of bitumen emulsions were collected at the outlet of colloid mill and stored in sealed containers and then left to cool at room temperature. The emulsions were then tested for surface charge through particle charge test as per ASTM D7402 as shown in Figure 2 (a) [14]. The values of different parameters considered in the emulsification process are given in table 2. The emulsification process was repeated three times for each sample in order to avoid error in the results.

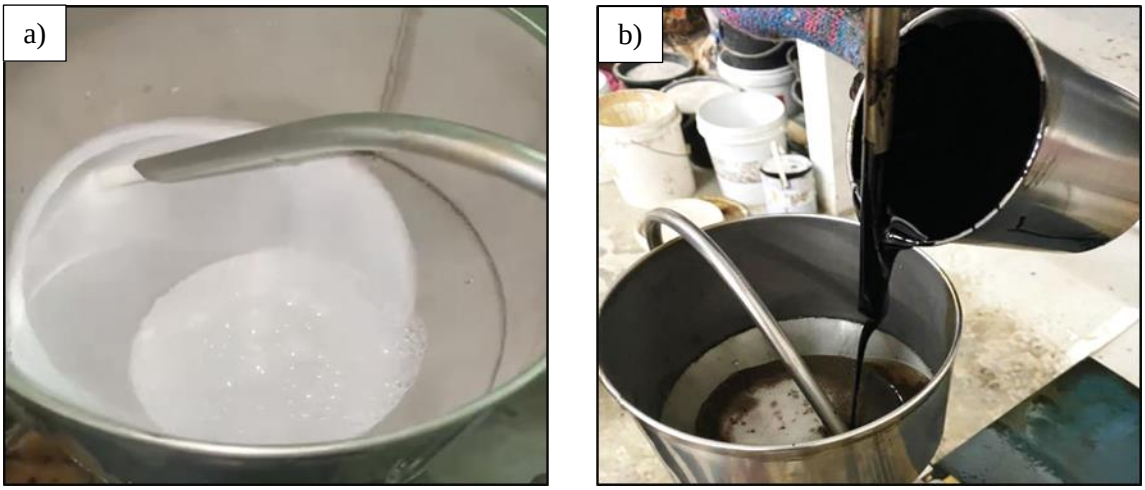

Figure 1. (a) Soap Solution in Colloid Mill and (b) Pouring bitumen binder in Colloid Mill.

Table 2. Selected Parameters during the emulsification process.

Emulsifier (%)	pH values	Mixing Time(mins)
0.5	2	5
1	4	5
1.5	6	5
0.5	2	5
1	4	5
1.5	6	5
0.5	2	5
1	4	5
1.5	6	5

2.3. Measurement of storage stability

The one-day storage stability of prepared samples was measured as per standard method of ASTM D6930 [12]. It is determined by the difference in percent of bitumen residue by evaporation of bitumen emulsions samples collected from the top and bottom from undisturbed samples placed in the cylinder for 24 hours shown in Figure 2 (b). The 50g samples from top and bottom each were collected in beakers and placed in an oven (temperature 163± 3°C) for 2 hours. Then the samples were stirred and kept for another 1 hour in the oven in order to get bitumen residues. The result for storage stability should be ≤ 1 and least value gives favourable storage stability.

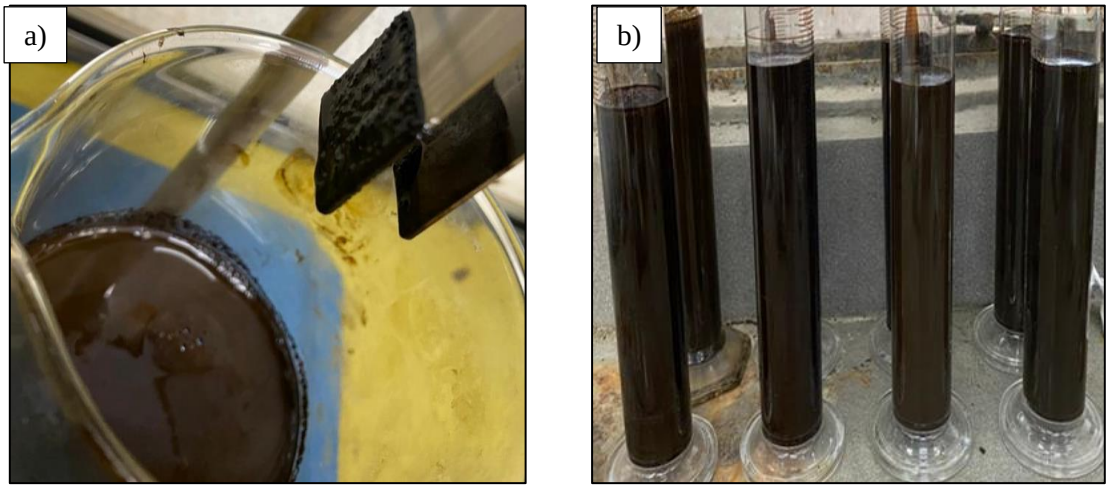

Figure 2. (a) Particle charge of emulsions; (b) Samples stored in cylinders for 1day storage stability.

2.4. Measurement of viscosity

The flow characteristic of bitumen emulsions affects its utility during the application. The bitumen emulsion should be viscous enough to be applied uniformly and resist flow from the crown of road pavement. The Saybolt Fural viscometer was used to measure the viscosity (SFS) of prepared emulsions at 25℃ as per standard method of ASTM D7496 [15]. SFS is the efflux time in seconds of 60 mL of bitumen emulsions flowing through a calibrated Furol orifice at specified temperature as shown in Figure 3 (a) & (b). The SFS was measure at 0.5%, 1% and 1.5% emulsifier concentration to examine its influence.

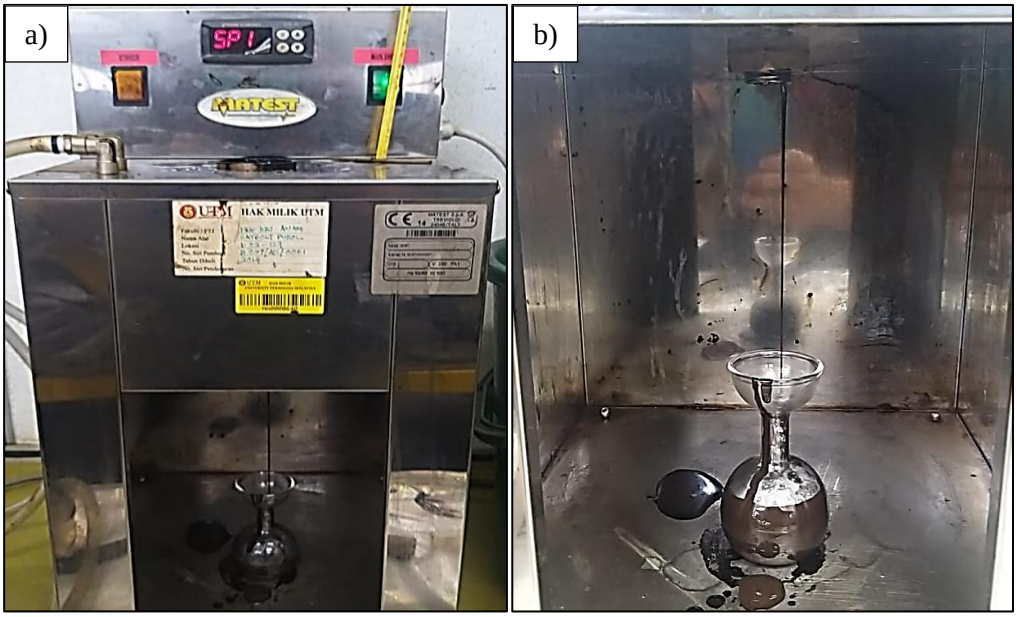

Figure 3. (a) Saybolt Furol Viscometer; (b) Bitumen emulsions through Furol orifice.

3. Results and Discussion

3.1. Effect of pH values on storage stability of bitumen emulsions

The pH creates an environment for emulsifier to exhibit better emulsifying ability in bitumen emulsion system. Since this particular value of pH needs to be optimized during emulsification process [9]. The emulsifier becomes soluble in water at high acidic levels, thus resulting in polarizing the positively heads of cationic molecules [3]. In addition, the high pH value increases the dilation modulus of the emulsifier and result in better stability of emulsions. However, it is shown (see Figure 4.) that the values for storage stability increases with increased pH values linearly. The emulsions become unstable above value of pH 4 since the emulsifier is unable to emulsify the bitumen at less acidic system. The pH values 2-3 give significant results in terms of storage stability where the values for storage stability 0-0.4 are more favourable. The size of emulsifier charge on bitumen droplet is strongly pH dependent, thus strong pH exhibit high repulsion of droplets and result in a uniform suspension of droplets [11].

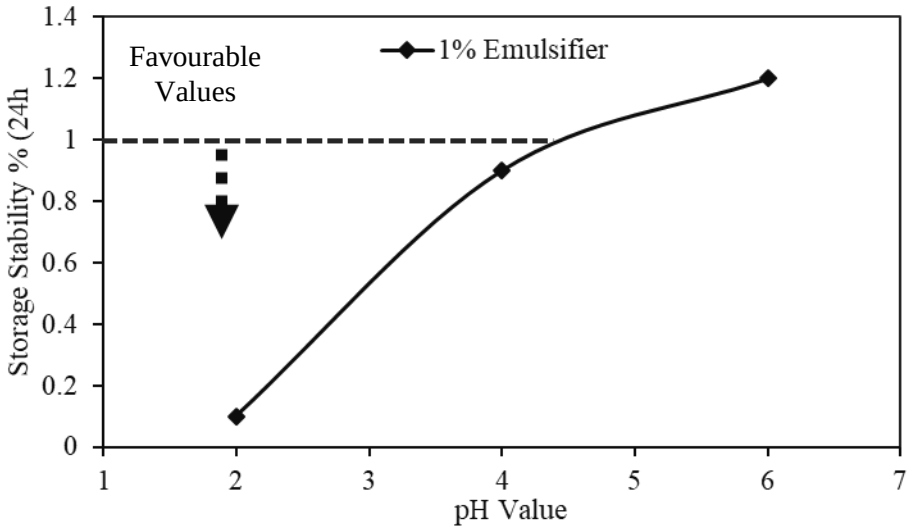

Figure 4. Effect of pH values on storage stability of bitumen emulsions.

3.2. Effect of emulsifier on storage stability of bitumen emulsions

The emulsifier reduces the interfacial tension on bitumen/water interfaces, resulting in a stable droplets suspension of bitumen and water. The emulsifier molecules are adsorbed on liquid-liquid interface on emulsion droplets and exhibit significant repulsion and suspension in the system [4]. Thus the storage stability can be defined as the resistance to coalescence of disperse droplets in emulsion system [7]. Since least values for storage stability ≤ 1 are favourable in bitumen emulsion stability. The results show (see Figure 5) that the values for storage stability decrease with increased percentage of emulsifier in the soap solution. However, the emulsifier dosage from 1% to 1.5% increment doesn't show any significant change in the results of storage stability since the storage stability for both concentrations remained unchanged. Despite, providing higher concentration of emulsifier up to 1.5%, it is feasible to provide emulsifier percentage of only 1%, resulting in saved quantity of emulsifier in terms of economy**.**

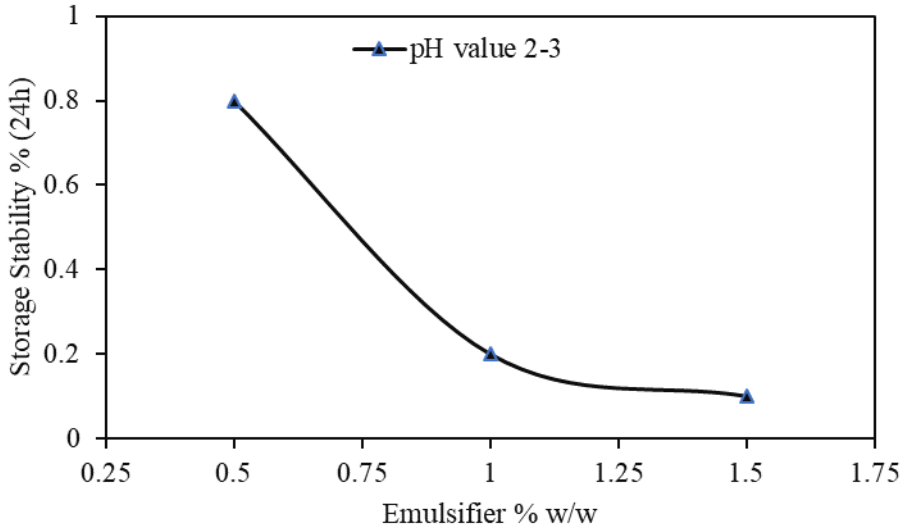

Figure 5. Effect of emulsifier % w/w on storage stability of bitumen emulsions.

3.3. Effect of emulsifier on viscosity of bitumen emulsions

The rheology of bitumen emulsions is identified for the purpose of its particular applications, although it doesn't affect the end performance, but emulsions with high viscosity are suitable for gape graded and low viscosity for dense graded mixture [10]. However, the viscosity of bitumen emulsion is generally influenced by several parameters such as emulsifier concentration, droplet size and distribution, shearing speed and viscosity of dispersion phase [4,5]. In addition, the emulsions with lower viscosity and small droplets size are more stable [16]. On contrary the stability of bitumen emulsions is very high in the existence of favorable amount of emulsifier concentration and least pH [17]. This can also be seen (see Figure 6) that the viscosity values increase with increased amount of emulsifier. Since higher amount of emulsifier produces strong charge on the surfaces of emulsion droplets, which also result in smaller size of droplet sizes. This leads to higher viscosity of bitumen emulsions [18].

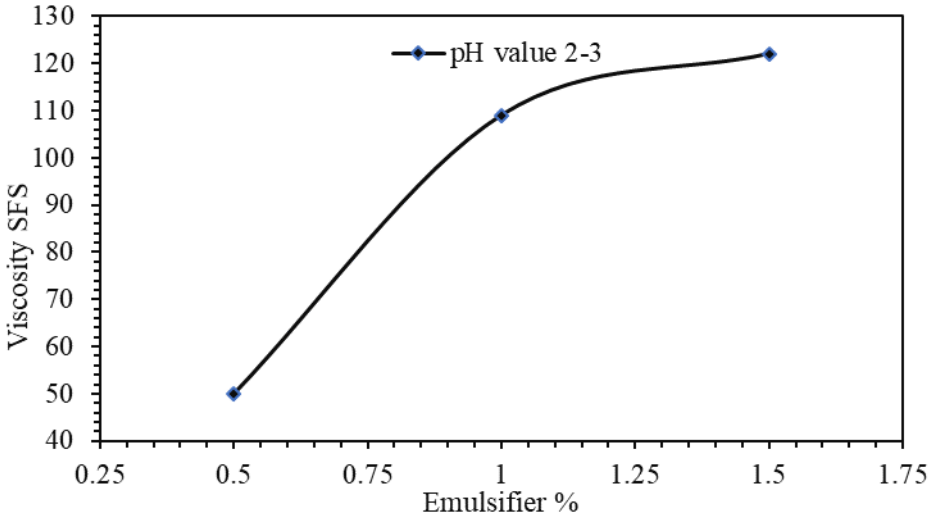

Figure 6. Effect of emulsifier % w/w on viscosity (SFS) of bitumen emulsions.

3.4. Effect of optimum pH value and emulsifier concentration on properties of bitumen emulsions

The parameters such as pH value and emulsifier concentration mutually play significant role in improving storage stability of formulated bitumen emulsions, since these both parameters are the function of Zeta potential which demonstrates the power of bitumen emulsion colloidal system in the presence of electrical charges [11]. However, these both variables in formulation are optimized as per required applications. The promising results of storage stability from (see figure 3) at pH value 4 or lower were at the existence of 1% emulsifier. While the results of storage stability at 1-1.5% emulsifier concentration in (see figure 4) were almost similar at pH value 2-3. Despite using higher amount of emulsifier, the optimum concentration should be used keeping the emulsification process of bitumen emulsions economical since emulsifier is an expensive material [4]. Hence, the bitumen emulsions can be formulated at optimum emulsifier concentration of 1% and pH values 2-3 resulting in a maximum storage stability. In addition, the interfacial viscosity of bitumen emulsion is associated with different formulation parameters, pH value and emulsifier concentration are two of them. However, the viscosity is optioned based on application specifications, since bitumen emulsions of high viscosity are more stable, this statement can be validated from Figure 3, 4 and 5 where bitumen emulsion with favorable stability are more viscous. The significant values of stability and viscosity of bitumen emulsions under the parameters such as pH value and emulsifier concentration are enlisted in Table 3.

Table 3. Optimized Values of bitumen emulsions.

Parameters		Properties	
pH Value	Emulsifier (%)	Stability (≤1)	Viscosity (SFS)
2-3	1	0.2-0.4	109

4. Conclusions

The objective of this study was to evaluate the influence of processing parameters on the characteristics of emulsified bitumen. The results indicate that the processing variables such as pH value and concentration of emulsifier have significant effect on the properties of bitumen emulsions such as storage stability and viscosity. The dependance shows that the storage stability improved at least pH value 2-3 producing high acidic level. However, the higher value of pH has negative impact on stability since stability values decreased with increased value of pH.

The results for emulsifier investigation show that the stability increases with increased amount of emulsifier since emulsifier works in the presence of strong pH values of soap solution. Thus, the emulsifier concentration of 1% at pH value 2-3 show significant results in terms of storage stability of 0.1%.

Although, the viscosity of bitumen emulsions is also influenced by mixing time and bitumen-water concentration, but this study only considered the effect of emulsifier concentration at pH of 2-3. However, the results show that the viscosity increases with increased emulsifier concentration since higher dosage of emulsifier results small droplet size and distribution which exhibit strong colloidal system of higher viscosity. Moreover, the choice of viscosity is based on application specifications of bitumen emulsions. This study also proposes to consider other mixing times for emulsification in future research in order to relate it with physical and rheological properties of bitumen emulsions.

5. References

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