Studying the effect of emulsified asphalt type on cold emulsified asphalt mixtures properties

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Abstract. Recently, due to environmental restrictions and economics, cold emulsified asphalt mixtures have become the dominant binder material used in cold mix applications. Cold emulsified asphalt mixtures are generally mixtures made using different types of emulsified asphalt with a range of aggregate materials. This research aims to test the physical properties of two types of emulsified asphalt binder (cationic and anionic) used as binders for paving mixtures to evaluate the properties of the resulting cold emulsified asphalt mixtures, using one type of quartz aggregate with different variables such as mixing procedures and compaction effort (75 or 150 blows). The evaluation includes the volumetric and mechanical properties of emulsified asphalt mixture such as bulk density, air voids, and Marshall properties, and compares these with the common local specifications. The results show that the quartz aggregate is more suitable for the cationic emulsified asphalt mixture, due to its negative charge and chemical composition which produce a good bond with a positive charge for cationic emulsified asphalt. However, the results obtained from a test of Marshall stability suggest maximum Marshall stability ratings for mixtures of cationic emulsified asphalt were greater than those for mixes with anionic emulsified asphalt mixtures. The stability values were 16.8 and 10.87, kN respectively, and the optimum emulsified content was 6.36%.

Keywords: Emulsified asphalt, Mix Design, Marshall Stability Test.

1. Introduction
Liquid asphalt is obtained by decreasing straight run asphalt viscosity in many different ways. One of these is emulsifying the asphalt in water in a process known as asphalt emulsification [1]. A cold emulsified asphalt mixture is generally a mixture made of emulsified asphalt mixed with aggregate. Emulsified asphalt is manufactured of base asphalt, an emulsifying agent, and water, with approximate percentages of 40% to 75% asphalt, 0.1% to 2.5% emulsifier, and 25% to 60% water, plus some other minor components. Asphalt emulsions are manufactured by using a colloidal mill to mix together the asphalt and water, which divides up and disperses the asphalt in the water phase. Cold mix asphalt concrete, or cold placed mixture, is generally made with emulsified asphalt or cutback asphalt, with the aggregate material being anything from a dense-graded crushed aggregate to a granular soil with a relatively high percentage of dust. Mixing may be performed either in the roadway, on the side of the roadway, or in a stationary mixing facility. The resultant mixtures are usually spread and compacted at atmospheric temperatures [2].

Recently, because of environmental restrictions and economics, emulsified asphalt has become the dominant binder material used in cold mix applications [3]. Cold asphalt mixtures are easy to mix and handle, while the size and time for installation process of their mixing plants are reduced compared to hot mix plants [4].

This research was designed to study the effects of two classes of emulsified asphalt materials on cold asphalt mixtures; these were evaluated by Marshall design and retained Marshall Stability. Two types of emulsified asphalt materials, cationic and anionic, one type of aggregate and one type of mineral filler were used, selected for their Marshall properties.

Experimental Procedure Materials Used
The type of aggregate used in the research was crushed quartz obtained from a local plant used for asphalt concrete mix, located in Al-Nibeay quarry. The aggregate properties were as shown in Table 1, and the aggregate gradation was as shown in Figure 1. These values are within the limits recommended by the specifications set out by the state commission of Roads and Bridges in Iraq (SCRB), to be used in surface course [5]. The type of mineral filler used was ordinary cement with the commercial name Al-mass; the specific gravity for this cement type filler was 3.13.

Table 1. Physical properties of Nibeay Aggregate

| Property                                      | ASTM Designation | Test Results | SCRB Specification |
|----------------------------------------------|------------------|--------------|--------------------|
| Coarse Aggregate:                           |                  |              |                    |
| 1. Bulk Specific Gravity                     | C-127            | 2.623        | ......              |
| 2. Apparent Specific Gravity                 | C-127            | 2.65         | ......              |
| 3. Water Absorption, %                       | C-127            | 0.45         | ......              |
| 4. Percent Wear by Los Angeles Abrasion, %   | C-131            | 16           | 35 Max             |
| 5. Soundness Loss By Sodium Sulfate Solution,% | C-88             | 3.21         | 10 Max             |

| Fine Aggregate:                              |                  |              |                    |
| 1. Bulk Specific Gravity                     | C-128            | 2.63         | ......              |
| 2. Apparent Specific Gravity                 | C-128            | 2.673        | ......              |
| 3. Water Absorption, %                       | C-128            | 0.5          | ......              |

Figure 1. Aggregate Gradation Distribution Chart.
Two types of emulsified asphalt, cationic slow setting low viscosity emulsified (CSS-1) and anionic slow setting low viscosity hard emulsified (SS-1H) were used throughout the research to produce dense mixtures. The results of the physical tests as determined by ASTM D244, D2397 and D977 are presented in Table 2 and 3, along with the specification limits [6]. Chemical composition of Quartz aggregate are shown in Table 4. The mixing procedure for both types emulsified asphalt with the aggregate materials mix was as follows: the coarse aggregate was first mixed with the emulsified asphalt; then, fine aggregate filler was introduced and mixed for a time ranging from 2 to 5 minutes. This gave better compatibility and led to a successful coating test, and this procedure is the simplest and most economical due to there being no need to dry the aggregate; hence, there is no need for water to be added to the mixture, which leads to adequate early strength requirements being met with a short curing period [7].

Table 2. Physical properties of asphalt emulation cations slow setting low viscosity (CSS1)
The above tests were done in the laboratories of the Building and Construction Engineering Department of the University of Technology

**Table 3.** Physical properties of anionic asphalt emulsion

| TEST                        | ASTM Designation | Test Result | ASTM SPECIFICATION |
|-----------------------------|------------------|-------------|-------------------|
| Particle Charge Test        | D244             | Negative    | Negative          |
| Viscosity Saybolt Furol at 25 °C(77°F) | D244            | 24          | 20 100            |
| Residue by Evaporation      | D6933            | 59.4        | 50 70             |
| Settlement Test 5day%       | D6933            | 0.9         | 0 1               |
| 1Day storage Stability test % | D2397          | 0.4         | 0 1               |

**Test on Residue**

| Penetration                 | D5               | 121         | 100 250           |
| Ductility 25 °C 77F cm/min | D113             | 41          | ......             |
| Density (gm./liter)         | D 6937           | 1014        |                   |

The above tests were done in the laboratories of the Building and Construction Engineering Department of the University of Technology

**Table 4.** Chemical composition of Quartz aggregate

| Chemical Compound                  | Content % |
|------------------------------------|-----------|
| Silica, SiO₂                       | 82.52     |
| Lime, CaO                          | 5.37      |
| Magnesia, MgO                       | 0.78      |
| Sulpharic Anhydride, SO₃            | 2.7       |
| Alumina, Al₂O₃                      | 0.48      |
| Ferric oxide, Fe₂O₃                 | 0.69      |
| Loss on ignition                    | 6.55      |
| Total                              | 99.09     |
2. Design of cold emulsified mixture
There is no universally accepted specification for cold emulsified asphalt mixtures mix design or testing. Mix design and test execution thus vary among road authorities, research institutions, and researchers in the field of asphalt [8]. In general, the design procedures adopted follow the following steps:
1. Determine aggregate gradation
2. Compute initial emulsified content
The adoption of trial mix derivatives referred to the specifications of the institutions authorised to create roads and bridges to Iraqi specifications [5], using asphalt cement content as a percentage of total mix as required for surface course (4%, 4.5%, 5%, 5.5%, and 6%) to determine initial residual asphalt content. The mixtures were evaluated by means of the mixing procedure, coating test, compaction efforts, curing, and Marshall Stability. These variables were used to verify that mixes containing initial residual asphalt content at percentage 5%, 5.5%, and 6%, which require a long curing period and are weak in the early stages show good coating and good mixing, despite requiring high effort for compaction. This means that these percentages can be adopted to determine initial residual asphalt content at percentages 2.5%, 3%, 3.5%, 4%, and 4.5%, testing these mixes for different variables such as compaction, mixing, coating, curing, and Marshall stability. Variables used to check the mixes' implementation that were determined by testing the above variables. Tests also determined the initial emulsified content by mass of the total mixture by means of the following equation:

\[ \text{IEC} = \left( \frac{P}{55} \right) \times 100\% \]  

(1)

where P =% Initial Residual asphalt content by mass of total mixture for percentages 2.5%, 3%, 3.5%, 4%, and 4.5%; X value was 55%.
The initial emulsified asphalt content (IEC) of different types of emulsified asphalt used was calculated from the equation and the results found to be 4.54%, 5.45%, 6.36%, 7.27%, and 8.18% by total weight of mixture.

3. Binder Coating Test
The test indicated the degree of compatibility between the binder material and the aggregates and provided a means of selecting the most suitable emulsified type according to the aggregate type used. Using the IEC value, a coating test was carried out by mixing all aggregates and fillers, with the two types of emulsified added afterwards and mixed for about 2 to 5 minutes until an even coating was obtained. The optimum compatibility degree for the cationic emulsified asphalt type with quartz aggregate was better than for the anionic emulsified asphalt type, due to its negative charge produced by the chemical composition of the aggregate (as shown in table [4]), which produces a good bond with a positive charge for the cationic emulsified asphalt. The degree of coating was not less than 50% by visual observation [1, 9].

4. Compaction effort
Compaction of the two types of cold emulsified asphalt mixture was carried out using a Marshall Hammer compactor. In this study, to determine suitable compaction levels, the compaction was carried out at a heavy compaction level of 150, adopted after many trials of blow numbers to reduce the air voids percentage to within the allowable range. The results show that the procedure with a compaction effort of 150 Marshall hammer blows gave suitable results corresponding to an air voids percentage within the allowable range of 5 to 10% [1, 10].
5. Curing of compacted samples for a dry stability test consisted of maintaining the compacted samples in mould compaction for 24 hours. The samples were then extruded and kept for 24 hours in an oven at 40 °C before being removed from the oven and stored for 24 hours at room temperature (25 °C) [1]. The samples were subsequently tested for Marshall stability at 25 °C and the results obtained recorded as dry Marshall stability. Other samples were immersed in water at 60 °C for 24 hours after removal from their moulds. During immersion, the samples rested on a bed of coarse aggregate in the water pan. The samples were subsequently well dried and tested for Marshall stability; the results thus obtained were recorded as wet stability.

2.1 Density and Porosity Calculations
To determine these properties, the samples were weighed dry in air and also when fully immersed in water. These values were used with other known parameter to calculate bulk density and specific gravity, and the maximum (theoretical) specific gravity of air voids according to specifications ASTM D2726, ASTM D2041, and ASTM D3203, respectively. These tests are also suitable for hot mixtures because there is no water added and they are carried out after the curing period.

2.2 Marshall Stability Test
The Marshall stability properties for the two types of emulsified asphalt mixture samples used the same Marshall Stability equipment as used to test hot asphalt mixtures. The procedure, however, was modified so that the samples were tested at room temperature, instead of at 60 °C, and the number of Marshall blows for compaction was 150 blows instead 75 blows for each face, as specified for hot mixtures. This is thus a modified Marshall test [11]. Different emulsified asphalt contents were selected, here specified as 4.54%, 5.45%, 6.36%, 7.27%, and 8.18% of the total weight of mixtures, to determine the optimum emulsified content. Fifteen specimens were prepared and tested, and the optimum emulsified asphalt was determined as a percentage by weight of the mixture. The testing was carried out according to ASTM D6927 [12]. The samples were maintained in the mould for 24 hours, then extruded and kept for 24 hours in a drying oven at 40 °C. After extraction from the oven, they were stored for 24 hours at room temperature of 25 °C; however, the curing time can be reduced on site by using additional densification processes depending on the environmental conditions. The specimens were then placed in a water bath at 25 °C for 30 to 40 minutes before testing for Marshall stability and flow, thus obtaining dry stability and density, and air voids. To obtain the wet Marshall stability, the same procedure was followed in the wet curing stage, as mentioned previously.

2.3 Retained Stability
The retained stability is the ratio of wet stability to dry stability for all mixtures. A minimum of three specimens are required to determine an average dry Marshall stability, and the stability of the wet specimens is similarly obtained to use as a comparison. A minimum of 50% retained stability must be achieved following the design curing procedure for early age testing, according to the references [1].

3. Results and Discussion
Figure 2 shows the bulk density of emulsified asphalt mixtures using two types of emulsified binder at various binder content percentages. The value of the cationic emulsified asphalt content has the greatest density until the maximum is reached. This maximum value is obtained at 6.36% emulsified content. The density tendency for the binder material content in the cationic emulsified asphalt mixture followed the same tendency as hot asphalt mixtures [13], which is to increase with increases of emulsified asphalt content as aggregate particles move closer to one another. Furthermore the results show a decrease in volume and increase in the sample weight that lead to increased density. After a certain percentage, known as the optimum percentage, the binder material (emulsified asphalt) starts to form a film with increased thickness that leads to a reduction in the contact distance between the aggregate materials, resulting in an increase in the sample volume that decreases the density of the sample. On the other hand, the bulk density trend for the anionic emulsified type decreases as the emulsified content increases,
due to the repulsion forces between the negative charges of quartz aggregate and negative charges of the anionic emulsifier, producing a poor bond and a reduced degree of coating, as shown in plate 1 below.

Plate 1. Compatibility and coating degree between different types of emulsified asphalt (cationic & anionic) with quartz aggregate.

Figure 3 shows that, as the cationic emulsified percentage increases, there is a rapid decrease in air voids until a specific percentage to this emulsified type is reached; at this point, the relationship between the emulsified percentage and air voids follows the same tendency as in hot asphalt mixtures. The air voids increase as the anionic emulsified content increases to a certain content close to the optimum emulsified content; after that, the air voids decrease, which can be attributed to reduced density and compatibility of the mixtures. The range of air voids percentage for emulsified asphalt mixtures is about 5 to 10% [1], with the range of air voids percentage for emulsified asphalt mixtures being about 6 to 12% [4]. All percentages of air voids of emulsified binder mixtures are within approved limits. Figure 4 shows that as the cationic emulsified percentage increases, the dry stability decreases continuously. Differences in dry stability for mixtures with respect to emulsified percentage can be attributed mainly to the coating degree based on the percentage of cationic emulsified content. The tuning rate of the emulsified material is reduced, and hence the coating degree of the mixture is improved. The slow-breaking rate results are also improved due to distribution of the binder and a decrease in the amount of aggregates, both of which contribute to improved stability [4]. While an increase in the amount of emulsified percentage improves the coating of aggregate and leads to better dry stability, where the amount of emulsified content is too great, it will produce a very soft mixture with two disadvantages: long curing time and increased loss of water. The soft mixture will require a long time for water to evaporate in order to reach a specific percentage of compaction, then a long time of cationic emulsification to complete setting. On the other hand, the relationship between anionic emulsified percentages and dry stability as an emulsified percentage leads to increases in the dry stability that may be credited to the opposing charges of the emulsion binder and aggregate, which lead to an increase of interface distances that will accelerate losses of water. In general, the dry stability value for cationic emulsified asphalt mixtures is higher than for anionic emulsified asphalt mixtures, which can be attributed to high bulk density, reduced air voids, and high compatibility of mixtures. Figure 5 shows that the wet Marshall stability continuously increases with an increase in emulsion percentage after reaching the maximum in terms of the specific proportion of emulsion content. The changes in low values of wet stability are due to samples during the soaking period absorbing water. The water absorbed may reduce the film between the binder and the total surface, causing a decrease in bonding and adhesion forces. The wet stability values for cationic emulsified asphalt mixtures are higher
than those of anionic emulsified asphalt mixtures, which can be attributed to high bulk density, lower air voids, and a high amount of water absorption. Figure 6 shows that the stability of retention increases with the emulsification content until the maximum optimum emulsifying content is achieved, and that the continuous stability of the retention decreases with the increase of binder content, where it can be determined based on the moisture effect on the mixture. In some cases, the Retained Marshall Stability values were found to be slightly greater than 100%; this was attributed to the development of pore water pressure within the sample during load application [11]. Figure 7 shows that the flow values of cationic emulsion binder mixtures began to decrease as the binder content increased to 5.45%. After this percentage, the flow began to increase with the increase of the emulsified content, reaching the maximum value at a higher percentage of emulsifier. Trend of flow values for anionic emulsified binder mixtures showed that these increased with increases in the emulsified content. The flows for anionic emulsified asphalt mixtures were lower than those for cationic emulsified asphalt mixtures; this can be attributed to the high percentages and hard asphalt residuals of anionic emulsified binders.

![Figure 2](image_url)

**Figure 2.** Initial Emulsion percentage (%) with bulk Density of Two Types of Emulsified Asphalt
Figure 3. Initial Emulsion percentage (%) with Air voids of Two Types of Emulsified Asphalt

Figure 4. Initial Emulsion percentage (%) with Wet Marshall Stability of Two Types of Emulsified Asphalt
Figure 5. Initial Emulsion percentage (%) with Retained Marshall Stability of Two Types of Emulsified Asphalt.

Figure 6. Initial Emulsion percentage (%) with Flow value of Two Types of Emulsified Asphalt.
4. **Optimum Emulsified Asphalt Content (OEAC)**

The OEAC for two types of emulsified asphalt (cationic and anionic) was determined based on wet specimens by optimising the main properties of wet stability, bulk density, and retained stability. Furthermore, air voids values were evaluated with reference to appropriate specifications. All the properties were shown in figures based on the initial emulsified asphalt content (IEAC) [1, 4, 14]. Based on Figure 2, for bulk density, cationic and anionic emulsified asphalt mixtures were rated best at 6.36% and 4.54%, respectively. Figure 5 shows that for wet stability, they were rated at 6.36% and 7.27%, respectively, and for retained stability, 6.36% and 7.27%, respectively. It can thus be concluded that the optimum initial percentage for the two types of emulsified binder percentage is 6.36% and the residual asphalt percentage should be 3.5%.

5. **Conclusions:**

1. Two types of binder materials, cationic emulsified slow setting low viscosity (Css-1), and anionic emulsified slow setting low viscosity hard asphalt residue (SS1h), are suitable for aggregate graduation requirements according to Iraqi specifications. Asphalt residual content of about 55% and water content of about 45% is suitable and more economical compared with hot asphalt mixtures because the aggregate does not require drying during the mixing process.
2. Coating tests show that the workability and compatibility degree for cationic emulsified asphalt types with quartz aggregate are better than for anionic emulsified asphalt types, as seen in plate (1), due to the negative charge from the chemical composition of aggregate that produces a good bond with the positive charge for the emulsified asphalt.
3. Trial mixes show that the best percentages of initial emulsified content based on sufficient results from the coating test, the mixing, the pressure test, and the curing and stability of the Marshall test range from 4.54% to 8.18%. The optimal value is 6.36%.
4. A suitable and efficient mixing procedure for creating an emulsified asphalt mixture consists of mixing coarse aggregate with either type of emulsifier initially before the fine aggregate is added to the mixture. This procedure produces a suitable degree of coating for aggregates and cationic emulsifiers.
5. Compaction effort was set at 150 blows per face of the Marshall mould, which developed porosity within the allowable range of 5 to 10%.
6. A suitable curing time consisted of 24 hours in the mould, 24 hours in the oven at 40 °C, followed by removal from the oven and storage at room temperature (25 °C) for 24 hours. This curing period can be avoided in the field by the application of more densification, depending on the environmental conditions.
7. The Marshall Stability properties showed that the cationic emulsified asphalt type mixture with a cement type filler was better than the anionic emulsified asphalt type.

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