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Characterization and Correlation Analysis of Mechanical Properties and Electrical Resistance of Asphalt Emulsion Cold-Mix Asphalt

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Abstract: Asphalt emulsion cold-mix asphalt (CMA) is generally characterized as an evolutive material, with its mechanical strength increasing with curing time. This study aims to characterize the development of the mechanical properties of asphalt emulsion CMA mixtures during the curing process and correlate them with their evolutive electrical resistances. To achieve this objective, the moisture loss, Marshall stability, indirect tensile strength (ITS), indirect tensile stiffness modulus (ITSM) and electrical impedance of CMA mixtures containing various percentages of Portland cement were evaluated at different curing times up to 28 days. These evolutive properties were then analyzed and their relationships were explored. It was found the mechanical performance of CMA was considerably improved by cement, and there were liner relationships between the Marshall stability, ITS and ITSM irrespective of cement content. Furthermore, the mechanical performance was closely related with the electrical resistance of each CMA mixture containing different amounts of cement, suggesting that it is potentially feasible to predict the evolutive mechanical performance of CMA from electrical resistance which can be measured non-destructively.

Key words: Cold-Mix Asphalt; Asphalt Emulsion; Evolutive Properties; Mechanical Performance; Electrical Resistance

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1. Introduction

Asphalt cement is a viscoelastic material, which generally has to be heated to a temperature of over 130℃ to compact hot-mix asphalt pavement [1, 2]. As a result, large amount of volatile organic compounds (VOC) and greenhouse gas (GHG) emissions may be produced during the manufacturing, transportation and paving processes of HMA [3]. On the contrary, cold-mix asphalt (CMA), typically using asphalt emulsion as the bonding material, is considered as a more eco-friendly paving material in terms of environment impacts and energy saving, because it can be produced at ambient temperature [4-7]. However, it takes more time for asphalt emulsion to cure and build its strength [8, 9], and asphalt emulsion CMA typically faces the concerns of low early strength and long curing time [10]. In fact, CMA is an evolutive material, for the strength increases slowly as emulsion breaking in the curing process. As a result, the applications of CMA are limited in the low and medium-traffic pavements, base courses or thin overlayers [11-14].

To improve the mechanical properties of bitumen emulsion based asphalt mixtures, additives such as Portland cement, fly ash and lime are commonly used [15-20]. It has been reported that an introduction of 1-3% cement can significantly improve the early strength of CMA, due to water adsorption and cement hydration reaction [21]. The combination of cement and bitumen emulsion provides CMA the features of both cement and bitumen: higher toughness, reduced temperature susceptibility, enhanced mechanical properties and fatigue life [22-24]. Niazi and Jalili [16] evaluated the effect of Portland cement and lime on the properties of CMA mixtures with bitumen emulsion and found that both Portland cement and lime can improve the Marshall stability and resilient modulus. Leng, et al. [19] investigated the influence of different contents of cement on the properties of CMA, and found that both the early-age strength and long-term performance were improved. Oruc, et al. [22] also evaluated the performance of CMA with different amounts of cement (0-6%), and concluded that cement acted as a secondary binder in CMA and could improve the resilient modulus, resistance to moisture damage and permanent deformation. Graziani, et al. [25] revealed that the mechanical performance of cold cement-bituminous mixtures evolved over time and generally stabilized after 28 days, which was similar to the findings by some other studies [14, 18]. The same group further explored to simulate the evolutive properties of CMA using numerical models [25, 26], and found that Michaelis-Menten (MM) model was effective in characterizing the performance evolution of CMA.

Electrical impedance spectroscopy (EIS) is an effective method to evaluate the drying behavior of cement mortar [27-29]. The electrical resistance obtained from this test showed an upward trend as the extension of curing time [30]. Haddock, et al. [31] applied electrical resistance as an indicator to evaluate the curing process of chip seal. They found that the mechanical strength of chip seal was closely related to its measured electrical resistance.

The objective of this study is to characterize the performance development of asphalt emulsion CMA containing various dosages of cement with curing time and investigate the relationships between different properties of CMA during the curing process. To achieve this objective, various properties of CMA, including moisture loss, Marshall stability, indirect tensile strength (ITS), indirect tensile stiffness modulus (ITSM) and electrical resistance, were evaluated at different curing times up to 28 days. These evolutive properties were then analyzed and their relationships were explored.

2. Materials and testing methods

2.1 Materials

Base bitumen Pen 60/70 was used to prepare cationic slow setting bitumen emulsion, and the solid content was 63.0%. The basic properties of base bitumen are presented in Table 1. ASTM Type I Portland cement was applied to improve the early strength of asphalt emulsion CMA. Table 2 shows the chemical composition of the cement. Crushed granite aggregate and mineral filler were obtained from a local supplier. The gradation and basic properties of aggregate are shown in Fig. 1 and Table 3, respectively, and the maximum particle size of the aggregate is 14 mm.

Table 1 Basic properties of base bitumen

Penetration (25 °C, 0.1 mm)	Softening point (°C)	Viscosity at 135 °C (mPa·s)
64.5	50	477.5

Table 2 Chemical composition of Portland cement

Substance	Content (%)
CaO	64.51
SiO_2	19.57
Al_2O_3	3.81
Fe_2O_3	3.12
SO_3	5.43
MgO	1.48
TiO_2	0.27
K_2O	0.69

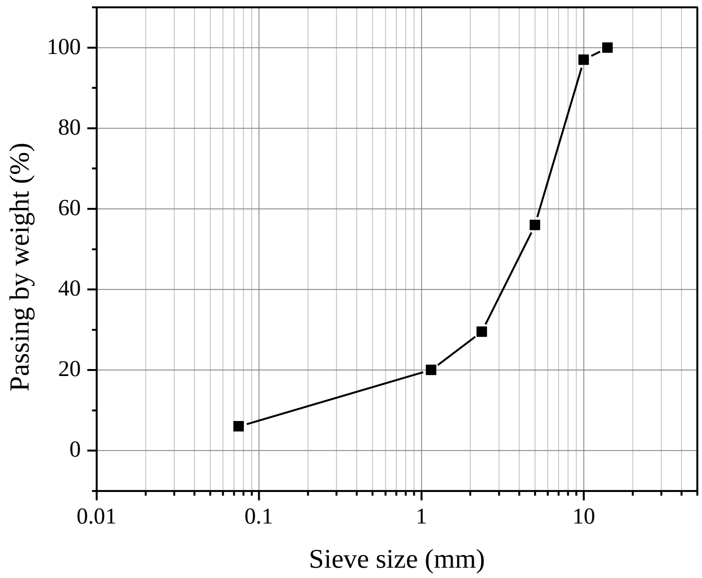


Fig. 1 Aggregate gradation

Table 3 Physical properties of aggregates

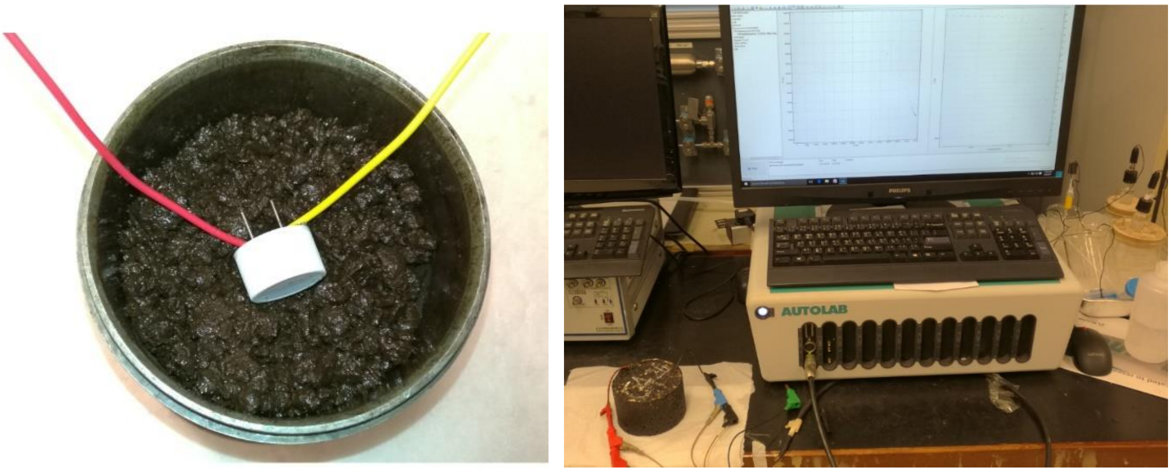
Properties	Value
Course aggregate (5-14 mm)	
Bulk specific gravity (g/cm^3)	2.581
Apparent specific gravity (g/cm^3)	2.598
Moisture content (%)	0.13
Fine aggregate (0.075-5 mm)	
Bulk specific gravity (g/cm^3)	2.577
Apparent specific gravity (g/cm^3)	2.601
Moisture content (%)	0.13
Mineral filler	
Apparent specific gravity (g/cm^3)	2.631
Moisture content (%)	0.85
Portland cement	
Apparent specific gravity (g/cm^3)	3.150
Moisture content (%)	0.31

2.2 Specimen preparation and testing

In the first phase, the optimum emulsion content was determined based on the volumetric properties, Marshall stability and ITS test results of cured CMA prepared with different asphalt emulsion contents (6-10% by weight of aggregates). To improve the absorption between bitumen emulsion and aggregates [25], the aggregates were first pre-wetted and mixed with water for about 1 min. Pre-wetting water content of 1.5% by weight of aggregates was found to be appropriate by experiments. Then, the pre-wetted aggregates were mixed with bitumen emulsion for about 3 min until a homogeneous state was achieved. Afterwards, the loose mixture was transferred to a Marshall mould and subjected to 50 blows of compaction on each side using the Marshall hammer [17]. The specimens were then cured for 1 day at ambient room conditions before being extruded from the moulds, followed by oven conditioning at 60 °C for 3 days [32]. The high temperature conditioning at 60 °C was chosen to accelerate the curing rate. The percent air voids, Marshall stability and ITS were tested to determine the optimal bitumen emulsion content.

In the second phase of the study, the evolutive properties of CMA were measured and analysed. Portland cement were added to replace part of mineral filler. The percentages of cement were 0%, 2%, 4% and 6% by weight of aggregates, and are denoted as C0, C2, C4 and C6, respectively. The 6% of cement represents fully replacement of mineral filler by cement. Upon being extruded from the moulds, the Marshall specimens were kept under room conditions (temperature: 23 ± 1 °C, relative humidity: 55 ± 5%) for up to 28 days. The moisture loss and mechanical strength were tested after 1, 3, 7, 14 and 28 days of curing. Following 28 days of ambient conditioning, moisture susceptibility test of CMA was performed using both Marshall stability and ITS after water conditioning for 24 h at 60 °C. In addition, normal dense-graded HMA fabricated with the aggregates of similar gradation and base bitumen Pen 60/70 served for comparison purpose in this study. The weight ratio of base bitumen Pen 60/70 to aggregates was 5.0% for the HMA. The HMA in this study was compacted with the standard Marshall method (75 blows on each side), and resulted with an average air void content of 2.95%.

The electrical resistance of CMA Marshall specimens was also measured in this study. The electrodes used to evaluate the electrical resistance were custom-made in lab. Two steel rods spaced 10 mm apart were fixed and moulded using epoxy resin. The electrodes were then embedded in the middle of the mixture during sample preparation. The electrical resistance of the specimens was measured with the Metrohm High Performance Modular Potentiostat over a frequency range from 1 MHz to 0.01 Hz using alternative current (AC) signal after curing for 1, 3, 7, 14 and 28 days (Fig. 2).



(a) (b)

Fig. 2 Electrical resistance testing (a) preparation of testing specimen, (b) test setup

Table 4 provides a summary of the tests conducted in this study. As the ITSM test is non-destructive, the specimens after ITSM test were subsequently used for ITS measurement. Two replicates were prepared for all the tests, and a total of 124 CMA Marshall specimens were fabricated in this study.

Table 4 Tests conducted in this study

Test item		Conditioning	Test temperature (°C)	Standard
Optimum emulsion content	Marshall stability	60 °C for 3 days	60	ASTM D6927
	ITS		25	ASTM D6931
Moisture loss		1, 3, 7, 14 and 28 days ambient	Ambient	/
Marshall stability		1, 3, 7, 14 and 28 days ambient	60	ASTM D6927
Indirect tensile stiffness modulus (ITSM)		1, 3, 7, 14 and 28 days ambient	20	BS EN 12697
Indirect tensile strength (ITS)		1, 3, 7, 14 and 28 days ambient	20	ASTM D6931
Moisture susceptibility	Marshall stability	28 days ambient + 24 h 60 °C	60	ASTM D4867
	ITS		20	
Electrical impedance		1, 3, 7, 14 and 28 days ambient	Ambient	/

3. Results and discussion

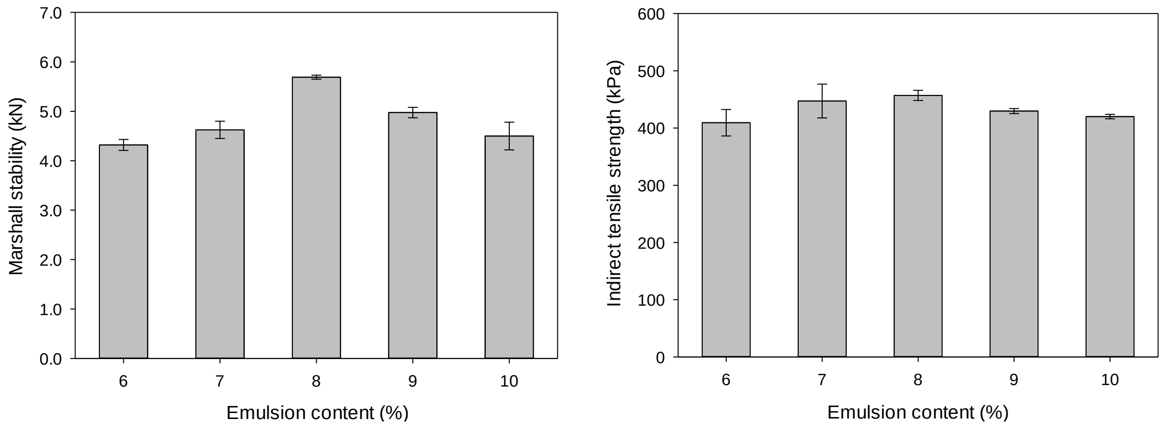
3.1 Optimum emulsion content

The densities and air void contents of the cold mixtures with different contents of asphalt emulsion are shown in Table 5. It can be seen that the bulk density increased to 2.135 g/cm3 with 8% of asphalt emulsion, and then decreased with the further increase of the asphalt emulsion content. The air void contents of the CMA specimens are distributed within the range of 12.9% to 14.9%, and show a decreasing trend with the increase of asphalt emulsion content.

Table 5 Density and air voids of CMA with different contents of asphalt emulsion

Asphalt emulsion content (%)	Bulk density (g/cm^3)	Maximum density (g/cm^3)	Air void content (%)
6.0	2.121	2.491	14.9
7.0	2.124	2.480	14.4
8.0	2.135	2.465	13.4
9.0	2.127	2.445	13.0
10.0	2.123	2.438	12.9

Fig. 3 presents the Marshall stability and ITS tests results of the CMA with different asphalt emulsion contents. It is clear that 8% of bitumen emulsion by weight of aggregates resulted in the largest Marshall stability and ITS. The Marshall stability first increased and then decreased with increasing emulsion contents (Fig. 3(a)), while the values of ITS are relatively close for CMA with different emulsion contents (Fig. 3(b)). As shown in Table 5, the air void contents are very large within the range of 12.9% to 14.9%. In addition, the residual moisture within the mixture significantly affects the cohesion and adhesion of CMA [33]. As the large air void content and residual moisture content dominate the ITS of the mixtures, the ITS values of CMA with different amounts of emulsion are in generally close to each other. On the other hand, the Marshall stability is also influenced by the internal friction of the mixture. It can be seen that the bulk density of the mixture with 8% of emulsion was the largest (Table 5), which led to higher Marshall stability as a result of the good inter-locking of the aggregates. Thus, the optimum emulsion content was determined to be 8% (equivalent to 5.04% of residue bitumen binder) and was used in the following study.



(a) (b)

Fig. 3 Influence of emulsion content on (a) Marshall stability and (b) ITS

3.2 Moisture loss of CMA

Fig. 4 shows the moisture loss ratio of CMA with a curing period of 1 day to 28 days. It can be observed in Fig. 4 that the moisture loss curve can be divided into three stages: the sharp increasing period during the first 3 days (Stage 1), followed by a slower increasing period during curing period of 3 days to 14 days (Stage 2), and then reached a plateau during curing period of 14 days to 28 days (Stage 3). Similar results were also reported by some other studies [14, 26]. Moreover, it can be noticed that with the increase of cement content in CMA, the moisture loss rate decreased, which is mainly attributed to the hydration of cement [34]. At the end of the conditioning period (28 days), the moisture loss ratios were 69.3%, 57.9%, 50.2% and 37.6% for C0, C2, C4 and C6, respectively, which indicated that the cement hydration consumed part of the water and lowered the water evaporation substantially.

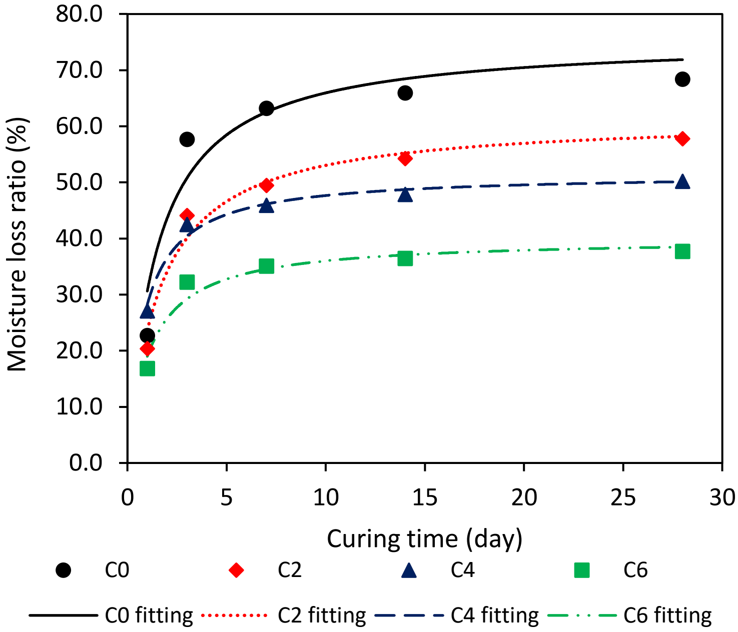


Fig. 4 Moisture loss with curing time

The Michaelis-Menten model, frequently used to describe the chemical reaction rate against the substance concentration [35, 36], was employed to fit the moisture loss and mechanical performance evolution in the later sections of this paper. The MM model can be described by the following equation.

$$f(t) = \frac{y_A t}{K_c + t} \qquad (1)$$

where f(t) is the performance evolution with curing time (t), y_A is the asymptotic value in the long term, and K_c is the Michaelis constant, which is the time when f(t) = $y_A/2$.

The nonlinear-least square minimization method was applied to fit the test data to the MM model using MATLAB®. Table 6 presents the fitting values of y_A, and K_c in the MM model as well as the corresponding determination coefficient (R^2) and the root mean square error (RMSE). It can be observed that the measured data and the estimated results were in good agreement, with the R^2 for all the fitted regression was larger than 0.90.

Table 6 Numerical fitting of moisture loss by MM model

CMA	y_A (%)	K_c (day)	R^2	RMSE

C0	75.6	1.496	0.91	6.54
C2	61.5	1.603	0.97	3.06
C4	51.6	0.828	0.98	1.45
C6	40.0	1.107	0.95	2.23

3.3 Mechanical performance evolution with curing time

Fig. 5 demonstrates the Marshall stability, ITS and ITSM evolution as a function of curing time. As expected, all mechanical properties increased with curing. Similar to moisture loss, the mechanical properties also showed a three-stage development (Fig. 5), i.e., the first rapid increment stage (0-3 days), the second slower increasing stage (3-14 days) and the third relatively stable stage (after 14 days). It is also obvious that the mechanical properties increased considerably faster in the first stage with larger cement concentrations. In other words, the addition of cement was effective in increasing the early strength of CMA. In addition, the increase of cement content led to improved final mechanical properties. After 28 days, the Marshall stabilities for C0, C2, C4 and C6 were 5.7 kN, 10.5 kN, 16.0 kN and 21.0 kN, respectively. In comparison with the control CMA (C0), the Marshall stabilities of C2, C4 and C6 increased by 84.9%, 181.4% and 269.2%, respectively. The mechanical properties, including Marshall stability, ITS and ITSM after 28 days are summarised in Table 7. It is clear that larger quantities of cement resulted in improved mechanical performance, with the most remarkable increment in ITSM. Compared with the dense-graded HMA, C6 had larger Marshall stability, ITS and ITSM after only 3 days. However, it should be noted that the stiffness modulus for C4 and C6 are very high (13,805.2 and 17,656.9 MPa, respectively) after curing for 28 days, which may lead to increase of brittleness of the cold mix, and thus the fatigue life could be adversely affected [37]. In summary, the incorporation of cement could significantly improve both the mechanical strength and stiffness modulus of CMA, and such effect is more significant in stiffness modulus [18].

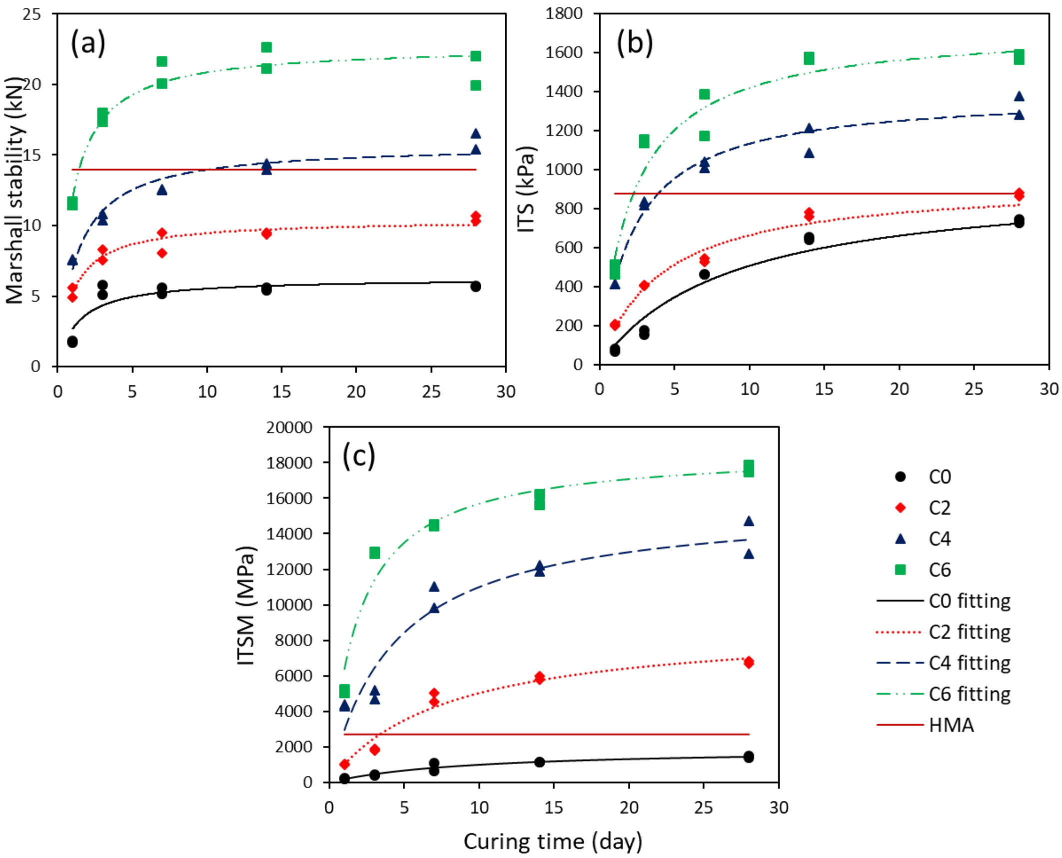


Fig. 5 Mechanical performance evolution with curing time (a) Marshall stability, (b) ITS and (c) ITSM

Table 7 Mechanical strength of different CMA after 28 days and HMA

Mix	Marshall stability (kN)	ITS (kPa)	ITSM (MPa)
C0	5.7	677.0	1437.0
C2	10.5	806.8	6764.2
C4	16.0	1330.7	13805.2
C6	21.0	1576.6	17656.9
HMA	13.9	874.7	2705.5

Similar to moisture loss development, the mechanical performance evolution data were also fitted to the MM model. The regression parameters y_A, and K_c as well as R^2 and RMSE are presented in Table 8. As mentioned earlier, y_A stands for the asymptotic value in the long term, and K_c is the time needed to reach the value of $y_A/2$. From the data in Table 8, it can be observed that y_A shows an increasing trend with larger quantities of cement while Kc demonstrates the opposite trend, except for some variances in the case of Marshall stability. The obtained results revealed that higher concentrations of cement in CMA leads to better long-term mechanical performance. In addition, the decreased K_c values indicates that the initial curing rate was increased, i.e., less time is needed to reach $y_A/2$. Thus, it can be confirmed that the cement could not only improve the long-term mechanical performance, but also increase the initial curing rate of CMA. Table 8 further verifies that the measured results were in good agreement with the MM model, with most of the R^2 exceeded 0.90 except for the Marshall stability for C0. As a result, it is reasonable to use the MM model to describe the mechanical performance evolution of CMA.

Table 8 Regression parameters obtained using MM model for mechanical performance

Mix	y_A	K_c	R^2	RMSE
Marshall stability	kN	day		
C0	6.27	1.34	0.79	0.78
C2	10.42	1.00	0.92	0.56
C4	15.75	1.28	0.94	0.80
C6	23.49	1.00	0.98	0.65
ITS	kPa	day		
C0	950	8.80	0.95	60.2
C2	939	4.17	0.98	38.0
C4	1393	2.30	0.97	56.9
C6	1733	2.03	0.95	100.5
ITSM	MPa	day		
C0	1928	9.40	0.99	55
C2	8906	7.70	0.97	470
C4	15790	4.35	0.93	1124
C6	18730	1.93	0.95	1060

3.4 Moisture susceptibility

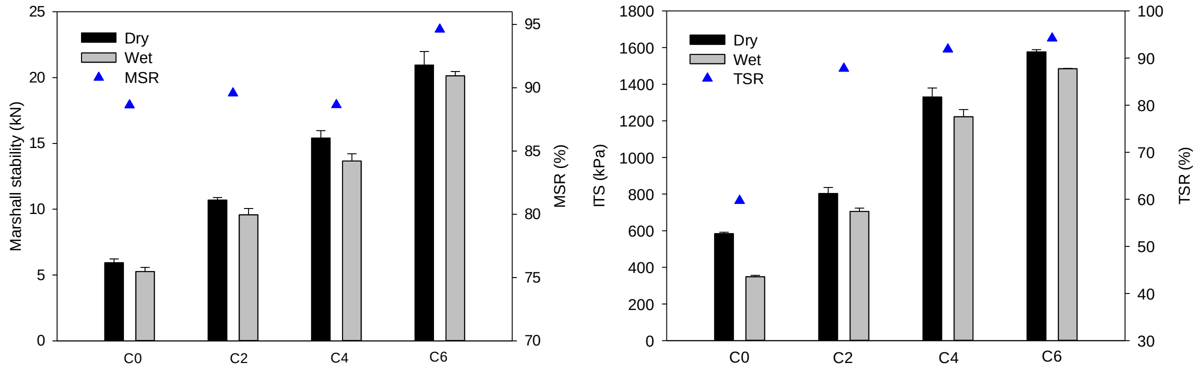
The moisture susceptibilities of CMA with different percentages of cement were evaluated through the Marshall stability and ITS tests with and without moisture conditioning. The Marshall stability ratio (MSR) and tensile strength ratio (TSR) were calculated using Equation (2) and Equation (3), respectively.

$$\mathrm{MSR} = \frac{MS_{wet}}{MS_{dry}} \qquad (2)$$

Where MS_{wet} = average Marshall stability of the 24 h soaked subsets, and MS_{dry} = average Marshall stability of the dry subsets.

$$\text{TSR} = \frac{ITS_{wet}}{ITS_{dry}} \qquad (3)$$

Where ITS_{wet} = average ITS of the 24 h soaked subsets, and ITS_{dry}= average ITS of the dry subsets.



(a) (b)

Fig. 6 Moisture susceptibility of CMA (a) Marshall stability and MSR, (b) ITS and TSR

Fig. 6 (a) and (b) illustrate the moisture susceptibility testing results based on Marshall stability and ITS, respectively. It is obvious that the both Marshall stability and ITS dropped after water conditioning for all the specimens. However, no significant difference was observed with respect to MSR between CMA with different cement dosages, which was larger than 80% for all the four groups of mixtures (Fig. 6 (a)). By contrast, the TSR increased considerably with the addition of cement. This result indicates that cement increased the cohesion of the bitumen emulsion based CMA, and that TSR is more critical than MSR in characterizing the moisture susceptibility of CMA. It can be noticed that the ITS of C0 decreased the most after wet conditioning in comparison with their dry counterparts, which led to the lowest TSR (60%) among the four subsets. The significant moisture damage for normal CMA without cement is generally attributed to large air void content and lack of adhesion between the emulsion residue and aggregate [38, 39]. What stands out in Fig. 6 (b) is that the TSR for C2 reached 88%, which is notably larger than that of C0. The TSR did not change much with further addition of cement. It is generally recognized that a TSR of 80% or above represents acceptable value for moisture resistance [16]. The MSR and TSR results lead to the conclusion that cement can effectively improve the resistance to moisture damage. This improvement is primarily due to that the cement hydration products acting as a secondary binder which reinforced the bonding between aggregates [18, 22, 40, 41].

3.5 Electrical impedance spectroscopy

Fig. 7 shows the schematic of the Nyquist curve obtained from the EIS measurement, which consists of the real impedance (horizontal) and the imaginary impedance (vertical). The left smaller arc, resulted from the high frequency AC current, is attributed to the CMA bulk resistance effect, while the right larger arc belongs to the electrode-mixture interface effect in Fig. 7 [29, 42], and R_b is determined as the bulk electrical resistance of the tested specimens. The EIS spectra of C4 from 1 day to 28 days are displayed in Fig. 8 as an example in this paper. The electrical resistance values for 1, 3, 7, 14 and 28 days were then obtained and presented in Fig. 9 for C0, C2, C4 and C6. It can be seen that the electrical resistances for all CMA mixtures increased with curing time (Fig. 9), which is primarily caused by the curing of emulsion and evaporation of water. As aggregates and bitumen are not conductive, with the breaking of bitumen emulsion and loss of water in CMA, the resistance to electricity flow would increase. This trend is similar to the findings from cement mortar EIS testing during the initial drying period [43]. The electrical resistance was the largest for C0 at all the testing ages, since the water loss ratio was the highest for C0 as discussed in Section 3.2. With the increase of cement quantities, the electrical resistance decreased substantially. The electrical resistance of C2 was almost two orders lower than that of C0. As the cement hydration decreased the water loss ratio, the residual water content in CMA containing cement was always larger than that of C0, thus the conductivity of these mixtures increased.

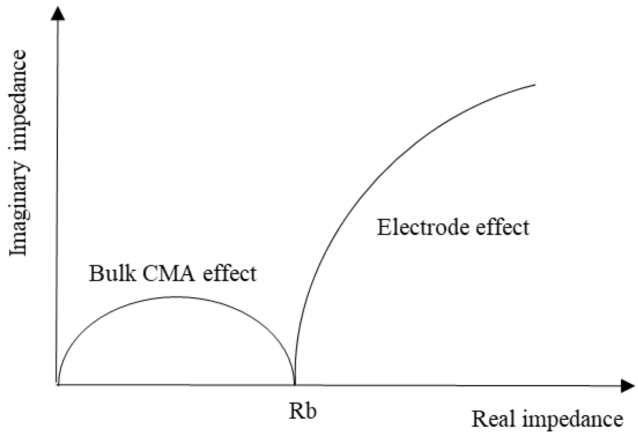


Fig. 7 Schematic of Nyquist EIS spectrum

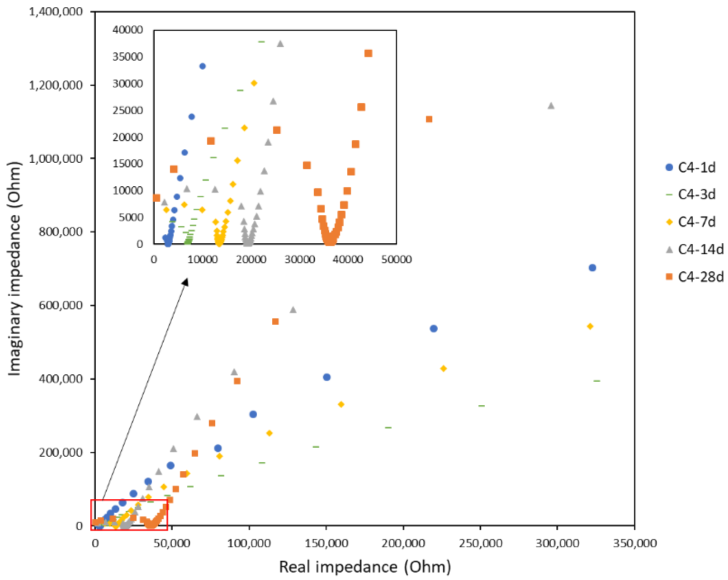


Fig. 8 Nyquist curves of CMA after conditioning for 1, 3, 7, 14 and 28 days

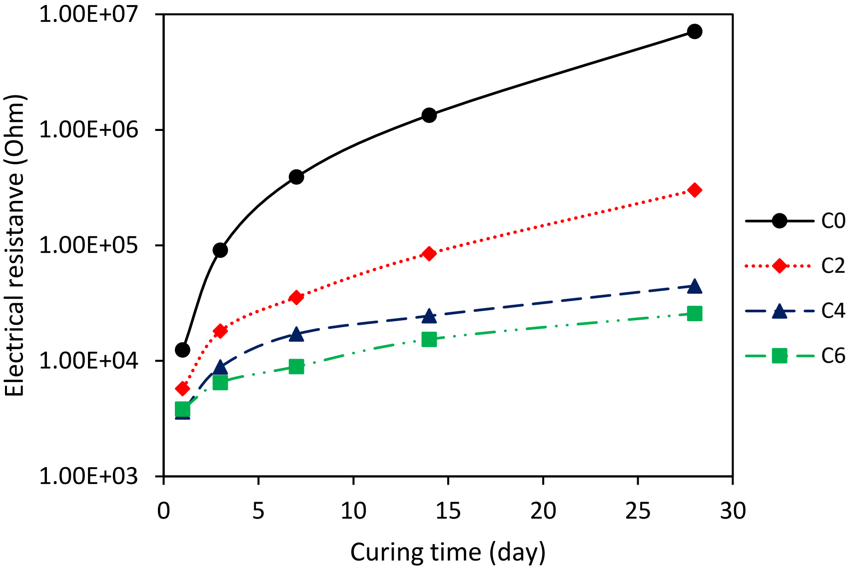


Fig. 9 Electrical resistance evolution with curing time

4. Correlation between the evolutive properties

4.1 Relationships between different mechanical properties

Fig. 10 illustrates the relationships among Marshall stability, ITS and ITSM. It is noticeable that the three mechanical properties had good linear relationships irrespective of cement contents. The values of R^2 for the linear regression models between Marshall stability and ITS, Marshall stability and ITSM, and ITS and ITSM were 0.85, 0.94 and 0.88, respectively. This result indicates that overall the mechanical performance indexes, including Marshall stability, ITS and ITSM, are closely inter-depended.

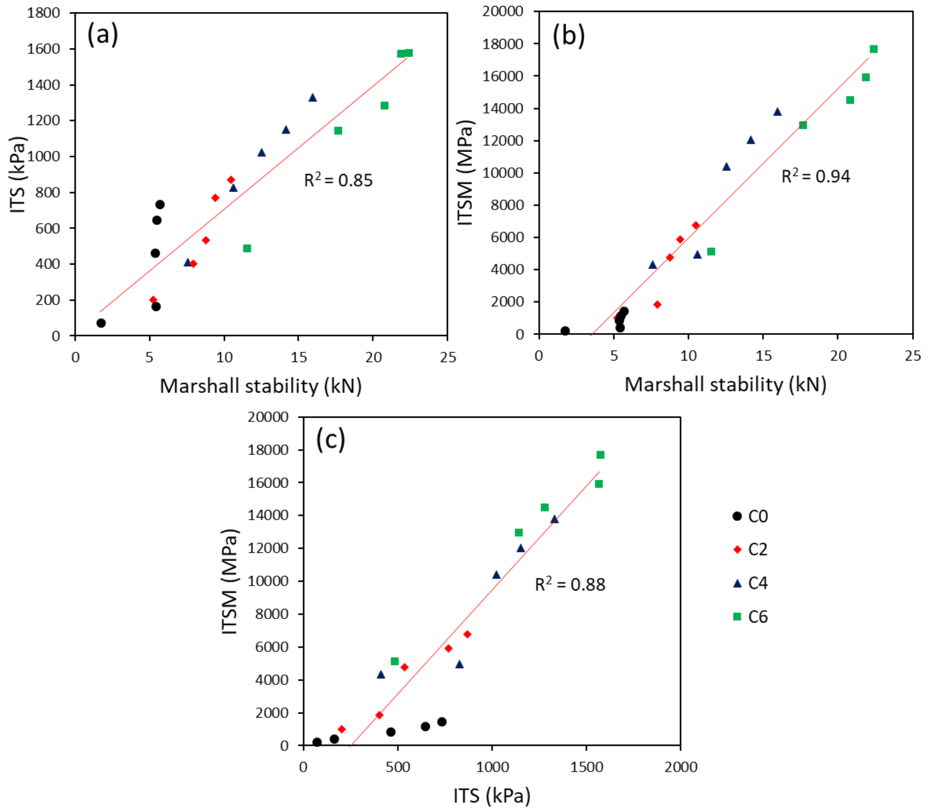


Fig. 10 Relationships between different mechanical properties (a)Marshall stability-ITS, (b)Marshall stability- ITSM and (c) ITS-ITSM

4.2 Relationships between electrical resistance and mechanical properties

The correlations of Marshall stability, ITS and ITSM with electrical resistance are plotted in Fig. 11. It can be found that although there are no clear relationships between electrical resistance and strength performance overall, roughly linear regression between (lg) electrical resistance and strength performance indexes including Marshall stability, ITS and ITSM was established for each specimen, with all the average determination coefficient R^2 exceeded 0.80. More specifically, the average values of R^2 for ITS and ITSM were both 0.90 or above, which indicated that relatively good linear regression was obtained for each sample. These results reveal that it is possible to use the electrical resistance test as a potential non-destructive method to characterize the evolutive mechanical performance of bitumen emulsion based CMA. For a given CMA mixture, if the relation models can be developed in the laboratory, it is possible to use these models to predict its in-situ curing condition.

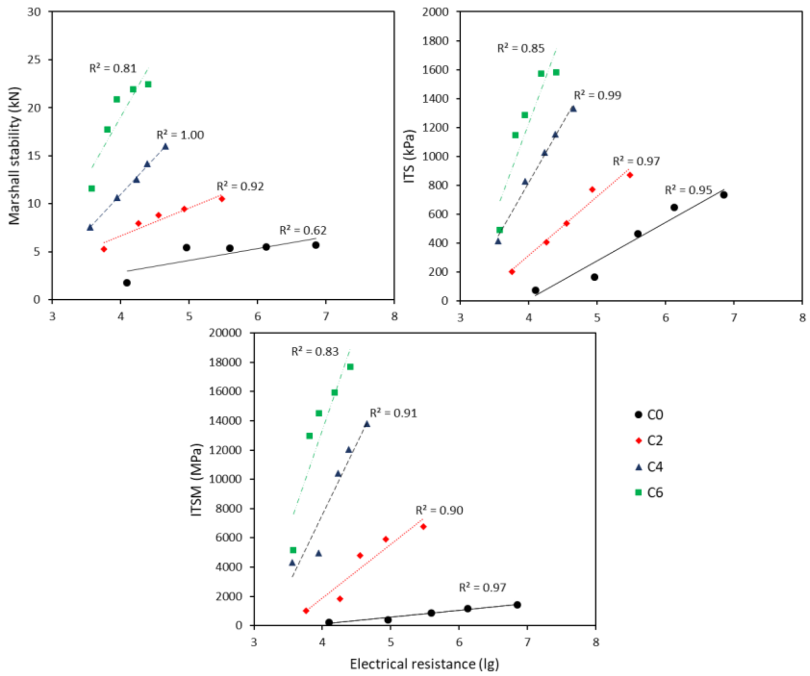


Fig. 11 Relationships between electrical resistance and mechanical performance indexes (a) Marshall stability, (b) ITS and (c) ITSM

5. Conclusions

In this study, the evolutive properties of cold-mix asphalt mixture (CMA) including moisture loss, Marshall stability, ITS, ITSM as well as electrical resistance, were evaluated. Based on the outcome of this study, the following conclusions can be drawn:

(1) With the incorporation of cement into CMA, the moisture loss rate decreased significantly, while the early strength, the final mechanical performance and the resistance to moisture susceptibility of CMA were improved due to cement hydration.

(2) The mechanical performance evolution with time was successfully fitted with the Michaelis–Menten model.

(3) The Marshall stability, ITS and ITSM were linearly correlated with each other regardless of cement content.

(4) The mechanical performance was closely related with electrical resistance for each CMA containing different amounts of cement. Thus, it is potentially applicable to characterize the evolutive performance of CMA using electrical resistance as a non-destructive testing method.

Overall, the findings from this study indicated that the incorporation of Portland cement could effectively increase the mechanical performance of asphalt emulsion based cold mixture. It should be noted that with larger amounts (4% and 6%) of cement, the modulus become very high, which makes the mixture very rigid and may increase the mixture fragility. Therefore, further research should be undertaken to investigate other properties of the cementitious asphalt emulsion based cold mixture including fatigue, rutting and complex modulus.

6. Acknowledgement

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