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ORIGINAL ARTICLE

Enhancing fatigue resistance and low-temperature performance of asphalt pavements using antioxidant additives

Yongping Hu · Xu Cheng · Anand Sreeram · Wei Si · Bo Li · Georgios Pipintakos · Gordon D. Airey

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Abstract Ageing results in significant performance deterioration of asphalt, especially in relation to its fatigue and low-temperature performance. This performance deterioration can theoretically be lowered by incorporating antioxidants in asphalt mixtures. Although there are several promising studies that have shown the potential efficacy of antioxidants such as zinc diethyldithiocarbamate (ZDC), no work has comprehensively evaluated its performance. In this regard, ZDC was employed to evaluate its effect as an antioxidant to slow down the ageing related performance deterioration of bitumen and asphalt mixtures. Both ZDC-modified (3% and 5%) and unmodified bitumen and asphalt mixtures were subjected to short-term and long-term ageing. Afterwards, linear amplitude sweep (LAS) tests and low-temperature frequency sweep tests were carried out on the bitumen samples using a dynamic shear rheometer (DSR). Four-point bending (4PB) fatigue tests were carried out at 25 °C, and indirect tensile asphalt cracking tests (IDEAL-CT) were carried out at 25 °C and −10 °C on the various asphalt mixtures. It was seen that properties of long-term aged bitumen and asphalt mixtures measured at low temperature and intermediate temperature could be improved by 13–69% for mixtures and 1–44% for bitumen with the addition of ZDC, compared to the unmodified samples. The ageing-mitigation efficiency of ZDC was more pronounced for the low-temperature performance-based metrics since its performance deterioration rate was significantly reduced. Overall, a comprehensive performance evaluation of the effectiveness of antioxidants at different scales provided robust evidence for the potential extension of this technology to field trials and application.

Keywords Bitumen · Asphalt mixture · Ageing · Antioxidation · Fatigue · Cracking

1 Introduction

Due to the combined effects of thermal, UV, reactive oxygen species (ROS) and visible radiation etc.,

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Y. Hu · A. Sreeram (✉) · G. D. Airey
Nottingham Transportation Engineering Centre (NTEC), Faculty of Engineering, University of Nottingham, Nottingham NG7 2RD, UK
e-mail: anand.sreeram@nottingham.ac.uk

X. Cheng · W. Si
Key Laboratory for Special Area Highway Engineering of Ministry of Education, Chang'an University, Xi'an 710064, China

B. Li
Gansu Industry Technology Center of Transportation Construction Materials Research and Application, Lanzhou Jiaotong University, Lanzhou 730070, Gansu, China

G. Pipintakos
SuPAR Research Group, University of Antwerp, Groenenborgerlaan 179, Antwerp, Belgium

bitumen within asphalt mixtures undergoes oxidation over time, inevitably leading to deterioration in its performance and usability [1–4]. The interaction between bitumen and atmospheric oxygen leads to ageing of bitumen, resulting in the formation of increased carbonyl and sulfoxide-related functional groups. These functional groups have been recognised to act as metrics of bitumen ageing and show generally an increasing trend at severe levels of ageing [5]. In terms of its polarity spectrum, bitumen can be separated into asphaltenes and maltenes with asphaltenes being the most polar fraction of bitumen. Maltenes can be further separated into resins, aromatics and saturates. This polarity-based fractions are commonly termed as the "SARA" fractions of bitumen [6]. Ageing leads to a decrease in the less polar fractions such as aromatics while leading to an increase in the more polar fractions such as resins and asphaltenes [7, 8]. The changes in chemical composition during ageing result in changes in its physical properties, such as reduced penetration, ductility, and phase angle, together with increased stiffness, softening point and viscosity [9]. These changes generally improve the rutting resistance of asphalt mixtures while deteriorating the fatigue and thermal cracking resistance [10–12]. The accumulation of ageing-induced damages can eventually lead to more severe distresses such as map cracking and others [13, 14].

Ageing mainly deteriorates the low-temperature and intermediate-temperature (fatigue) performance of bitumen, resulting in cracking issues [15, 16]. At the bitumen scale, ageing results in decreased ductility of bitumen, leading to more negative values of ΔTc, which consequently results in higher susceptibility to thermal cracking [17, 18]. Moreover, ageing reduces the fatigue life of bitumen when the strain levels are relatively high and is detrimental to the durability of asphalt pavements [19, 20]. At the mixture scale, ageing reduces the adhesion between bitumen and aggregates, resulting in adhesion-related damage such as ravelling [21]. Moreover, ageing is detrimental to the relaxation ability, self-healing ability, and cracking resistance of asphalt mixtures, reducing its overall longevity [13, 22].

One of the most promising methods to reduce the negative effects of ageing is incorporating additives termed as "antioxidants" [23, 24]. Antioxidants are chemical additives that can theoretically slow down the rate of oxidation and extend the life of asphalt pavements [25, 26]. Antioxidants could reduce the rate of stiffening of bitumen during ageing, thereby reducing the rate of performance deterioration [27–29]. Several additives have been used as antioxidants in previous research, including Zinc diethyldithiocarbamate (ZDC), kraft lignin, phenols, quercetin, gallic acid, phenothiazine calcium hydroxide, and Vitamin E [28–31]. Literature reports that there are mainly two categories of antioxidants based on their theoretical mechanisms of action in bitumen [25]. The first category of antioxidants are used as co-additives with modifiers. Its mechanism of action is related to improving the stability and compatibility between bitumen and modifiers, thereby slowing down the performance deterioration of asphalt pavements [25]. The second category of antioxidants are normally used to capture or eliminate free radicals and associated species which are responsible for initiating and extending the oxidation process, hence the rate of oxidation is slowed down [25, 26]. Out of many additives that have shown promising effects, ZDC has been reported to be highly effective and promising for reducing the rate of ageing effect of bitumen [32]. Recent global collaborative research tested 28 types of bitumen modified with ZDC and characterised the trends relating to oxidative behaviour of modified bitumen using Fourier Transform Infrared Spectroscopy (FTIR) and Dynamic Shear Rheometer (DSR) measurements. The study indicated that the addition of ZDC maintained the high-temperature performance effectively while lowering the content of carbonyl functional groups and reducing the stiffening effect caused by 20 h of PAV ageing by up to 50%, as compared to some unmodified binders [32]. Moreover, ZDC has been reported to be highly effective in improving the efficiency of rejuvenators as the simultaneous addition of ZDC can improve the permanent deformation resistance and fatigue cracking resistance of rejuvenated bitumen [29, 33], and reduce the susceptibility of rejuvenators to oxidation [29]. Literature has reported that the addition of ZDC also effectively improved the antioxidation of SBS modified binders [34].

Although using antioxidants, such as ZDC, to inhibit the oxidation process in bitumen has proven to be a promising approach, a comprehensive understanding of its effectiveness and broad applicability remains confined. Previous works generally focused on basic rheological properties of bitumen to illustrate the effectiveness of ZDC such as stiffness, and performance grade (PG) [29, 32, 33, 35]. However, these properties cannot reveal the complete performance-related effectiveness of ZDC, as some characteristic properties such as complex shear modulus are not deemed the best metric of the rheological performance of bitumen. The performance of bitumen is determined by its modulus, viscoelastic balance, elastic recoverability, strain/stress resistance etc. Therefore, comprehensive evaluation requires considering multiple rheological properties to accurately determine the impact of antioxidants [36, 37]. Moreover, most studies have only focused on the bitumen scale, without sufficient evidence of effectiveness at the mixture scale. Therefore, a more thorough evaluation is required to determine the effectiveness of antioxidants on specific properties of bitumen and mixtures, especially related to fatigue performance and low-temperature performance. In this regard, this study aims to investigate the low-temperature and fatigue performance of ZDC-modified bitumen and asphalt mixtures. It is envisaged that comprehensive performance evaluation of the effectiveness of antioxidants can potentially provide further robust evidence for the extension of this technology to field trials and application.

2 Scope

This study aims to comprehensively assess the effectiveness of ZDC in mitigating the negative effect of ageing on the fatigue and low-temperature performance of bitumen and asphalt mixtures. Bitumen was firstly modified with ZDC at two percentages, 3% and 5% respectively. Subsequently, both bitumen and mixtures were subjected to short-term and long-term ageing. Linear amplitude sweep (LAS) tests and low-temperature frequency sweep tests were conducted to evaluate the fatigue and low-temperature performance of bitumen. Then, Four-point bending (4 PB) tests, indirect tensile asphalt cracking tests (IDEAL-CT) at 25 °C and −10 °C were carried out to evaluate the performance of mixtures. The results were analysed using an ageing degree index, which compared to what extent bitumen and mixtures were aged. Furthermore, an efficiency index was employed to characterise the antioxidation efficiency of ZDC in relation to different properties. Overall, this study provides a comprehensive assessment of the efficiency of the antioxidant and provides critical data to guide and facilitate the potential application of such technology.

3 Materials and methods

3.1 Materials

3.1.1 Bitumen and asphalt mixture

The bitumen used in this study was an unmodified 70/100 penetration grade bitumen, the basic properties of bitumen are listed in Table 1. The SARA fractions i.e. the content of saturates, aromatics, resins and asphaltenes are also illustrated.

The aggregates in this study were limestone. The filler was a limestone powder. The bitumen-aggregate ratio was 5.0% (binder content 4.8%). The asphalt mixture was an AC-20 mixture with maximum nominal size of aggregates being 19 mm, and the gradation curve is shown in Fig. 1.

3.1.2 Antioxidant

The antioxidant employed in this study was Zinc Diethyldithiocarbamate (ZDC) provided by Shanghai Macklin Biochemical Technology Co., Ltd. The properties of ZDC are listed in Table 2.

3.2 Testing methods

3.2.1 Modification of bitumen

The antioxidant with dosages of 3% and 5% by mass of bitumen were blended with the neat binder using

Table 1 Basic properties of bitumen used in this study

Property	Penetration (0.1 mm)	Softening point (°C)	PG	Ductility (10 °C) (cm)	SARA
Value	89	49	PG 64–22	> 100	6:47:24:23

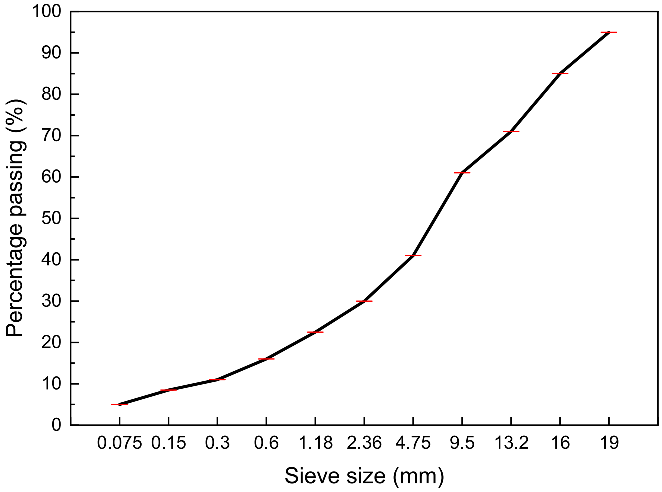
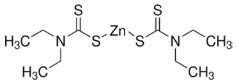


Fig. 1 Gradation curve of asphalt mixtures

Table 2 Details of the antioxidant used in this study

Purity	Density (g/cm^3)	Formula	CAS No	Chemical structure	Picture
98%	1.48	$C_{10}H_{20}N_2S_4Zn$	14,324–55-1		

a propeller type mixer. Based on the melting point of the antioxidant, the blending was carried out at 190 °C for a period of 20 min followed by reduced temperature blending at 165 °C for 40 min at 600 rpm [28, 32].

3.2.2 *Ageing of bitumen*

Both neat and ZDC-modified bitumen were subjected to short-term and long-term ageing. For the short-term ageing of bitumen, the rolling thin film oven tests (RTFOT) were carried out at 163 °C for 85 min as per ASTM D2872-22 [38]. Subsequently, the residue was subjected to the pressure ageing vessel (PAV) for long-term ageing at 100 °C and 2.1 MPa for 20 h in accordance with ASTM D6521-22 [39]. In addition to the standard PAV ageing, the bitumen was also subjected to extended ageing durations of 40 and 60 h.

3.2.3 *Bitumen tests*

The rheological properties of bitumen were tested using an Anton Paar M102e DSR. Linear amplitude sweep (LAS) test were carried out at 25 °C to evaluate the fatigue performance of bitumen in accordance with AASHTO T391-20 [40]. The low-temperature DSR tests using 4 mm parallel plates with a gap of 2 mm were performed for a frequency sweep at frequencies ranged from 0.1 rad/s to 100 rad/s with 10 readings per decade at temperatures of −6, −12, −18, −24 and −30 °C. The applied strain was 0.1% to ensure the bitumen tested were in the linear viscoelastic region (LVER) [41, 42]. For all bitumen tests, two replicates were performed.

3.2.4 *Ageing of asphalt mixture*

The loose asphalt mixtures were placed in a metal tray and conditioned in an oven in the laboratory at 135 °C for two hours to simulate the short-term ageing as per

AASHTO R30-22 [43]. Afterwards, the loose mixtures were separated into two sets. The first set was subjected to the manufacture of cylindrical specimens and asphalt slabs. The second set was conditioned in the oven at 95 °C for 120 h to simulate the long-term ageing in accordance with NCHRP 09–54 report and literatures [4, 44]. The long-term aged loose mixtures were then compacted for testing. The manufacture of specimens using short-term and long-term aged loose mixtures was identical.

3.2.5 *Manufacture of asphalt slabs for beams and cylindrical specimens*

Loose mixtures, conditioned through short-term and long-term ageing, were compacted into slabs using a segmented rolling compactor in accordance with ASTM D8079-23 [45]. Each slab measured 500 mm × 500 mm × 70 mm. Subsequently, beams with dimensions of 380 mm × 63 mm × 50 mm were prepared from the slabs for four-point bending fatigue tests. Cylindrical specimens for the indirect tensile asphalt cracking tests (IDEAL-CT) were prepared separately using a Marshall compactor in accordance with ASTM D6926-20 [46].

3.2.6 *Asphalt mixture tests*

The 4 PB fatigue tests were carried out to evaluate the fatigue performance of asphalt mixtures as per ASTM D8237-21 [47]. The testing temperature was 25 °C. Prior to testing, the specimens were placed into the environmental chamber at the same temperature for two hours of conditioning. The loading frequency was 10 Hz, and the loading strain amplitude was 500 με. The IDEAL-CT was carried out at 25 °C with loading rate of 50 mm/min for the intermediate-temperature tests and 12.5 mm/min for the low-temperature tests at −10 °C as suggested in NCHRP Project D9-29 FY’01 [48]. Prior to testing, the specimens were placed into a freezer at the same temperature for four hours of conditioning. For all mixture tests, three replicates were performed.

3.3 Data analysis

3.3.1 *Fatigue performance of bitumen*

In T391-20 standard, the definition of damage failure of bitumen corresponds to the 35% reduction in undamaged $|G^*|\sin\delta$. The damage accumulation at failure (D_f) is defined as per Eq. (1).

$$D_f = \left(0.35\frac{C_0}{C_1}\right)^{\frac{1}{C_2}} \tag{1}$$

where, C_0 is the average value of $|G^*|\sin\delta$ from the 0.1% strain interval, in MPa, and C_1 and C_2 are the curve-fit coefficients derived from Eq. (2) [49].

$$\log(C_0 - |G*|\sin\delta) = \log(C_1) + C_2\log(D) \tag{2}$$

The fatigue life can be calculated as per Eq. (3) with the interest strain γ.

$$N_f = A_{35}(\gamma)^B \tag{3}$$

where, the parameters A_{35} and B are material-specific parameters for calculating the fatigue life of bitumen, as shown in Eqs. (4) and (5).

$$A_{35} = \frac{f(D_f)^k}{k(\pi I_D C_1 C_2)^\alpha} \tag{4}$$

$$B = -2\alpha \tag{5}$$

where, f is the loading frequency, which is 10 Hz, I_D is the initial value of $|G^*|$ from the 1% applied strain intervals, in MPa, and k can be calculated using Eq. (6).

$$k = 1 + (1 - C_2)\alpha \tag{6}$$

3.3.2 *Low-temperature performance of bitumen*

Previous research efforts have verified that the 4 mm DSR method has good repeatability and can be used as a surrogate for BBR for low-temperature performance characterisation of bitumen [50–52]. The interpretation of 4 mm DSR data includes converting it to BBR data based on linear viscoelastic theory, empirical equations, or directly determining the equivalent cut-off values [53–55]. Komaragiri et al. concluded that a DSR-based G^* value of less than 160 MPa can be used in lieu of BBR stiffness (S) requirement of less than 300 MPa.

Phase angle δ of greater than 25° can be used in lieu of creep rate (*m-value)* requirement of 0.300 or higher [42]. This study adopted this method to determine the critical temperatures using Eqs. (7) and (8).

$$T_{C,S} = T_1 + \frac{(T_1 - T_2)(\log 160 - \log G_1^*)}{\log G_1^* - \log G_2^*} - 10 \tag{7}$$

$$T_{C,m} = T_1 + \frac{(T_1 - T_2)(25 - \delta_1)}{\delta_1 - \delta_2} - 10 \tag{8}$$

where $T_{C,\ S}$ and $T_{C,\ m}$ are the critical temperature controlled by stiffness (complex modulus in this study) and m-value (phase angle in this study), $G_1{}^*$ and $G_1{}^*$ are the complex moduli which pass or fail the criterion (less than 160 MPa), δ_1 and δ_2 are the phase angles which pass or fail the criterion (greater than 25°), T_1 and T_2 are the temperatures at which the complex modulus or phase angle passes or fails the criteria. In addition to the critical temperatures, another parameter, ΔTc, defined as the difference between these two critical temperatures, can also be employed to examine the low-temperature performance of bituminous materials [56].

3.3.3 *Fatigue life of asphalt mixture measured by four-point bending beam tests*

The fatigue life of asphalt mixture corresponds to the number of cycles at the peak value of normalised stiffness times normalised cycles. The normalisation of stiffness and cycle number was carried out using Eq. (9).

$$\hat{S} \times \hat{N} = \frac{S_i \times N_i}{S_0 \times N_0} \tag{9}$$

where, S_i is the flexural beam stiffness at cycle i, in MPa, S_0 is the initial flexural beam stiffness at approximately 50 cycles, in MPa, N_i is the cycle i, and N_0 is the cycle number where the initial stiffness is estimated, which is 50 in the tests for this study.

3.3.4 *Indirect tensile strength*

In accordance with ASTM D6931-17 [57], the IDEAL-CT strength is calculated using Eq. (10).

$$S_t = \frac{2000 \times P}{\pi \times t \times D} \tag{10}$$

where, S_t is the indirect tensile strength, in kPa, P is the maximum load, in N, t is the specimen

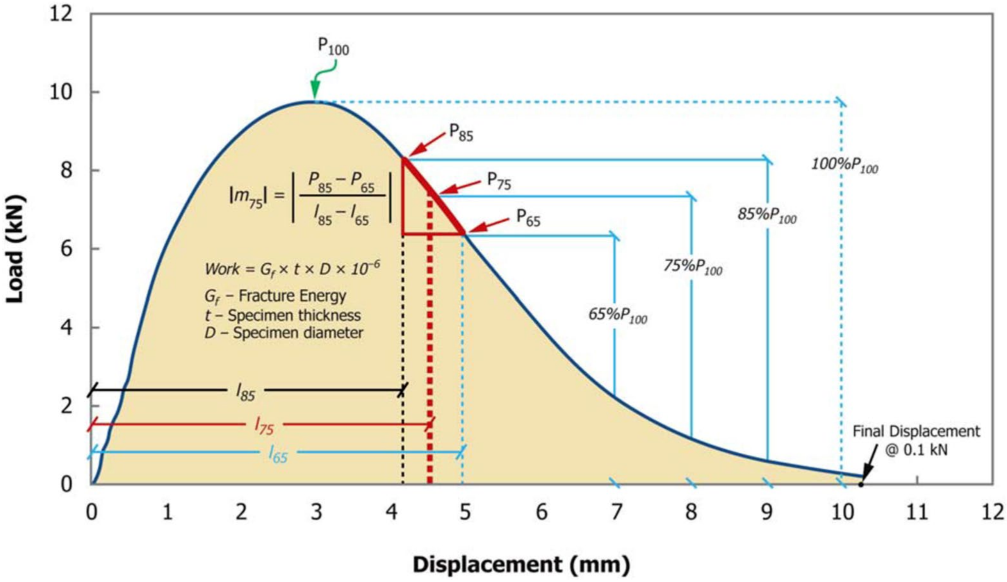


Fig. 2 Recorded Load (*P*) versus Load-Line Displacement (*LLD*) curve [58]

height immediately before test, in mm, and D is the specimen diameter, in mm.

3.3.5 *Cracking tolerance index (CT_{index}) and other cracking-related indices of asphalt mixtures*

In accordance with ASTM D8225-19 [58], the work of failure (W_f) is calculated as the area under the load versus Load-Line Displacement (LLD) curve (Fig. 2) through the quadrangle rule provided in Eq. 11.

$$W_f = \sum_{i=1}^{n-1}\left[\left(l_{i+1} - l_i\right) \times P_i + \frac{1}{2} \times \left(l_{i+1} - l_i\right) \times \left(P_{i+1} - P_i\right)\right] \tag{11}$$

where, P_i is the load at the i step, in kN, l_i is the LLD at the i step, in mm.

Failure energy (G_f) is calculated using Eq. (12).

$$G_f = \frac{W_f}{D \times t} \times 10^6 \tag{12}$$

CT_{index} is calculated using Eq. (13).

$$CT_{index} = \frac{t}{62} \times \frac{l_{75}}{D} \times \frac{G_f}{|m_{75}|} \times 10^6 \tag{13}$$

where, l_{75} is the displacement at 75% of the peak load after the peak, in mm, and $|m_{75}|$ is the absolute value of the post-peak slope, in N/m.

Cracking resistance index (CR_{index}) is calculated using Eq. (14):

$$CR_{index} = \frac{G_f}{P_{100}} \tag{14}$$

where, P_{100} is the peak load of the tests.

Flexibility index (FI) is calculated using Eq. (15).

$$FI = \frac{G_f}{|m_{75}|} \times 0.01 \tag{15}$$

3.4 Evaluation of the effectiveness of the antioxidant

To quantitively evaluate the effectiveness of the antioxidant on varying properties of bitumen and asphalt mixtures, a uniform ageing degree index (ADI) and Efficiency Index (EI) were employed in this study. Given that all bitumen and mixtures are subjected to short-term ageing in practice, the criterion of ageing degree index is normalising the ageing degrees of bitumen and mixtures, with the properties of short-term aged samples serving as a reference or baseline. The definitions of ADI and EI are presented in Eqs. (16) and (17) for all properties. The ADI represents the rate of ageing increase of long-term aged specimens with the reference of short-term aged specimens. The EI represents the ageing mitigation effect of ZDC.

$$ADI = \left|\frac{X_2 - X_1}{X_1}\right| \tag{16}$$

where, X_1 is any parameter after short-term ageing, and X_2 is the corresponding parameter after long-term ageing.

$$EI = ADI_{unmodified} - ADI_{modified} \tag{17}$$

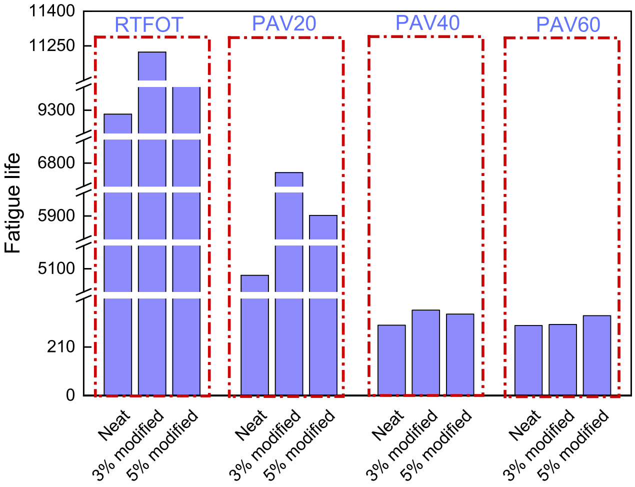
4 Results and discussion

4.1 Characterization of fatigue performance of bitumen

Fatigue cracking of asphalt pavements is one of the most common distresses caused by the accumulation of repeated axle loadings [59], which subsequently induces more severe distresses like map cracking [12, 60]. Based on the test results of linear amplitude sweep (LAS), the fatigue life of bitumen at any strain level of interest can be calculated. The AASHTO T391-20 standard recommends calculating the fatigue life of bitumen at strain levels of 2.5% and 5%. It has been reported that the fatigue life of bitumen at relatively higher strain levels is more correlated with the fatigue life of mixtures [61, 62]. Therefore, this study calculated the fatigue life of bitumen at the strain level of 5%, the results are shown in Fig. 3.

As observed from Fig. 3, the fatigue life of ZDC-modified bitumen was seen to be greater than that of neat (unmodified) bitumen, regardless of ageing levels, indicating that the addition of the antioxidant could enhance the fatigue resistance of bitumen. Consistent with previous studies, ageing reduces the fatigue life of bitumen when the applied strains are relatively higher [20, 63]. It is noteworthy that after 40 h of PAV ageing, the fatigue lives of all bitumen decreased dramatically, with the fatigue lives of modified bitumen being consistently higher than that of

Fig. 3 Fatigue life of bitumen at strain level of 5%



neat bitumen. For example, the fatigue lives of modified bitumen with dosages of 3% and 5% of the antioxidant were 21.3% and 15.7% higher than that of the neat binder after 40 h of PAV ageing, respectively. The fatigue lives of bitumen after 60 h of PAV ageing were similar to that after 40 h of PAV ageing. This observation has been reported previously as bitumen tends to reach a relatively stable status after certain levels of ageing. Therefore, progressively increased ageing would have limited impact on the properties of severely aged bitumen after it reaches generally stable values [18]. Nevertheless, it was still observed that the fatigue life of ZDC-modified bitumen was higher than that of unmodified bitumen after 60 h of PAV ageing. As the fatigue life of bitumen is strain-dependent, the fatigue lives of long-term aged bitumen at varying strain levels are shown in Fig. 4.

As illustrated in Fig. 4a, in double-logarithmic scale, the fatigue lives of long-term aged bitumen were linearly correlated with applied strains, with higher strain resulting in lower fatigue life [18]. Clearly, at all strain levels, the fatigue life of modified bitumen was greater than that of unmodified bitumen. However, the difference was insignificant, especially under the extended PAV ageing durations. It is reported that the slope of the fatigue life versus strain curves represents the strain-dependence of bitumen, while greater absolute value of the slope indicates

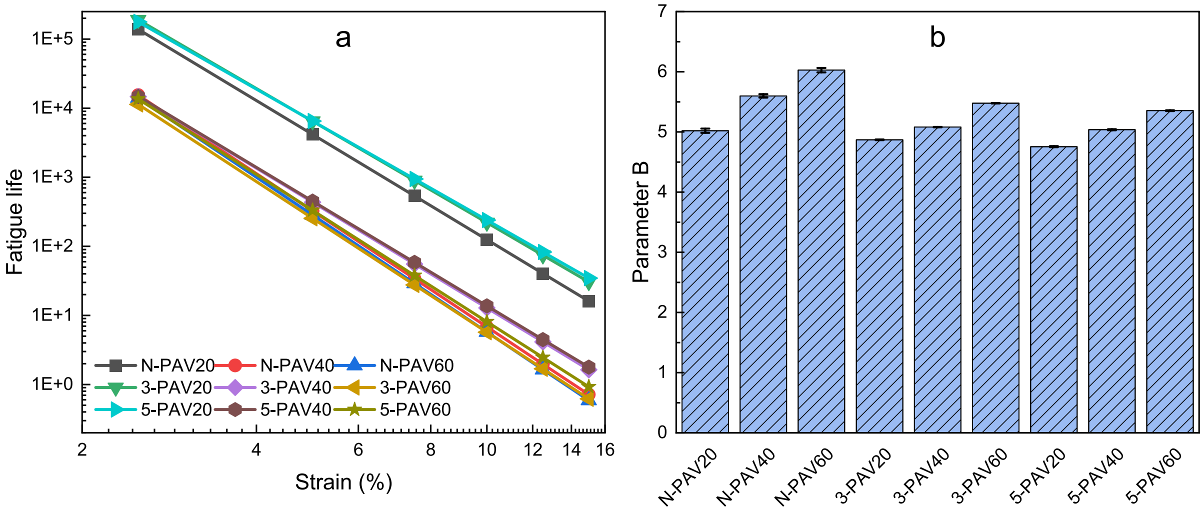


Fig. 4 Fatigue properties of long-term aged bitumen **a** fatigue life versus strain, and **b** Parameter B

higher strain-dependence, which is less desirable [64]. The slope is related to the parameter *B*, as calculated by Eq. (7). The results of parameter B are shown in Fig. 4b. It was observed that more severe ageing led to greater value of parameter B, thereby increasing the strain-dependence of bitumen. This increase was seen to be hindered by introducing the antioxidant to the bitumen, as the values of parameter *B* for modified bitumen were always lower than that of neat bitumen at the same ageing degrees.

4.2 Low-temperature performance of bitumen

The critical temperatures controlled by stiffness and m-value were calculated, as shown in Fig. 5. It was seen that ageing resulted in higher critical temperatures of bitumen, which is consistent with previous studies [18].

For the critical temperatures of virgin and short-term aged bitumen, there were no apparent differences between the unmodified and modified bitumen. However, with progressively increased ageing levels, significant differences could be observed between the critical temperatures of modified and unmodified bitumen. The ZDC-modified bitumen had lower critical temperatures compared to the unmodified bitumen when bitumen was severely aged, e.g. after 40 h and 60 h of PAV ageing. This difference was more pronounced for the critical temperatures controlled by m-value. After 60 h of PAV ageing, the $T_{C,m}$ of modified bitumen with dosages of 5% and 3% were 4.3 °C and 2.1 °C lower than that of unmodified bitumen, indicating that the incorporation of antioxidant could effectively mitigate the deterioration of low-temperature performance of bitumen caused by ageing, thereby improving the thermal cracking resistance of bitumen.

The difference between the critical temperatures controlled by stiffness and creep rate is defined as ΔTc [65]. If the value of ΔTc is positive, the lower PG of bitumen is stiffness controlled while if the value is negative, then the lower PG is creep rate controlled [17]. Additionally, it has been documented that ageing leads to more negative values of ΔTc [17]. The ΔTc results of bitumen are shown in Fig. 6.

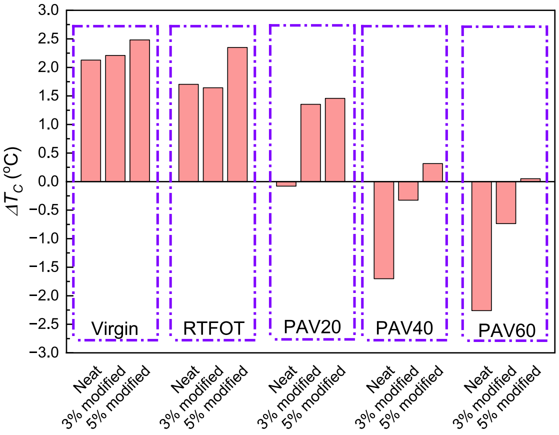


Fig. 6 ΔTc of bitumen with varying ageing situation

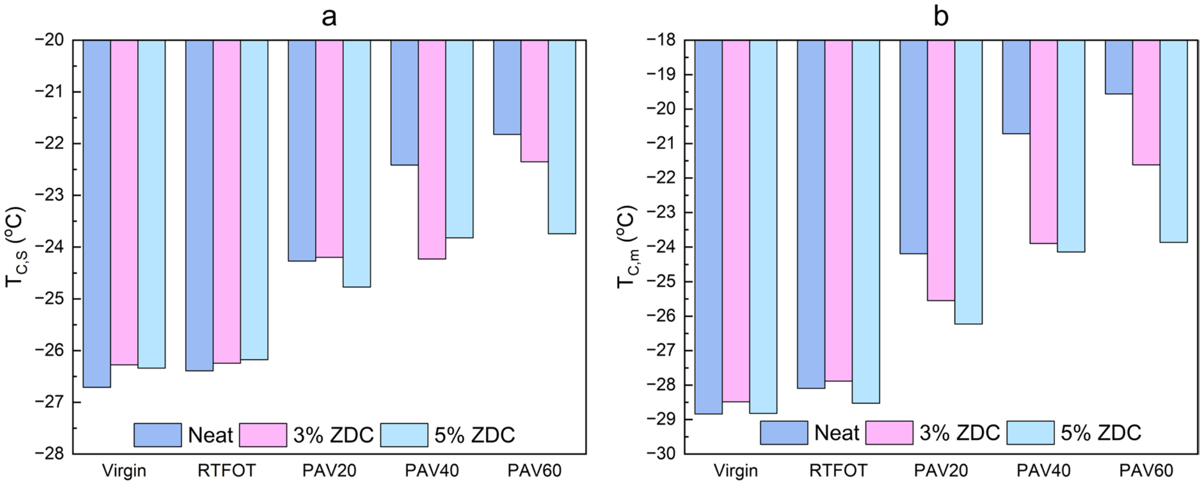


Fig. 5 Critical temperatures of bitumen **a** stiffness-controlled, and **b** m-value controlled

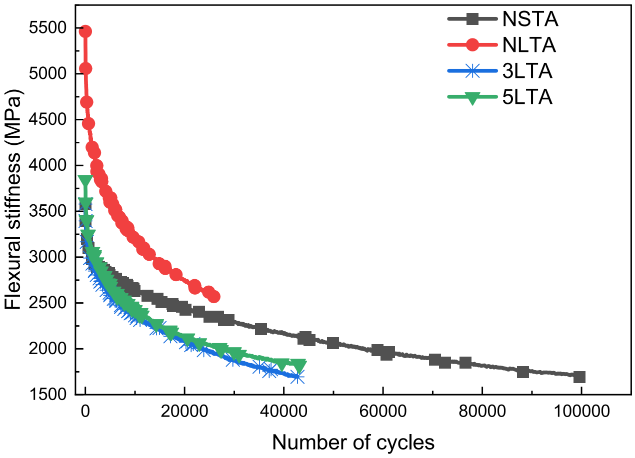
It was seen that for unaged and short-term aged bitumen, ΔTc was positive, indicating that the low-temperature performance of bitumen is stiffness controlled. However, for the long-term aged bitumen, the ΔTc of neat bitumen turned to negative and became more negative with ageing while that for modified bitumen remained positive after 20 h of PAV ageing. The ΔTc of modified bitumen with dosage of 3% turned to negative when the duration of PAV ageing reached 40 h while that for the modified bitumen with dosage of 5% remained positive even when the ageing duration reached 60 h. The results showed that the incorporation of antioxidant slowed down the rate of the ΔTc turning from positive to negative after long-term ageing. At a given low temperature performance grade, a more negative value of ΔTc indicates that the ability of bitumen to shed stress is lower, which makes it less flexible, more brittle, and more prone to cracking [17, 65, 66]. Therefore, the addition of ZDC sufficiently reduced the cracking susceptibility of bitumen. Higher dosage showed higher sufficiency, as it could be seen that the ΔTc of bitumen with higher dosage of ZDC was less negative. Overall, the low-temperature performance evaluation of bitumen showed clear evidence that the modified bitumen had better thermal cracking resistance. The improvement of low-temperature performance is caused by slowing down the increase of stiffness and loss of ductility [30]. Moreover, higher dosage of ZDC resulted in higher mitigating effectiveness of ageing-induced performance deterioration at low temperatures.

4.3 Fatigue performance of mixtures

The stiffness evolution trends of the mixtures measured from the four-point bending tests are shown in Fig. 7. As seen in Fig. 7, the initial stiffness of neat asphalt mixtures after long-term ageing (NLTA) was much higher than that of the mixtures after short-term ageing (NSTA), which was consistent with previous studies [67, 68]. However, the incorporation of antioxidants mitigated the stiffening effect caused by long-term ageing, as the initial stiffnesses of long-term aged asphalt mixtures with 3% and 5% of ZDC were significantly lower than that of the unmodified asphalt mixtures. Previous studies have shown that the stiffness measured by 4 PB in the laboratory has fairly good agreement with that measured in the field [69], therefore, the addition of ZDC effectively reduced the stiffness of asphalt mixture caused by long-term ageing, thereby reducing the cracking susceptibility and increasing the service life of asphalt pavements [70].

Moreover, it was seen that the evolution of stiffness showed a two-step trend. For the first step, the stiffness decreased dramatically while during the second step the stiffness decreased slowly. Figure 7 illustrated that the decrease rate of stiffness of neat long-team aged mixtures was highest, while the antioxidant slowed down the decrease rate of stiffness for modified asphalt mixtures, which improved the longevity of asphalt pavements. To quantitatively evaluate the effect of the antioxidant on fatigue performance of asphalt mixture, the fatigue life of each

Fig. 7 Stiffness evolution trends of asphalt mixtures



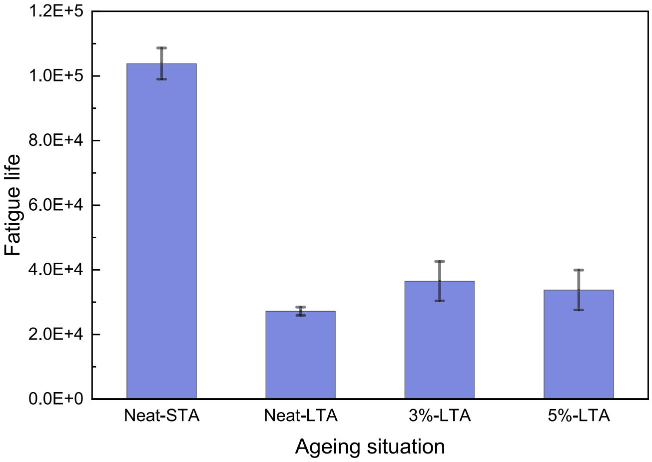


Fig. 8 Fatigue life of asphalt mixtures

asphalt mixture was calculated as per Eq. (13), the results are shown in Fig. 8.

As shown in Fig. 8, long-term ageing is significantly detrimental to the fatigue life of asphalt mixtures [13], as the fatigue life of neat long-term aged mixture was only one-fourth of that for neat short-term aged mixtures. The introduction of the antioxidant increased the fatigue life of long-term aged mixtures for around 30%, by mitigating the ageing-induced stiffening [70]. Therefore, the asphalt pavements with the antioxidants are expected to have longer service life and better durability. However, the difference between the fatigue lives of modified mixtures with varying dosages was insignificant, which suggested that the dosage had limited impact on the fatigue life of asphalt mixture as per this evaluation criteria. Moreover, the effect of ZDC on the fatigue life of asphalt mixtures aligned with its effect on the fatigue life for bitumen, providing reliable evidence that the incorporation of ZDC can extend the service life of asphalt pavements.

4.4 Cracking tolerance of asphalt mixtures at intermediate temperature

Based on the IDEAL-CT tests, various parameters could be derived, such as failure energy (G_f), post-slope of LLD curve, indirect tensile strength (*ITS*), flexibility index (*FI*), cracking resistance index (CR_{index}), and cracking tolerance index (CT_{index}). The indices based on the intermediate temperature tests are shown in Fig. 9.

As illustrated in Fig. 9a, the failure energies (G_f) of long-term aged asphalt mixtures were higher than that of short-term aged mixtures, indicating that more energy was required before the specimens were damaged. Interestingly, the G_f of modified mixtures after long-term ageing were even higher than that of the unmodified mixture at the same ageing levels. The correlation between G_f and cracking resistance of mixtures is controversial as previous studies have reported that G_f of long-term aged mixtures could be either higher or lower than that of short-term aged mixtures [71]. Therefore, to avoid any arbitrary judgment of the antioxidant's effectiveness, G_f was used solely to illustrate changes in the properties of asphalt mixtures.

When it comes to the slope of the load–displacement curve post the peak load, as depicted in Fig. 9b, the slope of short-term aged mixture was the smoothest while that for the long-term unmodified mixture was steepest. The slope of the long-term aged, modified mixtures was higher than that of short-term aged mixtures while lower than that of long-term aged unmodified mixture. A steeper slope represents that cracks could be developed quickly after the initial damage occurring [72]. Therefore, the introduction of ZDC could effectively slow down the development of cracking during the long-term ageing process when the mixtures are subject to field service. Moreover, it was seen that the dosage of antioxidant did not play a crucial role in the effectiveness of the effect of ZDC as the slope of mixtures modified with varying dosages of ZDC were almost identical.

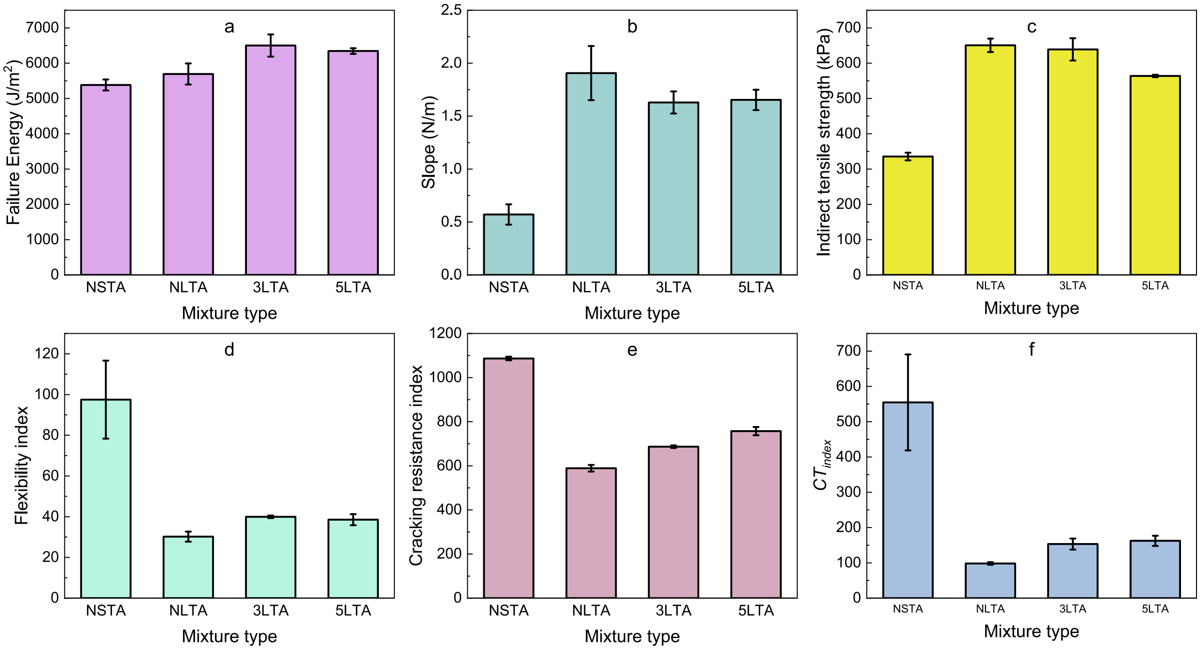


Fig. 9 Indices for the IDEAL-CT at intermediate temperature: **a** Failure energy (G_f), **b** Post-peak slope of load–displacement curve, **c** Indirect tensile strength (*ITS*), **d** Flexibility index (*FI*), **e** Cracking resistance index (CR_{index}), and **f** Cracking tolerance index (CT_{index})

For the indirect tensile strength of asphalt mixture specimens, it was observed from Fig. 9c that the short-term aged mixtures had the smallest strength while the long-term aged unmodified mixture had the largest strength. The strengths of antioxidant modified mixtures after long-term ageing resided between the short-term aged and unmodified long-term aged mixtures. It has been reported in previous studies that after long-term ageing, increase in stiffness could result in the increase of strength [73]. The introduction of the antioxidant can mitigate the stiffening effect caused by ageing, thereby reducing the strength of asphalt mixtures.

When it comes to the three cracking-related indices, e.g. *FI*, CR_{index} and CT_{index}, as illustrated in Fig. 9d–f, three indices followed identical evolution trend: short-term aged mixture had the best flexibility and cracking resistance/tolerance, while those for long-term aged unmodified mixture had the least.

4.5 Cracking tolerance of asphalt mixture at low temperature

Thermal cracking is the predominant distress in cold climates, causing transverse cracks perpendicular to the direction of traffic [74]. The results of IDEAL-CT tests carried out at −10 °C are shown in Fig. 10. As illustrated in Fig. 10a, at low temperature, e.g. −10 °C, the failure energies of mixtures showed opposite trends compared to those at intermediate temperature. As mentioned earlier, the failure energy is considered to be arbitrary and should not be correlated with the cracking resistance of mixtures [71]. For other parameters, the effect of the antioxidant on the low-temperature cracking resistance was identical with that at intermediate temperature. The primary difference between the low-temperature cracking resistance and intermediate temperature cracking resistance was the antioxidant showed much higher effectiveness in mitigating the negative effect of ageing at low temperature. This trend is in line with the obtained results at the binder scale. At intermediate temperatures, the incorporation of the antioxidant could improve the

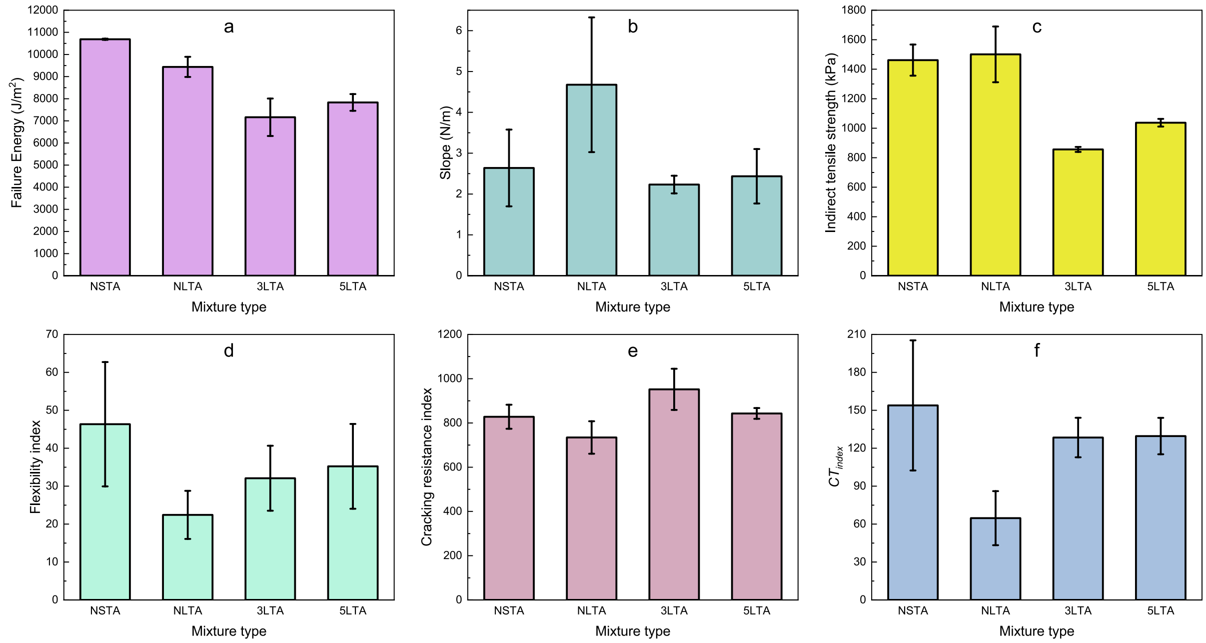


Fig. 10 Indices for the IDEAL-CT at low temperature: **a** Failure energy (G_f), **b** Post-peak slope of load–displacement curve, **c** Indirect tensile strength (*ITS*), **d** Flexibility index (*FI*), **e** Cracking resistance index (CR_{index}), and **f** Cracking tolerance index (CT_{index})

cracking resistance of asphalt mixture slightly, however, at low temperature, the cracking resistance of long-term aged asphalt mixture could be improved significantly. Some of the indices of the modified mixture after long-term ageing even were comparable to those for mixtures after short-term ageing. Therefore, the antioxidant could effectively mitigate the cracking-related distress of asphalt pavements in cold areas, thereby extending the service life of pavements.

The change in stiffness caused by ageing has a more significant influence on the low-temperature performance than on the intermediate-temperature performance of bitumen and asphalt mixtures.[75]. At low temperatures, asphalt mixtures tend to become very stiff and brittle, making them less able to resist loading and more prone to cracking. As mentioned earlier, the incorporation of ZDC could significantly reduce the stiffness of mixtures caused by long-term ageing, thereby improving the low-temperature performance more efficiently.

4.6 Effectiveness evaluation of the antioxidant

The effectiveness of ZDC was assessed by calculating the ageing degree index (*ADI*) and effectiveness index (*EI*). Firstly, a multivariate analysis of variance (MANOVA) was conducted under the assumption of a multivariate normal distribution. In the statistical analysis, the *ADI* was analysed in four categories: binder performance at low-temperature (Binder-LT), binder performance at intermediate-temperature (Binder-IT), mixture performance at low-temperature (Mixture-LT), and mixture performance at intermediate-temperature (Mixture-IT). The results of the MANOVA are shown in Fig. 11.

Firstly, it was observed from Fig. 11 that the ageing degree indices of ZDC-modified bitumen and mixtures were lower than those of unmodified specimens, indicating that the incorporation of the antioxidant could effectively and comprehensively mitigate the ageing degrees of both bitumen and mixtures. Afterwards, every two sets of data were compared within the same category, e.g., the comparison between unmodified specimens and specimens with 3% ZDC, comparison between unmodified specimens and specimens with 5%

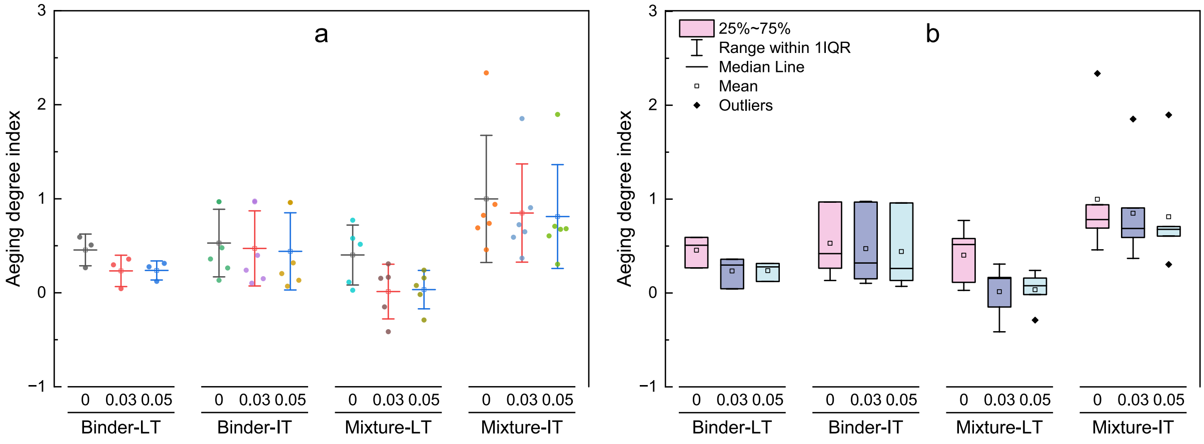


Fig. 11 MANOVA results: **a** Data point chart, and **b** box chat

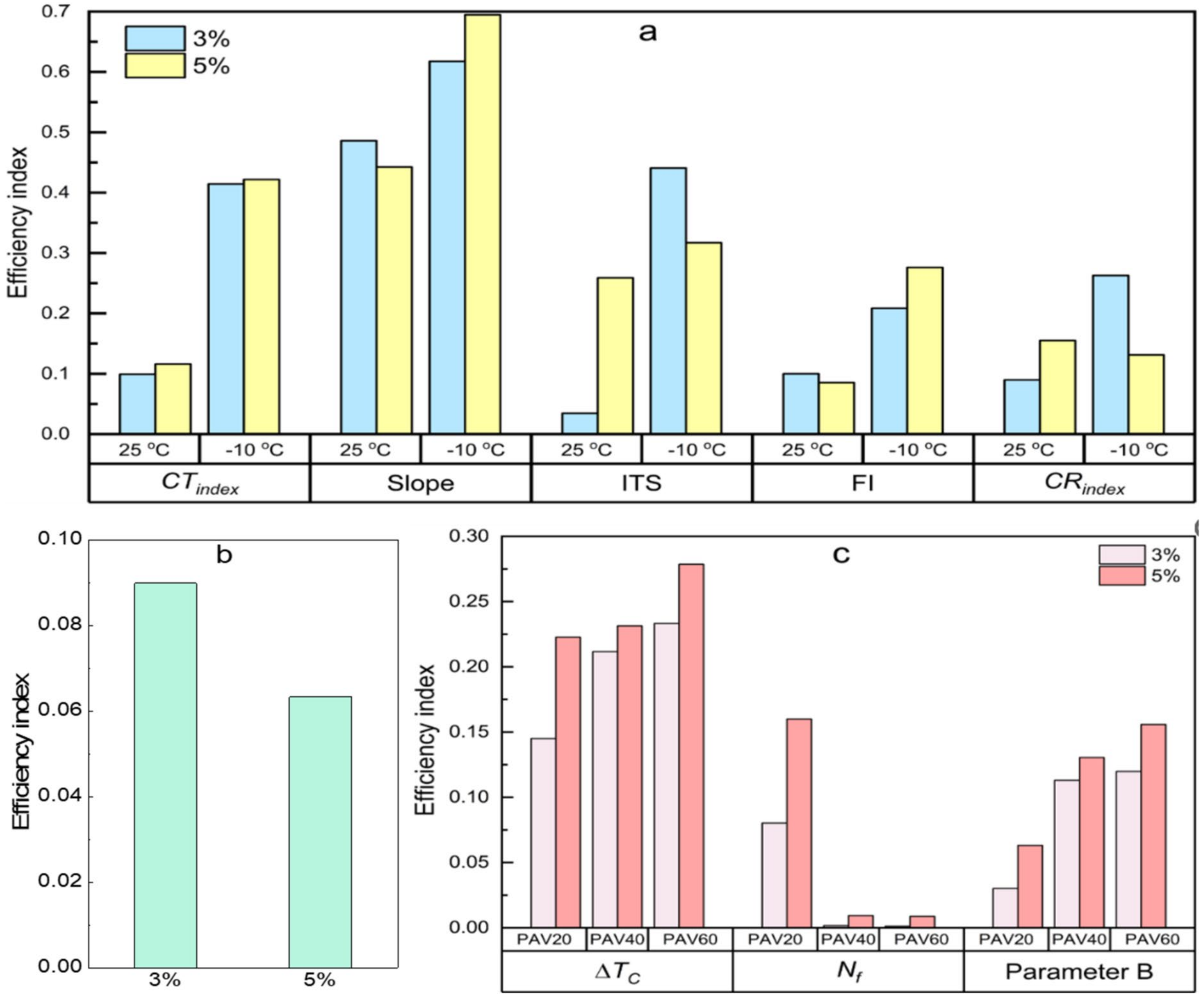


Fig. 12 Efficiency indices for **a** IDEAL-CT tests, **b** Four-point bending test, and **c** Bitumen tests

ZDC, and comparison between specimens with 3% and 5% ZDC. It was observed that while the ageing degree indices of modified bitumen and mixtures were lower than those of the unmodified counterparts, these differences were not statistically significant at a significance level of 0.05. One of the reasons for the insignificant differences could be attributed to the variation of ageing-mitigating effect of the antioxidant. For some properties in the same category, the antioxidant showed strong effectiveness, however, for other properties, only slight improvements of antioxidation effect could be observed, as analysed earlier.

Furthermore, the efficiency of the antioxidant was analysed and compared, as shown in Fig. 12. Positive values of *EI* indicate positive effectiveness of mitigating the effect of ageing. As observed in Fig. 12, the efficiency indices for all properties were positive, suggesting that the introduction of the antioxidant could effectively and systematically slow down the performance deterioration of bitumen and asphalt mixtures caused by ageing. It is noteworthy that the effectiveness was not directly correlated with the dosage of the antioxidant. As for some properties, the *EI* at dosage of 3% was higher than those at 5%, while for others opposite trends were observed. This phenomenon occurred for both bitumen and mixtures.

Overall, the ageing-mitigation effectiveness of ZDC was found to be higher at the mixture scale compared to bitumen scale. The performance of mixtures in relation to performance properties improved from 9% to up to 69%, while bitumen showed an improvement from 1 to 28%. Additionally, the effectiveness for low-temperature performance was greater than the fatigue performance. The improvement in low-temperature properties ranged from 13 to 69%, whereas the improvement in fatigue performance ranged from 1 to 44%. Several factors may contribute to these phenomena. Firstly, mixtures may have undergone more ageing compared to bitumen, considering the overall ageing durations. ZDC was observed to be more effective for highly aged bitumen, thereby possibly rendering it more effective for mixtures than for bitumen. Secondly, low-temperature performance is more sensitive to changes in stiffness [75]. As ZDC significantly reduced the increase in stiffness during ageing, therefore, it has more significant influence on the low-temperature performance of bitumen and mixtures.

5 Key findings and conclusions

This paper systematically characterised the ageing-mitigating effects for bitumen and asphalt mixtures of Zinc Diethyldithiocarbamate (ZDC) based antioxidant. Both ZDC-modified and unmodified bitumen and asphalt mixtures were subjected to short-term and long-term ageing. Afterwards, their low-temperature and fatigue performance were systematically evaluated. Based on the results, the following conclusions could be drawn.

(1) The incorporation of ZDC could mitigate the ageing effect, thereby slowing down the performance deterioration of both bitumen and asphalt mixtures. All properties of long-term aged bitumen and asphalt mixtures measured at low temperature and intermediate temperature could be improved with the addition of ZDC. The improvement in performance ranged from 1 to 69%.

(2) The ageing-mitigation efficiency of ZDC was more pronounced for the low-temperature performance (13–69%) of bitumen and asphalt mixtures compared to the intermediate temperature performance (1–44%).

(3) Though the ageing could be mitigated by the incorporation of ZDC, these differences between modified and unmodified samples were not statistically significant at a significance level of 0.05. This could be attributed to the variation of the ageing-mitigation effectiveness for different performance related properties of bitumen and asphalt mixtures.

(4) The dosage of ZDC was not directly correlated with the ageing-mitigation effectiveness of ZDC, as the lower dosage showed either higher efficiency of lower efficiency, for both bitumen and asphalt mixtures.

Although this study comprehensively evaluated the efficiency of the antioxidant in both bitumen and mixture levels, there are still some limitations requiring further investigation. Firstly, only one bitumen and one mixture were assessed, which limits its applicability to bitumen with diverse chemical compositions and mixtures with varying aggregates and gradations. Therefore, it is suggested to include more bitumen and mixtures in future investigations. Secondly, this study was a performance-based evaluation, without identifying the mechanisms of antioxidation, which requires further in-depth investigation. Moreover, ageing situations in the lab and field might be different, it is suggested to evaluate the efficiency of the antioxidant in field situations by means of long-term performance monitoring. Lastly, issues pertinent to industrial application, such as life cycle assessment (LCA), life cycle cost assessment (LCCA), health, safety and environment (HSE) concerns, and the recyclability of antioxidant-modified asphalt pavements, should be addressed in future studies.

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Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

Data availability Data will be made available upon request.

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