



# Improvement in performance properties of asphalt using a novel boron-containing additive



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## HIGHLIGHTS

- Asphalt was modified novel boron-containing additive named cyclic borate ester (CBE).
- CBE additive was used into an asphalt binder in the ratios of 1%, 2%, 4% and 6% (w/w).
- Conventional and Superpave tests were applied.
- Base asphalt binder's temperature sensitivity was decreased.
- The additive enhanced the elastic property of asphalt in addition to increasing its resistance towards aging and fatigue.

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## ABSTRACT

In current times, many additives are used to improve the performance characteristics of asphalt. In this study, a new additive that contains boron, given the short name of cyclic borate ester (CBE), was chemically synthesised in laboratory conditions. It was added to the asphalt binder as 1%, 2%, 4% and 6% ratios of asphalt by weight, and modified asphalt samples were produced. The effects of CBE on the conventional properties of asphalt binder (penetration, softening point, ductility, flash point, Fraass breaking point) were examined using a dynamic shear rheometer, rotational viscosity, a rolling thin film oven, a pressure aging vessel and bending beam rheometer test methods. Results showed that the use of CBE additive increased the hardness, softening point, viscosity, flash-point value and rutting resistance of asphalt binder and decreased temperature sensitivity. CBE also enhanced elastic responses (increased complex shear modulus and decreased phase angle), aging resistance and low-temperature cracking resistance and did not change the cohesion property.

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## 1. Introduction

Many researchers have modified asphalt by adding different additives at different ratios, in an attempt to improve pavement performance and decrease deformation of asphalt pavement caused by growing traffic loads and environmental and climatic conditions. Many types of modifiers have been studied. For example, styrene-butadiene-styrene (SBS) has been found to increase the elasticity of asphalt [1–3] and for this reason improve fatigue and cracking behaviours [4–6] and develop high-temperature stability by increasing the complex modulus [7–9], and enhancing rutting resistance [10–12] and aging resistance [13]. Another modifier, ethylene-vinyl acetate (EVA), has been shown to enhance the physical [14,15] and rheological [16,17] features of asphalt and improve fatigue cracking of road pavement by increasing elasticity

at low temperatures [18,19], as well as increasing viscosity features by changing the microstructure of asphalt dramatically [20]. It has been reported that the process of asphalt modification by means of SBS and EVA polymers improves asphalt's physical features [21]. It has also been determined that low-density polyethylene (LDPE) improves the low-temperature properties of asphalt, and the fracture strength of asphalt concrete was increased by enhancing LDPE concentration [22,23]. The additives poly(methyl methacrylate) (PMMA) and aluminium trihydrate (ATH) have been reported to improve viscoelastic and physical properties and decrease temperature and oxidation sensitivity [24]. Additionally, it has been determined that crumb rubber, another potential additive, improves pavement performance [25,26], permanent deformation and high-temperature properties [27] by contributing to the asphalt mixture's viscosity and elasticity [28]; however, performance against cracking formed at low temperatures was not good [29]. Furan resin modification has also

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been shown to significantly enhance the resistance of asphalt to rutting and flow, and improve the complex modulus [30]. Finally, it has been stated that boron-containing additives increase asphalt binder hardness, wheel rutting resistance, elastic property and viscosity [31].

The aim of this study was to improve asphalt binder performance properties using a novel boron-containing compound, cyclic borate ester (CBE) that was chemically synthesised under laboratory conditions, and to investigate the effects of the additive on the asphalt binder. In this context, conventional (penetration, softening point, ductility, flash point, Fraass breaking point), dynamic shear rheometer (DSR), rotational viscosity (RV), rolling thin film oven test (RTFOT), pressure aging vessel (PAV) and bending beam rheometer (BBR) experiments were performed on asphalt that was modified by CBE.

## 2. Materials and methods

### 2.1. Preparation of CBE additive

Three compounds were tried in the additive composition (Fig. 1): 1) ethyl-(E)-2-(1-ethoxyethylidene)hydrazine-1-carboxylate, 2) 4-(3-bromopropyl)-5-methyl-2,4-dihydro-3H-1,2,4-triazol-3-one and 3) 4-(3-hydroxypropyl)-5-methyl-2,4-dihydro-3H-1,2,4-triazol-3-one. These three compounds have been previously obtained through methods explained in the literature [31–34]. A drop of concentrated sulphuric acid was added to a mixture of compound 3 (10 mmol, 1.57 g), boric acid (10 mmol, 0.61 g) and 1,2-dihydroxypropane (20 mmol, 1.52 g), and then the mixture was refluxed for 18 h at 300 °C. Compound 4 was the viscous residue obtained from the cooled mixtures. Its systematic name is 5-methyl-4-(3-((4-methyl-1,3,2-dioxaborolan-2-yl)oxy)propyl)-2,4-dihydro-3H-1,2,4-triazol-3-one and its short name is cyclic borate ester (CBE). The additive can be produced industrially by a simple manufacturing process without requiring a special production technique. The typical reaction equation and CBE's visual appearance can be seen in Figs. 1 and 2.

### 2.2. Asphalt

In this study, B50/70 penetration-grade asphalt binder obtained from the Turkish Petroleum Refinery Corporation in Izmit was used. The asphalt binder's properties are listed in Table 1.

### 2.3. Preparation of modified asphalt

Modified asphalt samples with four different ratios were produced. These ratios were 1%, 2%, 4% and 6% (w/w). CBE was added to the base asphalt, liquefied and heated to 150 °C, in the previously mentioned ratios. Modified asphalt binder was produced by mixing for 15 min at 1000 rpm and 150 °C by means of a mechanical four-armed mixer.

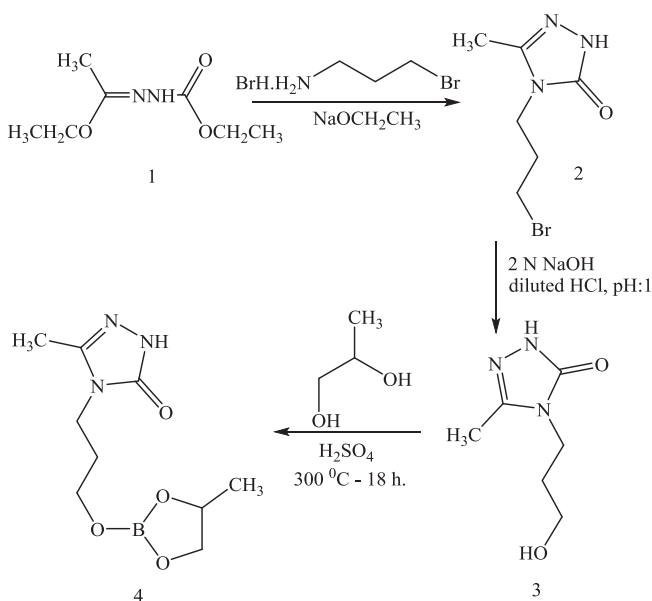


Fig. 1. General reaction equation of synthesised CBE additive.



Fig. 2. The visual appearance of CBE additive.

Table 1  
Properties of base asphalt.

Properties	Value	Specification
Penetration (25 °C, dmm)	57	ASTM D-5
Softening point (°C)	50	ASTM D-36
Ductility (25 °C, cm)	100+	ASTM D-113
Flash point (°C)	248	ASTM D-92
Specific gravity, (25 °C, g/cm <sup>3</sup> )	1.022	ASTM D-70
Viscosity at (135 °C, cP)	412.5	ASTM D-4402
Viscosity at (165 °C, cP)	112.5	ASTM D-4402

### 2.4. Conventional tests

Penetration is an experimental method applied to determine asphalt consistency. It is the depth a needle penetrates into the asphalt sample under 100 g at 25 °C for 5 s (ASTM D5) [35]. Increasing penetration value means decreasing and softening of the asphalt samples, whereas decreasing penetration measures the hardness and consistency. The softening-point test is an experiment that measures susceptibility of asphalt to temperature, as well as the temperature at which flow begins. In this experiment, steel balls (3.5 g/unit) were put onto an asphalt sample located in a standard ring. Those samples then were heated in a water bath at 5 °C/min. The softening-point value, the temperature when the balls touch the basement plaque covering the rings, was recorded (ASTM D36) [36]. Ductility can be defined as the length "cm unit" that a standard briquette can be pulled from the asphalt cement without breaking at the determined temperature and acceleration. The bonding capability of asphalt is dependent on its ductility. Asphalt with a high ductility value are highly useful. The ductility device is filled with water at 25 °C and the asphalt sample is pulled with 5 cm/min acceleration horizontally (ASTM D113) [37]. Flash-point temperatures of the binders were determined using the Cleveland Open Cup test (ASTM D92) [38]. The Fraass break point was determined to measure the performance of the asphalt binder at low temperature (IP 80) [39].

### 2.5. Dynamic shear rheometer test

The dynamic shear rheometer (DSR) experiment is used to determine resistance against fatigue cracks and rutting of the asphalt. In this experiment, the properties at medium and high temperatures are defined. For unaged binders and aged binders, the Rolling Thin Film Oven Test (RTFOT) is used to determine the asphalt binder's resistance against rutting. In order to measure the fatigue behaviour of the binders, the binders are aged by means of the pressure aging vessel (PAV) method. For determining rutting resistance, the samples have 25 mm diameter and 1000 μ gaps. For determining fatigue resistance, the samples have 8 mm diameter and 2000 μ gaps. The DSR experiment characterises the viscous and elastic behaviours of the asphalt binder by means of determining the complex shear modulus ( $G^*$ ) and phase angle ( $\delta$ ).  $G^*$  is an indicator of total resistance of the asphalt binder against deformation caused by recurrent shear stresses. Both  $G^*$  and  $\delta$  values dramatically change with the temperature and loading acceleration of the asphalt binder. At the end of the DSR experiment, the resulting values of  $G^*$  and  $\delta$ , are used to determine the rutting resistance parameter,  $G^*/\sin\delta$ . An increase in  $G^*$  and a

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