



# Improving the aging resistance of bitumen by addition of polymer based diethylene glycol

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## H I G H L I G H T S

- Bitumen was modified with newly synthesized diethylene glycol based polyboron compound (DEGPB).
- DEGPB improves the physical properties and the short-term aging resistance of DEGPBMB.
- The oxidation rates of DEGPBMB is examined through FTIR.
- Marshall Stability of the bituminous mixtures was increased.
- The additive is recommended to improve the roadway performance.

## A R T I C L E I N F O

### Article history:

Received 7 August 2017

Received in revised form 14 January 2018

Accepted 1 March 2018

### Keywords:

Modified bitumen

Aging resistance

Diethylene glycol

FTIR

Marshall stability

## A B S T R A C T

This study deals with improving the short-term aging resistance of bitumen with a new material, a diethylene glycol based polyboron compound (DEGPB). The compound was synthesized chemically and used 50/70 penetration grade bitumen. The bitumens are aged by means of Thin Film Oven Test (TFOT). The physical and aging properties of bitumens are evaluated by rotational viscosity (RV), penetration, softening point, ductility test at 15 °C and 25 °C and the Marshall stability test. Also, bitumen's oxidation rate was evaluated by Fourier Transform Infrared Spectroscopy (FTIR), which is a direct methodological approach. In light of experimental studies reported in this study, it has been shown that the additive at all tested rates increases bitumen's aging resistance. Compared to original bitumen's properties, DEGPB increases viscosity and the softening point of bitumens while decreasing its penetration and ductility. The Marshall stability tests have shown that the stability of bituminous mixtures was improved by DEGPB. In conclusion, DEGPB additive decreased the aging of bitumens by 41.9.

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## 1. Introduction

Increased traffic volume, environmental and external factors, and flaws incurring during construction phase cause early deterioration of the road pavement. One of the most important causes underlying pavement deterioration is the aging of bituminous binders used in road pavements. The Main factor causing the deterioration of pavements is asphalt aging [1,8]. The aging can be defined as the increase of hardness due to the volatilization and oxidation of light components of bitumen materials in road pavement during construction and service time. Loss of oil components due to the absorption or volatilization, reaction of bitumen with atmospheric

oxygen, and finally molecular structuring (steric hardening) producing thixotropic effects [2]. Also, the aging was reported to significantly affects the chemical and rheological properties of bitumen [3]. The structural changes and the permanent hardening occurring as a result of aging that in turn causes loss in the bituminous binder's elasticity and thereby induce effects such as rutting, fatigue and cracking, temperature cracking and moisture sensitivity. In turn, this decreases the estimated service time and comfort. The aging of the bitumen is an ongoing process divided into short-term and long-term aging processes. Short-term aging occurs during storing, mixing, and the asphalt paving procedure. Long-term aging, on the other hand, occurs throughout the service time of bitumens. It is indicated that the aging occurring until the pavement is put in service constitutes the 60–70% of total aging. The aging of bituminous binder results in the increase of hardness or

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viscosity. Oxidized bituminous mixtures have lesser durability in terms of water resistance and moisture sensitivity compared to normal mixtures [4].

Many studies about modified bitumens and their improving aging resistance properties are available. Phosphoric and polyphosphoric acid were reported to increase the aging resistance of bitumens [5]. Zinc dialkyldithiophosphate additive can also be used to increase the bitumen's aging resistance [6,7]. The use of highly reclaimed rubber in SBS modified asphalt also improves the aging resistance and physical properties [8]. Microwave activated crumb rubber increases aging resistance and fatigue cracking resistance [9]. Henglong et al. had reported that the ultraviolet aging (UV) resistance of bitumen can be effectively improved by inorganic nanoparticles [10]. Organic layered double hydroxides (LDHs) intercalated with sodium dodecyl sulfate (SDS) and sodium dodecyl sulfonate (SDSO) were enhancing UV aging resistance of bitumen [11]. A novel anti-aging modifier containing surface modified nano-zinc oxide (nano-ZnO) and organic expanded vermiculite (OEVMT) modified bitumens had showed better aging and rutting resistance [12].

The purpose of this study was to investigate improving short term aging properties of bitumen and bituminous mixtures with a novel material diethylene glycol based synthetic polyboron (DEGPB). Rheological properties of bitumens were studied by conventional tests (Aging of original and modified bitumens were realized by TFOT (ASTM D1754), softening point (ASTM D 36), ductility at 25 °C and 15 °C (ASTM D113), and penetration (ASTM D5). In addition to this, to determine the additives' effect on bitumens aging resistance and performance properties, the Marshall stability test (ASTM D 6927) has been applied to all original and aged bitumens with additives. Moreover, the structural characteristics and oxidation aging were investigated by FTIR analysis method.

## 2. Materials and methods

### 2.1. Bitumen

Original bitumen of 50/70 penetration grade was obtained from The Turkish Petroleum Refinery Corporation. Properties of this bitumen were listed in Table 1.

In this study, 50/70 penetration grade bitumen, which is the most commonly used bitumen in hot mixture pavements in the road network of Turkish General Directorate of Highways in accordance with TS 1081 EN 12591 standard, was used and procured from The General Directorate of Highways' Güvercinlik establishment.

### 2.2. Aggregate

For the aggregate material, basalt materials, obtained from Genç Osman quarry in the Sakarya Region of Turkey were used. Physical and chemical properties can be seen in Table 2.

The grading properties of the aggregate for Marshall specimens were selected as the average values of Type II wearing coarse gradation limits of the Turkish State Highway Specification (Fig. 1).

**Table 1**  
Properties of original bitumen.

Property	Value	Specification
Specific gravity, 25 °C	1,02	ASTM D-70
Penetration, 25 °C (dmm)	51	ASTM D-5
Viscosity, 130 °C, (Pa.s)	0316	ASTM D-4402
Softening point, (°C)	47,5	ASTM D-36

**Table 2**  
Physical and chemical properties of the aggregate.

Property	Value
<i>Specific gravity (coarse aggregate)</i>	
Bulk	2.825
Apparent	2.860
<i>Specific gravity (fine aggregate)</i>	
Bulk	2.832
Apparent	2.866
<i>Specific gravity (filler)</i>	
Bulk	–
Apparent	2.833
<i>Water absorption (%)</i>	
Coarse aggregate	0.44
Fine aggregate	0.42
<i>Chemical content (%)</i>	
SiO <sub>2</sub>	1.6
Al <sub>2</sub> O <sub>3</sub>	0.85
Fe <sub>2</sub> O <sub>3</sub>	0.57
MnO	0.03
MgO	21.50
CaO	29.67
Na <sub>2</sub> O	0.10
K <sub>2</sub> O	0.25
CO <sub>2</sub>	45.45
Cl	0.006
SO <sub>4</sub>	0.002

### 2.3. Preparation of DEGPB

DEGPB used as additive material was prepared in our laboratory, however the DEGPB used as the agent is 99% pure and purchased as Merck production. A polymer based additive is used to relieve aging in this study. Several materials were also used during the production of the additive besides the main agent to ensure that the additive is easily mixed with the bituminous binder. In the end, the ratio of the main agent to the obtained additive is as presented in Table 3.

To produce the additive, an electric heating mantle with an adjustable temperature and magnetic stirrer with adjustable velocity, thermoregulator, and condenser was utilized. The system used in the production of the additive can be seen in Fig. 2.

The synthesis has been occurred by reacting of boric acid with diethylene glycol in the presence catalyst (H<sub>2</sub>SO<sub>4</sub>). This chemical process was fulfilled under controlled temperature conditions. 150 g diethylene glycol was put into the reactor and started to heat. Immediately after the starting of heating, 0.75 ml of H<sub>2</sub>SO<sub>4</sub> was added to the diethylene glycol and mixed with magnetic stirrer. While the temperature of the mixture reached to 70 °C, 90 g of boric acid was added gradually. Meanwhile the mixing-heating processes continued. Temperature was kept at 120 ± 2 °C and the mixing process was continued for 30 min. At the end of the period, the system was completely turned off and the additive was cooled to ambient temperature then it was hermetically stored in the jar to use in modification of the bitumen.

### 2.4. Preparation of modified bitumens

DEGPB modified bitumens (DEGPBMB), obtained by the insertion of additives at 1, 2, 3 and 5% by-mass, were aged with TFOT (ASTM D 1754), which represents short-term aging. In this experiment, an ELE EL 22–4100 oven and its equipment were used. In TFOT aging, the bitumen sample was first heated in the oven until it had become flowable, similarly aging containers were also heated in the same oven, and next bitumens with 3.2 mm film thickness were poured into aging containers and finally placed in the oven, which was maintained at 163 °C. Bitumen samples were

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