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## Performance enhancement of the oxidized bitumen binder using epoxy resin

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## ABSTRACT

The effect of an epoxy resin modifier was studied on the performance of an oxidized bitumen binder. Four types of analysis were conducted including physical, mechanical, viscoelastic and thermal properties in order to study the impact of the epoxy resin on the bitumen performance. Fourier transform infrared (FTIR) and differential scanning calorimetry (DSC) studies revealed the reaction of the epoxy functional groups with the oxidized bitumen at elevated temperature. The DSC results of the epoxy-modified bitumen showed two glass transition temperature ( $T_g$ ), which are related to the maltene part of the bitumen ( $T_{g1}$ ) and the physical adsorption and/or chemical interactions between epoxy resin and asphaltene of the bitumen ( $T_{g2}$ ). 5% (w/w) of epoxy resin addition to the bitumen leads to the highest increase in softening point temperature of the bitumen (about 11 °C). Evaluation of the epoxy-modified bitumen showed an increase in hardness compared to the neat bitumen using shore 'A' test. Different epoxy-modified bitumens demonstrated an enhancement of storage modulus and also decrease in  $\tan \delta$  using dynamic mechanical thermal analysis (DMTA). Improvement in tensile strength and decrease in toughness and also elongation at break were observed for the oxidized bitumen modified with the epoxy resin. Thermogravimetric analysis (TGA) proved that the epoxy resin improved thermal stability of the bitumen.

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## 1. Introduction

Bitumen is a low molecular weight thermoplastic compound with complicated chemical composition obtained from refinery bottoms of petroleum products [1]. Bituminous materials contain maltenes and asphaltenes. The maltenes can also be divided further into resins, aromatics and saturates [2]. The asphaltenes are polar in nature and are responsible for the properties such as hardness, viscosity and softening point. The oxidized bitumen is achieved via chemical modification of the refined bitumen with air blowing at high temperature, 200 (°C). Oxygen can react with the bitumen to enhance asphaltene molecules causing a reduction in maltene percentage. Therefore, the glass transition temperature of the oxidized bitumen decreases [2,3].

In the past decades, bituminous organic coatings were extensively utilized for application in the pipelines industry to decrease the destructive effects of weather and chemicals [4–6]. Because of

its low cost, inherent cohesive nature, rheological behavior, water-proofing and anticorrosive properties, the bitumen coatings are still in use [7]. Neat bitumen shows weak physico-mechanical, chemical and thermal properties. These soft materials have also low cohesive strength at ambient temperature. They become brittle causing the crack formation on the coated surfaces at low temperatures and susceptible to damage during transport and handling [8,9]. Therefore, physical and chemical properties of the bitumen should be improved using additives such as polymers. The bitumen is permanently compatible with only a few polymers supplemented with small concentrations [10]. Giavarini [11] reported that the optimum properties of the polymer-modified bitumen could be achieved by 5–6% of polymer addition. Nadkarni et al., [12] examined bitumen modified with 6% (w/w) styrene butadiene styrene copolymer, in which the rheological properties of the bitumen were reinforced at low temperatures, whereas cohesive strength was not improved at high temperatures. In another study, it was declared that 5% (w/w) LDPE improved the physical and mechanical properties of 70/100 bitumen [13].

Modification of the bitumens with epoxy resin has been exploited as an alternative for toxic coal tar-epoxy binders. The choice of the epoxy resin as a polymeric modifier for the bitumens

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**Table 1**  
Physico-chemical properties of the bitumen and epoxy resin.

Property	Data
Oxidized bitumen	
Appearance	Black
Penetration, 25 °C, 100 gr, 0.1 mm	5–17
Softening point (ring and ball), °C	110–130
Density, 25 °C, gr/cm <sup>3</sup>	1.2–1.4
Flash point, minimum °C	260
Asphaltene, (w/w)%	40–45
Maltene, (w/w)%	55–60
Epoxy resin	
Appearance	Light Yellow
Epoxy Equivalent Weight, gr/eq	434–555
Density, gr/cm <sup>3</sup>	1.08
Solid content, %	74–76

was found to improve their strength, hardness, durability, chemical resistances, flexibility, adhesion to metals and high protective ability against corrosive species [14,15]. The epoxy resin provides both physical and chemical modifications of the bitumens. In the study of Cubuk et al. [16], the effect of the epoxy resin on the properties of 50/70 bitumen was investigated. Viscosity, penetration, softening point and glass transition temperature were examined. The epoxy-bitumen interaction and crosslinking network are formed. It was reported that using 2–3% (w/w) of the epoxy addition causes an increase in viscosity at ambient temperature, a decrease in penetration from 62 to 54, an enhancement in softening point from 49.2 to 52.2 (°C) and also increment of  $T_g$  from –22.50 to –13.85 (°C). Type and amount of the adsorbed resin on the asphaltene could be important, which is able to stabilize the asphaltenes resulted from strong physical interactions between them [17,18].

In this study, the effect of epoxy resin addition on the performance of the oxidized bitumen was evaluated using various methods. FTIR and DSC examinations were carried out to study the reactivity of epoxy resin and oxidized bitumen. The effect of the epoxy resin on  $T_g$  of the oxidized bitumen was investigated using DSC method. The epoxy-modified bitumens were studied in terms of physical softening point and mechanical shore 'A' hardness and tensile measurements. Also, DMTA and TGA were used in order to investigate viscoelastic and thermal properties.

## 2. Experimental

### 2.1. Materials

Two main components of the blends including oxidized bitumen according to EN10300 standard – Grade B (Tanguiran Co.) and epoxy resin based on bisphenol-A in a xylene solution, Epiran 01, (Khuzestan petrochemical Co.) were used. Some physico-chemical characteristics of these materials have been summarized in Table 1.

### 2.2. Preparation of epoxy-modified bitumen blends

The blends of the bitumen and epoxy resin were prepared as follow: (1) in the molten state, below 200 (°C) and (2) in the solution state at ambient temperature. In the molten state, the bitumen was pre-heated below 200 °C for 15 min to facilitate the subsequent mixing with the resin. Epoxy-bitumen mixings were carried out using an IKA RW-20 mixer at 1000 rpm for 30 min with different epoxy concentrations (2, 5, 10 and 15% (w/w)). In the solution state, the bitumen was dissolved in xylene (Mojallali Chemical Co.) and then mixed with epoxy resin for 15 min at 1000 rpm.

### 2.3. Study of epoxy and bitumen interactions

The possibility of chemical interaction between the epoxy resin and bitumen after mixing was evaluated using FTIR method at both ambient and elevated temperatures. The transmission mode of FTIR spectra were recorded through a Bruker IFS 88 spectrometer in the wavenumber range of 400–4000 (cm<sup>-1</sup>). The resolution was 4 (cm<sup>-1</sup>).

DSC measurements were carried out to study the reaction of the epoxy and bitumen in non-isothermal conditions up to 320 °C with a heating rate of 5 °C/min. Before DSC measurements, 5% (w/w) of the epoxy resin was blended with the solvent-based bitumen at ambient temperature, then the solvent was evaporated using a vacuum oven at 20 °C for 1 h. These tests were performed by means of 5–11 mg of the bituminous samples in sealed aluminium pans. Mettler Toledo DSC calorimeter was used and samples were purged with a nitrogen atmosphere by 80 ml/min flow rate.

### 2.4. Characterization of the epoxy-modified bitumens

To study the phase transitions, non-isothermal DSC measurements were carried out on the epoxy-modified bitumens during heating and cooling processes with a rate of 10 °C/min.

The softening point of each sample, which is a conventional physical property, was measured based on the ring & ball method according to ASTM D36-76 standard in duplicate.

The shore 'A' hardness of the epoxy-modified bitumens was measured using an indentation hardness tester (a PRÜFEN UND MESSEN device) in accordance with ASTM D2240. Measurements were performed on the cylindrical bituminous samples with heights of 3 cm and diameters of 4 cm. Testing duration for data recording and the applied durometer load were 5 s and 12.5 ± 0.5 N, respectively.

A Tritec 2000 dynamic mechanical thermal analysis was used to study the viscoelastic behavior of the epoxy-modified bitumens. DMTA measurements were performed in the compression mode on 10 × 10 × 3 mm<sup>3</sup> samples at a frequency of 1 Hz, heating rate of 2 °C/min and displacement amplitude of 0.02 mm. Furthermore, the temperature range of DMTA examinations was –60 to 60 °C, in which the bituminous samples become very soft.

Tensile tests were executed by a Galdabini 1890 tensile device (model Sun 2500) with a 10 mm/min strain rate using dumbbell shaped specimens at room temperature. Stress–strain curves were obtained and then tensile parameters were assessed. Three replications were made.

TGA coupled with derivative thermogravimetric (DTG) assessments were performed using a Mettler Toledo Thermogravimetric Analyzer (Model TGA/DSC 1, USA) with a heating rate of 10 °C/min in the 25–750 °C temperature range, under a nitrogen flow of 30 ml/min.

## 3. Results and discussion

### 3.1. Reactivity study of the bitumen and epoxy resin

#### 3.1.1. FTIR

Fig. 1 presents the FTIR spectra of the bitumen, the epoxy resin and blends of these materials at ambient and elevated temperatures. It can be seen that the main peaks of epoxide vibration bond are observed for the epoxy spectrum at 914 ( $\nu_{C-O}$ ) and 3050 cm<sup>-1</sup> ( $\nu_{C-H}$ ). In the case of the room temperature sample, these peaks were observable with a low intensity showing the presence of low epoxy percentage in the sample. However, they have been disappeared at the elevated temperature blend. IR absorption bonds of the groups such as C–O–C vibrations at 831 (cm<sup>-1</sup>), C–O alkoxy

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