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# Improving the performance of Crumb Rubber bitumen by means of Poly Phosphoric Acid (PPA) and Vestenamer additives

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#### ABSTRACT

Paralleled to the numerous researches on Crumb Rubber (CR) modified bitumen, some limited work-studies have also been implemented on the effects of specific chemical modifiers, such as Poly Phosphoric Acid (PPA) and Vestenamer, on the performance of bitumen. The main difference between present research and previous ones is simultaneous evaluation of these additives on bitumen performance grade. In this research, five samples of modified bitumen were prepared using PG58-22, PPA, Vestenamer, and various CR percentages. The samples were classified according to the superpave performance grading system. The results indicate that these modifiers have significantly improved the performance of bitumen.

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#### 1. Introduction

In recent years, many researches have been implemented on the effects of natural and chemical additives on the performance of unmodified bitumen.

CR has been used as an additive in asphalt mixes since the 1930s [1]. CR modification is known to improve performance aspects of asphalt mixture such as thermal susceptibility, elastic behavior, fatigue cracking resistance and aging stability. Aflaki and Tabatabaee researches on the effects of CR on the performance of PG58-22 shows that the modification of this bitumen with 14–16% CR, can improve the PG of bitumen at high, low and intermediate temperatures up to 15.6 °C, 3.7 °C and 5.1 °C respectively [2].

Moreover, many research works have been done about the effect of PPA on the performance of unmodified bitumen. Since the early 1990s, PPA has been used in combination with various polymer modifiers to enhance the quality of paving asphalt [3]. Many researchers have studied the effect of PPA modification on bitumen. Previous studies show that PPA modification mostly improves the high-temperature behavior of asphalt binders [4–6]. Recent researches show that addition of 1.6% PPA to PG58-22 can improve the high temperatures (HT) about 23.5 °C. PPA modification

demonstrated a twofold effect on low temperatures (LT). On one hand, it made the asphalt bitumen softer at low temperatures and therefore lowered the S-limiting temperature. On the other hand, it raised the m-limiting temperature and deteriorated low temperature by 3.2–8.3 °C [2].

In this research study, knowing about the effects of CR, PPA and Vestenamer on the behavior of unmodified bitumen, it was tried to improve the performance of PG58-22 bitumen, not only at high temperatures but also at low temperatures.

To evaluate the effect of these additives, firstly, the appropriate amount of PPA was specified by analyzing the effect of PPA contents on PG and bitumen physical properties. Secondly, different percentages of CR (5%, 8%, 10%, 12%, and 15%) were added to a combination of base bitumen, PPA and Vestenamer and the effect of CR content on bitumen performance was evaluated by testing and characterizing the produced bitumen according to the superpave PG system.

It is necessary to note that production of modified bitumen were implemented based on wet process and the amount of Vestenamer was selected 4.5 weight percent of CR, based on previous researches [7,8].

#### 2. Necessity of the research

In the first research on Iranian unmodified bitumen, eleven unmodified bitumen samples, including 40–50, 60–70, and 85–100 penetration grades, were obtained from seven mineral oil

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refineries in Iran. The PG of these bitumen determined according to superpave testing protocol (ASTM D6373) [9] as summarized in Table 1.

After the PGs of unmodified bitumen were specified, the results were compared against the list of the required PGs derived from a PG climate study conducted for Iran as summarized in Table 2. This table shows the 13 PG climatic categories in which average consecutive 7-day maximum pavement temperatures range from 52 °C to 76 °C, and low service temperatures range from -10 °C to -34 °C.

The results show that the available unmodified bitumen does not possess the variety required to cover climatic needs in terms of service temperature ranges. The 11 unmodified samples cover only four PGs (PG70-16, PG64-22, PG64-16, and PG58-22). As Iranian bitumen does not have an appropriate performance in various climatic conditions, modification of these samples is necessary.

#### 3. Experimental

#### 3.1. Materials

#### 3.1.1. Base bitumen (PG58-22)

To evaluate the effects of CR, PPA and Vestenamer on the performance of unmodified bitumen at different temperatures, PG58-22 was nominated for the following reasons:

- According to the PG classification of Iranian bitumen, presented in Table 1, the PG of most Iranian bitumen, which is used in road industries are PG58-22 and PG64-22. Since these PGs are the common products of most Iranian mineral oil refineries, we decided to opt the base bitumen between PG64-22 and PG58-22.
- In accordance with ASTM D6373, average 7-day maximum pavement design temperature is equal to 58 °C for PG58-22 and 64 °C for PG64-22. PG58-22 obviates the ASTM D6373 requirements at 58 °C and lower temperatures, while the latter one obviates them up to 64 °C. For this reason, the modification of maximum pavement design temperature is more important for PG58-22. Consequently, this research has focused on PG58-22. The physical properties of base bitumen (PG58-22) have been presented in Table 3.

**Table 1**The PGs of unmodified bitumen, which are produced in Iranian mineral oil refineries and used in road industries [2].

Sample code	HT (°C)	LT (°C)
A	70	-16
В	58	-22
C	64	-16
D	64	-22
E	58	-22
F	58	-22
G	58	-22
Н	58	-22
I	58	-22
J	58	-22
K	64	-22

**Table 2**Required PGs for different regions of Iran based on PG climate study [2].

•	•	• • •	
Category	HT (°C)	LT (°C)	
1	76	-10	
2	70	-28	
3	70	-16	
4	70	-10	
5	64	-28	
6	64	-22	
7	64	-16	
8	64	-10	
9	58	-34	
10	58	-28	
11	58	-22	
12	58	-16	
13	52	-22	

#### 3.1.2. Crumb Rubber (CR)

The type of CR and its particles' size are amongst the major factors, which affect on the behavior of CR modified bitumen [10–12]. In this research, the CR with specific gradation, which has been illustrated in Fig. 1, was used for modification of base bitumen. The physical properties of CR have been represented in Table 4.

#### 3.1.3. Poly Phosphoric Acid (PPA)

Poly Phosphoric Acid, or PPA, is a liquid mineral polymer and just one of many additives, which is used to modify and enhance paving grade bitumen. PPA can be an effective and economical tool for chemical modification, that is used alone or in conjunction with polymers. The appropriate utilization of PPA, in the right amount, can improve the physical properties of bitumen. It can improve the high temperature, and with some bitumen may improve the low temperature [13]. PPA reacts with many of the functional groups in bitumen. It breaks the asphalten agglomerates and creates the possibility for better distribution of asphalten in the malten phase as it has been illustrated in Fig. 2. As PPA breaks asphaltenes and turns them to Individual particles, it is more effective in contributing to elastic behavior [14]. The characteristics of PPA used in this research have been represented in Table 5.

#### 3.1.4. Vestenamer

CR alone as a modifier functions as a non-reactive additive while Vestenamer reacts chemically with CR and bitumen to produce a uniform, low tack, rubber-like composite. This chemical bond is attributable to the double-bond structure of the Vestenamer, which permits cross-linking with the sulfur associated with the asphaltenes and maltenes in the bitumen to create a macro-polymer network. The main role of Vestenamer is to improve the performance of pavement at high temperature conditions, where the rutting is the most prevalent type of pavement distress. The Vestenamer that was used in this research was a trade production of Evonic Degussa Corporation.

#### 3.2. Bitumen modification procedure

As modified bitumen in this research is a combination of CR, PPA and Vestenamer, firstly, the appropriate amount of acid was determined. For this purpose, three types of modified bitumen were made by mixing PG58-22 bitumen and various percentage of PPA (1, 1.5 and 2 weight percent of bitumen). Based on the results of PG classification and conventional bitumen tests, the optimum amount of acid was determined as 1% (as it will be described in Section 4.2).

Five modified bitumen were produced by mixing PG58-22 bitumen with different amounts of CR (5, 8, 10, 12, 15 weight percent of bitumen), PPA (1 weight percent of bitumen) and Vestenamer (4.5 weight percent of CR).

PG58-22 was blended with 1% PPA at  $160\,^{\circ}\text{C}$  for 60 min using conventional mixer. After 60 min, the temperature of PPA modified bitumen gradually increased, from  $160\,^{\circ}\text{C}$  to  $175\,^{\circ}\text{C}$ , and at  $175\,^{\circ}\text{C}$ , CR and Vestenamer were added to the bitumen, in separated stages, and blended for 4 h at 5500 rpm using a high shear mixer.

In this research, the modified bitumen was named as PV-CRx. In this labeling format, letter P stood for Poly Phosphoric Acid, V for Vestenamer and CR for Crumb Rubber. In all modified samples, the amount of Poly Phosphoric Acid was one weight percent of bitumen and Vestenamer was 4.5 weight percent of CR. CR content was variable in these samples. Based on the amounts of CR in the samples, X was equal to 5, 8, 10, 12 and 15. For instance, PV-CR5 was modified bitumen contained 5% of CR. According to this labeling system, samples contained 5%, 8%, 12% and 15% CR, labeled as PV-CR5, PV-CR8, PV-CR10, PV-CR12 and PV-CR15

To compare the results of these samples with the results of modified bitumen merely had CR (neither PPA nor Vestenamer), a sample of modified bitumen was made with 10% CR and labeled CR10 (10% is proposed as the optimal amount of CR).

#### 3.3. Bitumen tests

Softening point (ASTM D36) [15] and penetration grade (ASTM D5) [16] tests were performed on PPA modified bitumen.

**Table 3** Physical properties of base bitumen.

Test	Specification	Test method
Specific gravity @ 25 °C	1.012	ASTM-D70
Penetration @ 25 °C (0.1 mm)	65	ASTM-D5
Softening point (°C)	50.1	ASTM-D36
Loss of heating (%)	0.05	ASTM-D6
Flash point (°C)	298	ASTM-D92
Ductility @ 25 °C (mm)	≥100	ASTM-D113

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