

## INVESTIGATION OF THE EFFECT OF CARBONATION CAKE ON RHEOLOGICAL PROPERTIES OF BITUMINOUS BINDERS

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### Abstract

Aggregates and bitumen used in road flexible pavements are non-renewable natural materials and in terms of sustainability, it is of great importance that the service life of road pavements is long. Waste materials are also rising with increasing products due to industrial development. The evaluation of waste materials both prevents the destruction of nature by reducing the use of very limited natural materials and reduces the environmental problems in case of storage of waste materials. In this study, carbonation cake, which is a waste of sugar factory, was added to bitumen. Penetration, softening point, rotational viscosity and dynamic shear rheometer tests were applied on pure and modified bitumen. According to the results, it was determined that carbonation cake had no positive or negative effect on medium and high temperature rheological properties of bitumen. From the experiments, promising results have been obtained for the disposal of waste carbonation cake in a different area.

**Keyword:** Carbonation Cake, Bitumen, Addition, Rheology.

### 1. Introduction

Almost all highways in Turkey was applied in the form of flexible pavements and there are 63415 km of asphalt pavements on motorways, state and provincial roads as of 2019 [1]. The basic materials used in

highway pavements are aggregate and bituminous binders. Aggregates are obtained by crushing and sieving suitable rocks, while bituminous binders are obtained by refining oil.

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Aggregates and bitumen used in hot mix asphalts are non-renewable materials and re-use rates are low at the end of the service life of the pavements. Therefore, it is necessary to produce solutions that increase the service life of the pavement during design, construction and application. Due to the development of the industry, the amount of waste material is increasing day by day. Ashes formed in thermal power plants, blast furnace and steel mill slag in iron and steel industry, kiln dusts which are by-products of cement industry, marble dust wastes formed in marble industry, scrap automobile tires, glass shards obtained from glass industry are classified as industrial solid waste. In the world, most of these industrial wastes have the possibility to be used in every layer from the base course to the wearing course on highways [2]. Some of the waste materials (such as slag, fly ash and marble dust) are used as aggregates, while others (such as rubber and shingles) can be used as bitumen additives [3-7]. In our country, approximately 11 million tons of sugar beets are processed every year during the sugar production campaign. During this production, approximately 1 million tons of carbonation cake with 50% dry matter is disposed as waste [8]. At the end of the sherbet treatment processes applied in sugar production from sugar beet, carbonation cakes are formed which contain a large amount of calcium carbonate and also contain colloidal substances of organic origin coming from beet. These cakes are then thrown by mixing with water. These residues, which have a high pollution load of organic matter, are removed by purification in some sugar factories. In many sugar factories, they are directly fed to surface waters. Various methods have been proposed for the recovery of lime, its use in cement production and the use of acidic soils for the evaluation of these wastes [9-11]. In this study, the usability of the carbonating cake, a waste material during sugar production, as a bitumen additive was

investigated. The carbonation cake was added to the bitumen in 3 different ratios by weight of bitumen. The penetration, softening point, rotational viscosity and dynamic shear rheometer experiments were performed on the modified bitumen and the effect of carbonation cake on the rheological properties of bituminous binders was evaluated.

## 2. Materials and Methods

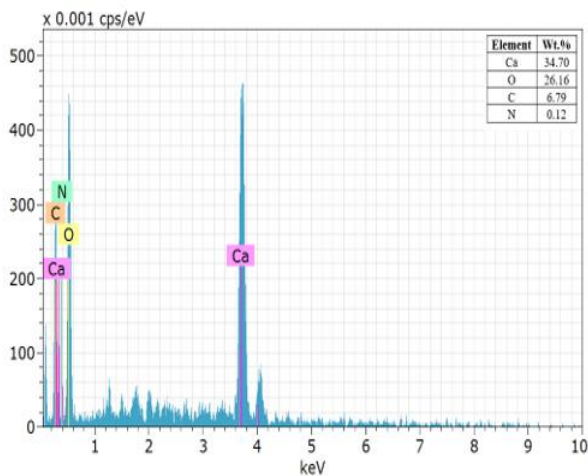
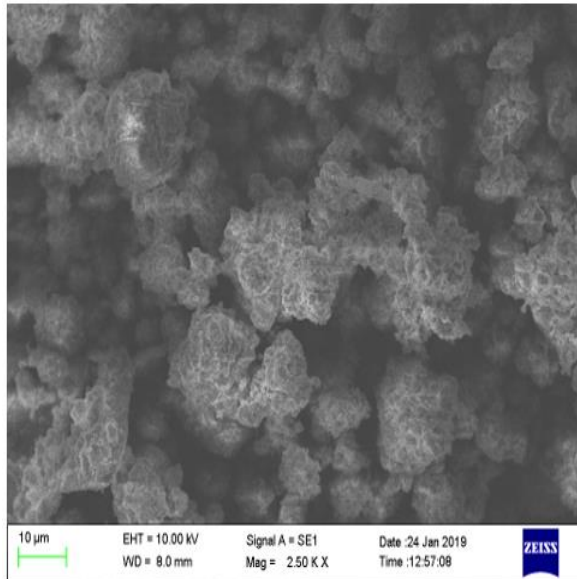
A B50/70 class bituminous binder procured from Batman TÜPRAŞ refinery was used as the main binder. Carbonation cake, which was used in the experiments, was obtained from Elazığ Sugar Factory in 2016 campaign period. The carbonation cake was supplied from the outlet of the rotary filter. The dried carbonation cake plant grinder was also milled and sieved. The -200 mesh fraction was dried for 2 hours in an oven at 80°C and kept in glass jars with lids during the experiments. The chemical composition of the carbonation cake used in the experiments is shown in Table 1 in terms of oxides. The XRD diffractogram, SEM-EDX image and particle size distribution of the carbonation cake were also characterized.

**Table 1.** Chemical composition of carbonation cake used in experiments

Component	Compound (%)
CaO	49.8
MgO	3.56
K <sub>2</sub> O	0.11
Na <sub>2</sub> O	0.15
FeO	0.12
Analysis undetectable	2.46
Weight Loss (50-850 °C)	43.8

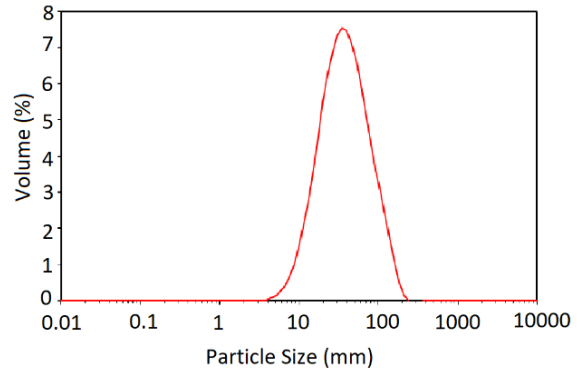
In Figure 1, it is seen from the SEM image that the carbonation cake has a small particle structure. It was also determined from the EDX analysis that the calcium compound in the structure was intensely present. It is seen that the carbonation cake has a particle size of less than 30 µm.

The carbonation cake can be considered to have formed as a very small particle by the reaction of dissolved calcium hydroxide with carbon dioxide in the carbonation process (Figure 2).



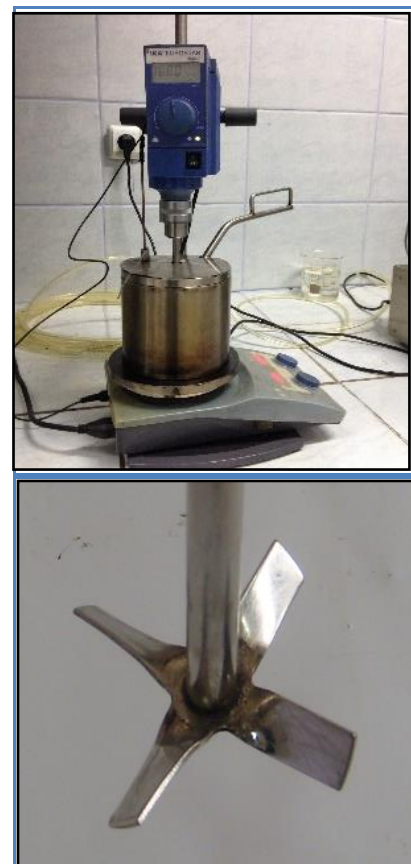
**Figure 1.** SEM image and EDX evaluation of carbonatation cake.

Modified bitumen were obtained by the addition of carbonation cake in the rates of 5%, 10%, and 15% per bitumen weight to the bitumen. Mixing temperature was selected as 180°C during the preparation of modified bitumen. Neat bitumen and the bitumen modified with the selected ratios of additive were stirred at 1000 rpm rate of rotation for 1 hour (Figure 3).



**Figure 2.** Particle size distribution of carbonation cake

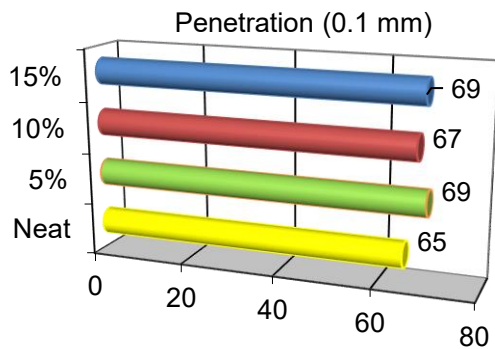
Penetration (EN 1426), softening point (EN 1427), rotational viscometer (ASTM D 4402), and dynamic shear rheometer (AASHTO TP5) tests were conducted on neat and modified bitumens.



**Figure 3.** Modified bitumen mixer and mixing cap

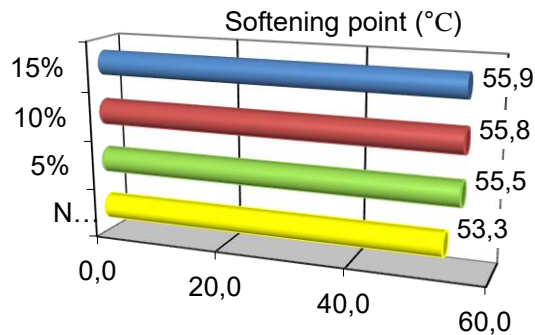
### 3. Results and Discussion

Results of the penetration tests conducted on neat and modified binders are displayed in Figure 4. As shown in Figure 4, when the 5%, 10% and 15% carbonation cake was added to the pure bitumen, there was no significant change in penetration values. Although the penetration values of 5% and 15% carbonated cake added binders appear slightly higher than other binders, this change does not make a significant difference when evaluated for consistency.



**Figure 4.** Average penetration values of pure and modified binders.

Softening point test results of neat and modified binders are shown in Figure 5. As shown in Figure 5, softening point values were slightly increased by adding carbonation cake to pure bitumen. Similar softening point values were obtained by adding 5%, 10% and 15% carbonation cake. With the addition of 5% carbonation cake to the pure bitumen, the softening point value increased by 4.2% compared to pure bitumen, 4.7% when 10% was added and 4.9% when 15% was added.

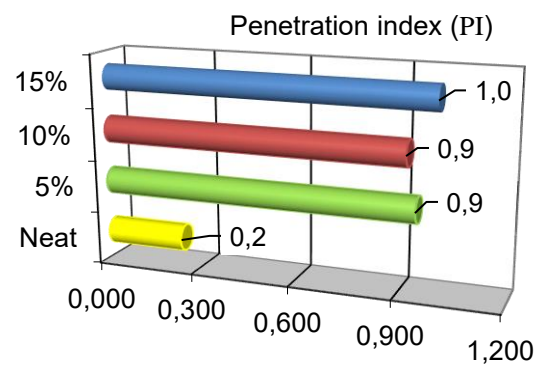


**Figure 5.** Softening point values of binders.

The penetration indexes showing the heat sensitivity of pure and modified bitumen were determined using the following formulas:

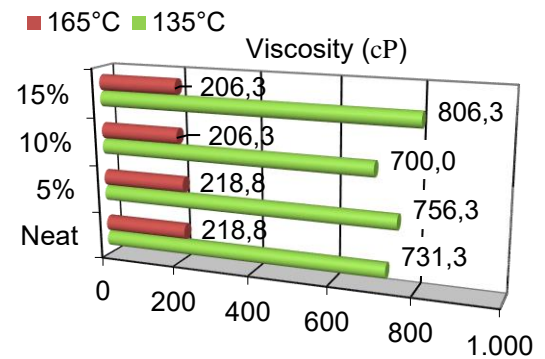
$$A = \frac{\log 800 - \log P_{25}}{T_{YN} - 25} \quad (1)$$

$$PI = \frac{20 - 500A}{1 + 50A} \quad (2)$$



**Figure 6.** Penetration index values of binders

The penetration index values of the binders are shown in Figure 6. As can be seen in the figure, the penetration index values were generally increased with the use of additives compared to the pure binder. This increase in penetration indexes indicates that the use of carbonating cake reduces the heat sensitivity of the pure binder.



**Figure 7.** Viscosity values of binders at 135 and 165°C



The results obtained from the rotational viscometer tests applied to the binders at 135°C and 165°C are shown in Figure 7. When the viscosity values at 135°C were examined, it was found that the lowest value was 10% carbonation cake added binder and the highest value was 15% carbonation cake added binder. At 165°C, it was determined that there was a very small difference (5.7%) between the viscosity values of all binders. Although the biggest difference between viscosity values is between binders containing 10% and 15% carbonation cake (14.3%) at 135°C, when the most commonly used bitumen additives styrene-butadiene-styrene (SBS) [12] and waste rubber (CR) [13] additives are taken into consideration, this change can be said to be negligible.

From the obtained results, it was determined that not all binders exceed the maximum 3000 cP which is the limit value in terms of pumpability.

In order to determine the effect of the use of carbonation cake as bitumen additive on the mixing and compaction temperatures of hot mix asphalt production, temperature values at recommended viscosities (280 ± 30 cP for compaction and 170 ± 20 cP for mixing) were determined [14]. The calculated values are given in Table 2. It was determined from the obtained results that modified bitumens containing three different ratios of carbonation cake need similar temperature and similar energy during mixing with aggregate.

**Table 2.** Mixing and compaction temperature ranges of pure and modified bitumens

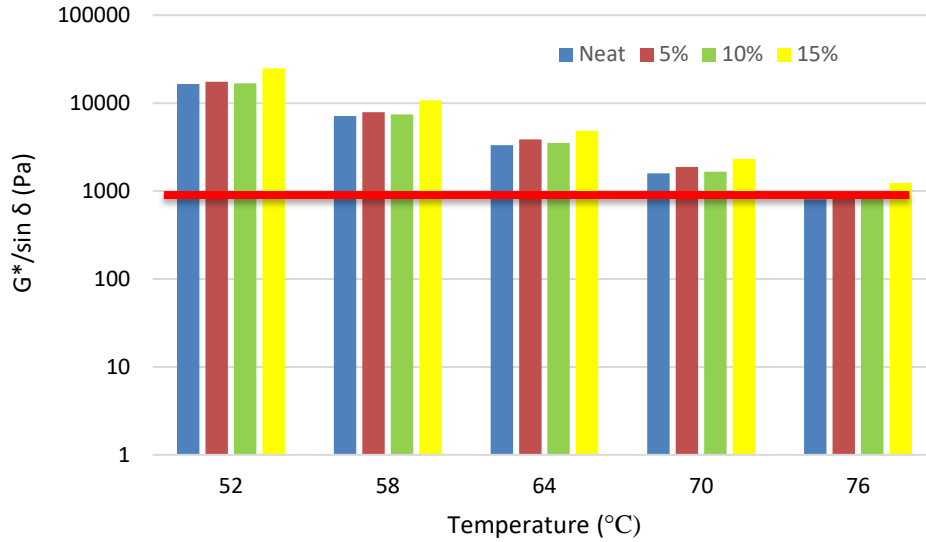
		Equation	Mixing temperature range (°C)	Compaction temperature range (°C)
B 50/70		$y = 166954e^{-0,04x}$	169,5-175,4	157,2-162,6
Carbonation Cake	5%	$y = 200860e^{-0,041x}$	169,8-175,6	157,9-163,2
	10%	$y = 171106e^{-0,041x}$	165,9-171,7	154,0-159,2
	15%	$y = 372229e^{-0,045x}$	168,4-173,7	157,6-162,4

Dynamic shear rheometer (DSR) test is used to evaluate the viscoelastic behavior of bituminous binders at medium and high temperatures. The DSR test characterizes the viscous and elastic behavior of asphalt cement by determining the complex shear modulus ( $G^*$ ) and phase angle ( $\delta$ ).  $G^*$  is an indicator of the total resistance of asphalt cement against deformations caused by repeated shear stresses. Both  $G^*$  and  $\delta$  values vary significantly with the heat and loading speed of asphalt cement [14-15]. In this study, neat and modified bitumen samples were filled into 25 mm diameter silicone containers and cooled to ambient temperature. The samples were then placed in the DSR test instrument until the temperature of the test was reached and the different temperature was tested. The effect of the additives on the rheological behavior of the pure binder was tried to be evaluated

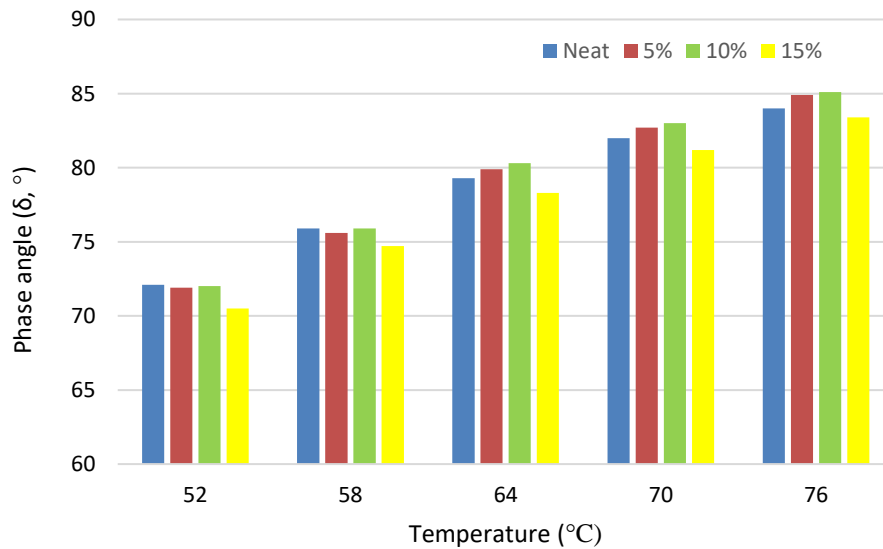
in a wider range by applying the DSR test. Rutting parameters ( $G^*/\sin\delta$ ) obtained from the DSR experiment are shown in Figure 8 and phase angles are shown in Figure 9. When Figure 8 is examined, the rutting parameters of all binders decreased with increasing temperature. It was determined that the binder containing 5% and 10% carbonation cake with pure binder had similar rutting parameter values. According to the Superpave specification, pure binders containing 5% and 10% carbonation cake meeting the 1000 Pa requirement provided 70°C and binder containing 15% carbonation cake at 76°C. According to the results, it was determined that the additive binders other than 15% carbonation cake did not significantly change the rutting parameter compared to pure bitumen.

Figure 9 shows that the phase angle values increase with increasing temperature, hence they exhibit more viscous behavior. It was found that the binder containing 5% and 10% carbonation cake and pure binder, had similar phase angle values and only the

binder containing 15% carbonation cake had lower phase angle values than the other binders. It can be said from the phase angle values that the elasticity of the binders increases with the use of 15% carbonation cake by bitumen weight.



**Figure 8.** Variation of rutting parameters of binders with temperature.



**Figure 9.** Variation of the binder phase angle values with temperature.

#### 4. Conclusions

In this study, the usability of carbonating cake which is a waste material in sugar factories as bitumen additive was

investigated. For this purpose, 3 different ratios (5%, 10% and 15%) of the bitumen weight carbonation cake were added to the pure bitumen.

Penetration, softening point, rotational viscosity at 2 different temperatures (135 and 165°C) and dynamic shear rheometer at 5 different temperatures (52, 58, 64, 70 and 76°C) were applied on pure and modified bitumens. It was found from the obtained results that the penetration values did not change significantly with the use of carbonation cake, the softening point values increased by about 5%, and the sensitivity of the binders to heat decreased with the use of carbonation cake. It was observed from the rotational viscosity tests that the use of a 15% carbonation cake at 135°C increased the viscosity values, that all binders at 165°C had similar values, and that there was no significant difference in mixing with aggregate and compaction temperatures. Dynamic shear rheometer experiments show that all binders exhibit more viscous behavior with increasing temperature, binders containing 5% and 10% carbonation cake have similar rutting parameters and phase angle values, whereas binder containing 15% carbonation cake has higher rutting strength and lower phase angle values.

When all the test results were evaluated, it was determined that the medium and high temperature rheological properties of the bituminous binder were not adversely affected when carbonation cake was added to the bitumen. This is hopeful that the waste carbonation cake can be used in a different area to prevent environmental damage. However, in order to determine the usability of a material as bitumen additive, the effect of hot mix asphalts on different parameters such as resistance to low temperature cracks, moisture damage and fatigue cracks should be examined. Therefore, it would be useful to prepare hot mix asphalt samples with binders containing carbonation cake and keep them in various performance tests.

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