



## Farklı İçeriklerde PVC Atığı ile Hazırlanan Sönmüş Kireç İçeren Bitümlü Karışımların Suya Bağlı Bozulmalara Karşı Direncinin Belirlenmesi

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### Öz

Dünya genelinde artan ticari hareketlilik yollara olan ihtiyacı artırmış ve bunun sonucunda yeni yol ağları ortaya çıkmıştır. Ülkemizde inşa edilen karayollarının üstü yapısı önemli ölçüde esnek kaplamalardan oluşmaktadır. Ağır taşıt sayısı, tekrarlanan yükler, çevre ve iklim koşulları gibi parametreler esnek kaplamalar üzerinde farklı deformasyonlara neden olmaktadır. Bu deformasyonlardan biri de su kaynaklı hasarlardır. Özellikle değişen iklim koşulları bazı bölgelerin yıl boyunca alması gereken yağışları çok kısa sürelerde yoğun olarak almasına neden olmaktadır. Bunun sonucunda esnek kaplamalar hasar görmekte ve hizmet ömürleri beklenenden daha kısa olmaktadır. Artan çevre bilinciyle birlikte, bazı atık malzemelerin bitümlü karışımlarda kullanılması son derece önemli bir konu haline gelmiştir. Bu çalışmada, farklı Polivinil Klorür (PVC) içerikleri ile modifiye edilmiş bitümlü malzemeler uygun gradasyonlu agregalar ile karıştırılmış ve su kaynaklı hasarlara karşı direnci değerlendirilmiştir. Ayrıca, sönmüş kireç ilavesinin numunelerin su kaynaklı hasara karşı direnci üzerindeki etkisini değerlendirmek için çeşitli performans testleri gerçekleştirilmiştir. Bu kapsamda, modifiye edilmiş bitüm ve agregaların özellikleri, geleneksel bitüm ve agrega testleri yapılarak belirlenmiştir. Çalışmanın son hedefi olarak, bitümlü malzemelere PVC ilavesinin su kaynaklı hasara karşı dirence etkisini belirlemek amacıyla karışımlara Modifiye Lottman (AASHTO T 283) Deneyi uygulanmıştır.

**Anahtar kelimeler:** PVC atığı, Bitümlü karışımlar, Bitüm modifikasyonu, Suya bağlı bozulmalar, Modifiye Lottman deneyi

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## Determination of Resistance to Water-Induced Damage of Bituminous Mixtures Containing Hydrated Lime Prepared with Different Contents of PVC Waste

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### Abstract

Increasing commercial mobility throughout the world has increased the need for roads and as a result, new road networks have emerged. The pavement type of the highways constructed in our country consists of flexible pavements to a significant extent. Parameters such as the number of heavy vehicles, repeated loads as well as environmental and climatic conditions cause different deformations on flexible pavements. One of these deformations is water-induced damage. In particular, changing climatic conditions cause some regions to experience intense precipitation in a very short period of time, which they should receive throughout the year. As a result, flexible pavements are damaged and their service life becomes shorter than expected. With increasing environmental awareness, the use of some waste materials in bituminous mixtures has become an extremely important issue. In this study, bituminous materials modified with different Polyvinyl Chloride (PVC) contents were mixed with properly graded aggregates and their resistance to water-induced damage has been evaluated. In addition, several performance tests were carried out to evaluate the effect of hydrated lime addition on the resistance of the samples to water-induced damage. In this context, the properties of the modified bitumen and aggregates were determined by performing conventional bitumen and aggregate tests. As the final objective of the study, Modified Lottman Test (AASHTO T 283) was performed on the mixtures to determine the effect of PVC addition to bituminous materials on resistance to water-induced damage.

**Keywords:** PVC waste, Bituminous mixtures, Bitumen modification, Water induced damage, Modified Lottman test

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## **1. Introduction**

The durability of bituminous mixtures is greatly affected by environmental factors such as temperature, air and water. The use of good quality bituminous materials and aggregates during the construction of flexible pavements at mild climatic conditions generally results in deformations such as fatigue cracking and rutting caused by stresses due to traffic loads [1]. When tough climatic conditions are taken into consideration, stresses due to traffic loads increase as a result of inadequate material quality and the service life of flexible pavements decreases. Because water or moisture in the environment is relatively neutral in the acid-basic balance, it interferes with the ability of the bituminous material to bond to the aggregate. This causes a type of deformation known as “stripping” in flexible pavements [2]. Because the mixture no longer acts as a coherent structural unit and strength is compromised. The cohesive resistance of the interstitial bitumen body is rendered useless by the loss of adhesion. Water can penetrate the interface by diffusion through the bituminous films and can enter directly into the particulate-coated aggregate [3]. There are five different mechanisms by which water can cause stripping, including detachment, displacement, spontaneous emulsification, pore pressure, and hydraulic scour [1].

Bituminous materials and crushed stone aggregates are the main constituents used in flexible pavements. Due to the limited availability of these materials in nature, the use of recycled materials has become a necessary issue to consider [4]. Therefore, many countries are actively researching and developing methods to minimize their impact on the environment by reusing waste materials [5]. This increasing trend towards the use of waste materials in flexible pavements aims to (I) reduce the use of limited natural resources, (II) minimize environmental degradation and problems caused by waste disposal, (III) increase production efficiency, and (IV) reduce material costs [4, 6]. The waste materials that can be incorporated into bituminous mixtures can be classified into three main categories: (I) industrial waste, which includes cellulose waste, wood lignins, slags, bottom ash, and fly ash; (II) municipal/domestic waste, which encompasses incinerator residue, scrap rubber, waste glass, and roofing shingles; and (III) mining waste, specifically coal mine refuse [7].

Polymer Modified Bitumens (PMB), one of the new asphalt materials developed in recent years to improve the mechanical properties of pavements, make the pavement more resistant to fatigue cracking, rutting and thermal effects [8]. However, despite these advantages, the average market price of PMB material is high, resulting in a 30-40% cost increase in asphalt mixtures [8, 9]. Therefore, the use of recovered/waste polymers as an alternative to reduce the cost of PMB material to be used in pavements has become an approach of interest [9]. In this context, in this study, waste Polyvinyl Chloride (PVC), a polymer from the thermoplastic elastomer class, will be used as a binder modifier. By adding thermoplastics to bitumen, bituminous materials are made harder and bituminous mixtures can withstand heavy loads without flexing too much. Some thermoplastics used in bitumen modification are polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS) and ethylene vinyl acetate (EVA). When mixed with bituminous materials, they increase the viscosity and stiffness of bitumen at service temperatures [10]. PVC accounts for 10.1% of European plastic production and is used in the manufacture of window frames, profiles, cable insulation and garden hoses [9]. Waste PVC material to be used as bitumen modifying agent can be pipes [11, 12], window frames or cables [13]. The usability of PVC waste in bituminous materials has been demonstrated in many studies. The use of PVC particles in the paving industry has been shown to protect the environment, reduce pavement production and maintenance costs, and improve the mechanical properties of asphalt mixtures [14]. The addition of PVC waste to bituminous materials results in an improvement in phase angle and complex modulus values and an increase in indirect tensile strength (ITS) values. However, the rutting values were significantly reduced by the use of waste PVC in the asphalt mix [15]. It was studied the effects of different PVC wastes including blinds, window wastes and cable wastes, PVC based on bitumen. The results showed that the addition of PVC blinds and window waste in the range of 1-3% improved the performance of the bitumen modified at high temperatures. Also, 5% of cable waste causes bitumen to perform better at low temperatures [14]. The ductility, penetration and solubility values of bituminous mixtures decrease with increasing addition of PVC waste to bituminous mixtures [16]. Salman et al. studied the physical properties of bitumen mixed with PVC waste at different percentages ranging from 2.5% to 15% by weight of the bituminous material. According to the results obtained, the penetration decreased significantly with the addition of PVC waste additive, the ductility also reduced and the softening point increased as the PVC

percentage increased [17]. Ezzat et al. evaluated the aging values of bituminous materials by FTIR test. The addition of PVC waste to bitumen has a positive effect on the aging resistance of bituminous materials, according to the results obtained [18].

Durability of bituminous mixtures can be significantly affected by environmental factors such as temperature, weather and water [19]. Especially in regions exposed to heavy rainfall and high groundwater levels, bituminous mixtures often suffer early water-related damage and their performance under load is reduced [20]. The presence of water on the asphalt surface, combined with other environmental factors such as freeze-thaw cycles, can have negative consequences [21]. Moisture-induced damage is a complex form of deterioration that results in a reduction in the stiffness and strength of bituminous mixtures, ultimately leading to costly maintenance of the entire pavement structure [22]. Moisture-induced damage can be managed or reduced through a variety of methods, such as selecting appropriate materials, designing appropriate mixtures, increasing asphalt film thickness, using additives, implementing an effective surface design, ensuring proper compaction, and developing drainage systems [23]. Among these, the use of anti-stripping additives to improve the adhesion between bitumen and aggregate has become attractive over the years to limit or control moisture-related damage on bituminous mixtures [26]. Although a wide variety of anti-stripping additives have been used recently, hydrated lime is reported to be the most commonly used [19, 22, 25].

In light of the literature review, one of the most economical methods of reducing moisture sensitivity in bituminous mixtures is the addition of hydrated lime. The relatively high silica content in limestone aggregate makes it difficult for the aggregate to bond with bitumen in highly moisturized environmental conditions. When added to the bituminous mixture, hydrated lime reacts with the aggregate, strengthening the bitumen/aggregate interphase. When dry hydrated lime is added to a wet aggregate, the additive completely covers the aggregate and reduces the silica content of the aggregate. Thus, by strengthening the bond between the bituminous material and the aggregate, it reduces the moisture sensitivity of flexible pavements and reduces water-induced damages [19, 26]. Considering these advantages, hydrated lime was used in the experiments to evaluate the effect of different contents of PVC waste on the resistance to moisture-induced deterioration of bituminous mixtures conditioned and unconditioned with this anti-stripping agent.

When hydrated lime is added to the bituminous mixture, it reacts with the aggregate and increases the bond between the bitumen and aggregate phase. Lime reacts with highly polar molecules to inhibit the formation of soluble soaps that promote stripping. When these molecules react with lime, they form salts that are not affected by water. The ability of hydrated lime to make an asphalt mixture harder, more durable and resistant to rutting is a reflection of its superior performance as an active mineral filler [27].

There are five methods for introducing hydrated lime into bituminous mixtures. These are: (I) lime slurry to dry or wet aggregate, (II) dry lime to wet aggregate, (III) dry lime to dry aggregate (IV) dry lime to bitumen, and (V) quicklime slurry to dry or wet aggregate [28, 29].

The addition of hydrated lime to wet aggregate is generally a better method and provides good mixing, coating and processing, especially with weak materials. It is also best suited for application under laboratory conditions [28-29].

Furthermore, recent research has shown that hydrated lime has additional effects on bituminous mixtures that can act as active fillers, an antioxidant and an additive that interacts with fine clay grains in bituminous mixtures [30]. Several studies have highlighted the use of hydrated lime as a means of increasing resistance to moisture damage in bituminous mixtures, as well as providing other advantages such as increased stiffness and better resistance to rutting [31].

## 2. Materials and Methods

### 2.1. Aggregate

The aggregate materials used in the study were obtained from 2<sup>nd</sup> Region and 26<sup>th</sup> Branch of General Directorate of Highways. Aggregate properties were determined by standard aggregate tests, where specified in the General Directorate of Highways, Highways Technical Specification (KTŞ), such as specific gravity, Los Angeles abrasion, flatness index, and Sodium soundness test. The results are given in Table 1. After the tests on the utilization of aggregate properties in bituminous mixtures were performed, Sieve Analysis Test was applied according to the Type-1 gradation limits specified in the Highways Technical Specifications for wearing courses.

**Table 1.** Properties of limestone aggregate

Test	Specification	Results	Spec. Limit
Sieve No	ASTM C 136		
¾"		100	100
½"		92	88-100
⅜"		80	72-90
No 4		47.3	42-52
No 10		33	25-35
No 40		13	10-20
No 80		9	7-14
No 200		5.3	3-8
Specific Gravity (Coarse Agg.)	ASTM C 127		
Bulk		2.645	-
SSD		2.666	-
Apparent		2.713	-
Specific Gravity (Fine Agg.)	ASTM C 128		
Bulk		2.643	-
SSD		2.672	-
Apparent		2.722	-
Specific Gravity (Filler)		2.686	-
Los Angeles Abrasion (%)	TS EN 1097-2	27.4	Max. 27
Flatness Index (%)	BS 812	5	Max. 10
Freeze-thaw resistance (MgSO <sub>4</sub> ) (%)	TS EN 1367-2	4.154	Max. 6

### 2.2. Bitumen

50/70 penetration grade bitumen obtained from İzmir Refinery of Turkish Petroleum Refineries Inc. (TÜPRAŞ) was used as bituminous material. Penetration, softening point, viscosity and etc. tests were performed on base bitumen to determine the properties of the binder. The test results are given in Table 2.

**Table 2.** Properties of base bitumen

Test	Specification	Results	Spec. Limits
Penetration (25 °C;0,1 mm)	ASTM D5 TS EN 1426	55	50-70
Softening Point (°C)	ASTM D36 TS EN 1427	49	46-54
Viscosity at (135°C)-Pa.s	ASTM D4402	412.5	-
Thin Film Oven Test (TFOT) (163°C, 5 hr.) Loss on Mass (%)	TS EN 12607-1	0.04	0.5 (Max.)
Thin Film Oven Test (TFOT) (163°C, 5 hr.) Penetration after TFOT (%)	ASTM D5 TS EN 1426	75	50 (Min.)
Softening Point after TFOT (°C)	ASTM D36 TS EN 1427	6	9 (Max.)
Ductility (25°C), cm	ASTM D113	100	-
Specific Gravity	ASTM D92	1.03	-
Flash Point (°C)	EN 22592	260	230 (Min.)

## 2.2. Hydrated lime

The hydrated lime admixture used in the experimental studies, which contains a high percentage of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ), was obtained locally in Muğla, Türkiye. The properties of the hydrated lime admixture used are given in Table 3. The usage characteristics of hydrated lime are described in the previous section. Since the main focus of the study was on the proportions of PVC content, hydrated lime was used at 1.5% as recommended in previous studies [19].

**Table 3.** Properties of hydrated lime layer

Components after analysis			Components after calculation		
Name	Formula	%	Name	Formula	%
Silicon dioxide	$\text{SiO}_2$	0.40	Calcium carbonate	$\text{CaCO}_3$	3.95
Ferrous oxide+	$\text{Fe}_2\text{O}_3+$	0.12	Calcium hydroxide	$\text{Ca}(\text{OH})_2$	95.03
Aluminum oxide	$\text{Al}_2\text{O}_3$	0.10	Magnesium carbonate	$\text{MgCO}_3$	–
Calcium oxide	$\text{CaO}$	–	Magnesium hydroxide	$\text{Mg}(\text{OH})_2$	–
Magnesium oxide	$\text{MgO}$	–	Calcium sulfate	$\text{CaSO}_4$	–
Sulphur trioxide	$\text{SO}_3$	–	Sulphur	S	–
Loss on heating	$\text{CO}_2$	–	Sieve number		
Surface moisture	$\text{H}_2\text{O}$	0.40	Sieve size: 25 $\mu$		
Relative humidity	$\text{H}_2\text{O}$	–	% Passing: 99.74		
Insoluble materials		–			

## 2.4. Test applied on aggregates

The limestone aggregate used in the study was subjected to standard aggregate testing. Sieve analysis (ASTM C 136), specific gravity determination for coarse/fine/filler materials (ASTM C 127, ASTM C 128), Los Angeles abrasion (TS EN 1097-2), flatness index (TS EN 933-3) and soundness test (TS EN 1367-2) were performed on aggregate samples.

### 2.4.1. Sieve analysis test (ASTM C 136)

This test method involves determining the grain size distribution of coarse and fine aggregates by sieving. To determine the particle size distribution, the aggregate sample of known dry weight is sieved through progressively smaller apertures. This method is especially used to determine the gradation of the materials to be used as aggregate. The results provide the necessary data for verifying the conformity of the particle size distribution to the specifications and the aggregates or aggregate mixtures produced. Figure 1 shows the set used in the sieve analysis.



**Figure 1.** Set used in sieve analysis

#### **2.4.2. Specific gravity determination for coarse/fine/filler materials (ASTM C 127, ASTM C 128)**

The specific gravity of an aggregate is the ratio of the weight per unit volume of that aggregate to the weight of water in the same volume and at 25°C. There are different specific gravity types of particles, depending on the volume definition.

Apparent Specific Gravity is the ratio of the weight in air of the unit volume containing the impermeable voids of the aggregate at a given temperature to the weight of distilled water at the same temperature and in the same volume.

Bulk Specific Gravity is the ratio of the weight in air of the unit volume of the aggregate containing the permeable and non-permeable voids of the aggregate at a given temperature to the weight in air of the same temperature and volume of the same volume of distilled water at the same temperature and volume.

#### **2.4.3. Los Angeles abrasion test (ASTM C131, AASHTO T96, TS EN 1097-2)**

The Los Angeles Abrasion Test determines the wear of aggregate particles due to impact and abrasion. With this test, the abrasion resistance of coarse aggregates with a grain size of less than 75 mm is determined by Los Angeles Abrasion Machine. In the experiment, metal abrasive spheres are placed in the machine, which is in the form of a two-ended closed cylinder, together with the aggregate, and the machine is operated at a certain speed and rotation. As a result, the percentage by weight of the material abraded by the metal spheres falling on it compared to the material taken at the beginning of the experiment is given as abrasion loss. The test apparatus for the Los Angeles abrasion test is given in Figure 2.



**Figure 2.** Device used for the Los Angeles Abrasion Test

#### **2.4.4. Flatness index test (TS EN 933-3)**

It is a method based on the identification of aggregate particles with a thickness less than 0.6 of their nominal size as flat. The flatness index of aggregate samples is expressed as a percentage of the ratio of the weight of flat particles separated using a template with specific openings to the total sample weight. The template for flatness index determination is shown in Figure 3.



**Figure 3.** Flatness Index Determination

#### **2.4.5. Soundness test (TS EN 1367-2)**

This test is a rapid test used to measure the resistance to freezing and thawing of aggregates that have been exposed to weathering for a long time.  $\text{Na}_2\text{SO}_4$  and  $\text{MgSO}_4$  solutions can be used in the test. The test is applied to the aggregate above 4.75 mm for 5 freeze-thaw periods and the percentage loss at the end of these processes is calculated. The effect on the aggregate is equivalent to about 500 freezing and thawing events in nature. The meshed aggregate bucket and the  $\text{Na}_2\text{SO}_4$  and  $\text{MgSO}_4$  solutions used in the experiment are shown in Figure 4.



**Figure 4.** The meshed aggregate bucket and the  $\text{Na}_2\text{SO}_4$  and  $\text{MgSO}_4$  solutions

#### **2.5. Test applied on bitumen**

As part of the study, conventional bitumen tests were performed on the bituminous materials used. Summary information on the tests is provided below as well as bituminous materials containing PVC waste as an additive.

##### **2.5.1. Preparation of bituminous materials containing PVC waste additives**

The PVC waste used in the study was obtained from the chips generated during window production. PVC wastes were added to bituminous materials in powder form (Figure 5).





**Figure 5.** PVC waste additive

The additive rates were 0%, 0.5%, 1% and 1.5% by weight of the bituminous material. PVC waste was slowly added to the bituminous material heated to 150°C in an air circulation oven and mixed at 1500 rpm for 60 minutes while keeping the temperature constant (Figure 6). The PVC-modified bitumen produced was used immediately after production.



**Figure 6.** Production of PVC Waste-Modified Bituminous Material

### **2.5.2. Penetration test (TS EN 1426)**

This method covers the determination of hardness and viscosity of bitumen and bituminous binders. This method is applied for samples with penetration values up to 330\*0.1 mm, while a different method is applied for samples with penetration values between 330\*0.1 mm and 500\*0.1 mm. It is the depth of penetration of a standard needle into a conditioned specimen. The test conditions are 25 °C, 100 grams application load and 5 seconds loading time for penetrations up to 330 x 0.1 mm. For penetrations exceeding 330 x 0.1 mm, the test temperature is reduced to 15 °C but the application load and time remain the same. The needle is slowly

lowered downwards to the zero position on the sample surface. The needle holder is released to allow the needle to penetrate the sample. The first reading is recorded. A clean needle is used for each reading. At least three valid readings are taken at points on the surface of the test sample with three separate needles. The distance from the remaining edges of the measured points and the distance of each point from each other should not be less than 10 mm. Figure 7 shows the setup of the penetration test.



**Figure 7.** Penetration test machine

### **2.5.3. Softening point test (TS EN 1427)**

This method covers the determination of the softening point of bitumen and bituminous binders between 28 °C and 150 °C. The method is also referred to as the Ring & Ball Method. Two horizontally positioned disk-shaped bituminous binder samples, each with a ball on top, cast in brass rings are heated in a controlled manner in a test bath. The softening point is given as the average of the temperatures read from the thermometer when each ball embedded in the disc-shaped bitumen samples falls  $(25.0 \pm 0.4)$  mm into the test bench.

To obtain the initial temperature, the bath is placed in ice water or a thermostatic device and the water is cooled to  $(5 \pm 1)$  °C. This temperature should be maintained for 15 minutes but should not exceed 20 minutes. Using a pair of pliers, one ball is inserted into each ball center guide. The bath liquid is stirred to distribute the temperature evenly and the temperature is increased by 5°C per minute. The thermometer reading is recorded as soon as the bituminous binder on both balls touches the baseplate when using a non-automatic tester, or cuts the light beam when using a semi-automatic or automatic tester.

For modified bitumen, the test is repeated if the measured temperature difference of the two samples exceeds 2 °C either if the ball breaks the thin layer without touching the bottom plate or if partial separation between the ball and bitumen is observed. The apparatus for the softening point experiment is given in Figure 8.



Figure 8. Ring and ball test apparatus

#### 2.5.4. Rolling thin film oven test (TS EN 12607-1)

This method is used to measure the combined effects of air and temperature on a thin film of bitumen or bituminous binder. The method is not applicable to some modified binders or binders with very high viscosity values. The method is also referred to as rolling thin film oven test (RTFOT). A moving film of bituminous binder is heated in an oven at a specified temperature. The effects of air and temperature are determined by changes in mass or changes in properties of the bituminous binder such as penetration, softening point or dynamic viscosity.

Make sure that the oven is in equilibrium. Preheat the oven to the experimental temperature. Weigh at least two empty glass containers separately and pour  $(35 \pm 0.5)$  grams of sample into each container. To determine the percentage change in mass, two containers (designated A and B) containing bituminous binder (empty weights  $M_0$  and  $M'_0$ ) are taken and cooled to room temperature in a desiccator for 1 hour.

The empty containers are filled into the cavities in the carrier system and the lid is closed. The oven is rotated at a speed of  $(15.0 \pm 0.2)$  r/min. The specimens are exposed to air flow at  $(4.0 \pm 0.2)$  l/min. The specimens are kept in the oven and air is blown for  $(75 \pm 1)$  minutes and until the temperature is  $1^\circ\text{C}$  below the test temperature. If the experimental temperature of  $(163 \pm 1)^\circ\text{C}$  is not reached in the first 15 minutes, the experiment is terminated. At the end of the experiment, the containers are removed from the oven. The two containers (A and B) are allowed to cool down to room temperature in a desiccator for 1 hour. The relative masses,  $M_2$  and  $M'_2$ , are found to a precision of 1 mg. For the other containers, the binders are poured into the same collection vessel before it cools and hardens and before the containers are heated. The container is shaken to ensure homogeneity and no air bubbles are formed. Within 72 hours,  $P_2$ , penetration at  $25^\circ\text{C}$  (TS EN 1426);  $T_2$ , Softening Point (TS EN 1427) and  $H_2$ , viscosity at  $60^\circ\text{C}$  (TS EN 12596) of the hardened binder are measured. The RTFOT device is shown in Figure 9.



Figure 9. RTFOT Device

#### 2.5.5 Ductility test (ASTM D113)

This method covers the procedure for measuring the tensile properties and determining the ductility of a bituminous material. Unless otherwise specified, the test is performed at a temperature of  $(25 \pm 0.5)^\circ\text{C}$  and a speed of  $5\text{ cm/min}$  ( $\pm 5.0\%$ ).

The mold is mounted on a brass plate. The surface of the plate and the surfaces a and a' of the mold are covered with a mold release agent. The plate on which the mold is placed must be flat and stable. The sample is heated until it is sufficiently liquid. After complete mixing, the sample is poured into the mold. While filling the mold, care should be taken to ensure that the mold parts do not move so that the sample does not deteriorate. The mold is filled with a little more material than the full level. The filled mold is kept at room temperature for  $35 \pm 5$  minutes. Then it is placed in a water bath at the experimental temperature and kept for  $35 \pm 5$  minutes. The sample is removed from the water bath and the excess material is shaved off with a spatula. The shaved specimen and mold are placed in the water bath for  $90 \pm 5$  minutes. The specimen and the plate are separated with a shearing motion, avoiding to bend of the specimen. The side parts (a and a') are removed without damaging the specimen. The rings at the end of the clamps are attached to the pins or hooks on the ductility device. The two clamps are pulled at the specified constant speed until the specimen breaks or reaches its length limits. When breakage occurs or the final length is reached, the length obtained is measured in centimeters. Figure 10 shows the ductility test device.



**Figure 10.** Ductility Test Device

### **2.5.6. Flash point determination test with Cleveland open cup method (TS EN ISO 2592)**

This method covers the determination of flash and fire points of bitumen and bituminous binders using the Cleveland Open Cup. The open cup method can be used for bituminous materials with flash points between  $79^\circ\text{C}$  -  $400^\circ\text{C}$ .

It is the value of the lowest temperature at which a flame source ignites the vapor of the test sample under the specified test conditions, corrected for 101.3 kPa pressure. Knowing the Flash Point of a material is very important to prevent any ignition and fire hazard that may occur when that material is heated during application. The test vessel is filled to a certain level with the test specimen. The temperature of the test specimen is increased at a constant rate, first rapidly and then slowly. At certain temperatures, a small flame is passed over the test vessel. Under the specified test conditions, the lowest temperature at which the vapor of the test sample ignites is taken as the flash temperature. The vapor is allowed to burn for at least 5 seconds to determine the fire temperature. The values obtained are calculated using an equation. Flash And Fire Point Test apparatus is shown in Figure 11.



**Figure 11.** Cleveland Open Cup Flash and Fire Point Test Apparatus

## **2.6. Performance determination test on bituminous mixtures**

### **2.6.1. Marshall stability and flow test (AASHTO T 245)**

In this study, the use of waste PVC in bitumen modification at 0.5%, 1% and 1.5% by weight and its effects on pavement performance were investigated. Mixture performance was investigated according to American Association of State Highway and Transportation Officials (AASHTO) T245 standard, also known as Marshall Stability and Flow Test. The aggregate mixture is obtained by the method given in AASHTO. The weight of aggregates contained in the samples was determined as 1150 gr. The aggregates were kept in an oven at 160°C for 24 hours before mixing and the bitumen was kept for 3 hours to minimize loss on heating. Control sample sets were prepared with B50-70 bitumen with 0.5% intermediate values between 3% and 7%, three for each ratio, and the optimum bitumen percentage was determined. After determining of the optimum bitumen content, a sufficient amount of bitumen was modified with 0.5%, 1% and 1.5% PVC and viscosity values were determined in accordance with AASHTO T316 standard to obtain the appropriate mixing temperature. The bituminous mixture samples were prepared according to the mixing procedure and compacted to a void percentage in the range of 3%-5% with Marshall Compactor. After resting for 24 hours, the specimens were tested in Marshall Testing Machine and the stability and flow values of the specimens were obtained.

The aggregate sample weighing 1150 g with predetermined gradation is kept for one day (24 hours) and the bitumen is kept for 2-3 hours in an oven set at 160°C. Materials such as molds and aggregate containers to be used during the experiment should be kept in an oven at 160°C to avoid any heat loss during the experiment. The aggregate mixture is removed from the oven. It is placed in the mixer container and a well is made in the center to add the bitumen. A certain amount of bitumen (with respect to optimum bitumen content-OBC) weighed on a scale is poured onto the aggregate. Aggregate and bitumen are mixed in the mixer for 1.5-2 minutes. Meanwhile, the mixer container is placed in the mixer heating device to prevent the temperature of the mixture from dropping. When the mixing of the sample is about to end, the molds are removed from the oven and lubricated to prevent sticking. Place a sheet of greaseproof or impermeable paper cut to the appropriate size on the bottom of the mold. Take the mixture from the mixer and place it into the mold by pre-compressing. Measure the temperature of the mixture at this stage and place greaseproof or impermeable paper on it again. The temperature of the mixture should not fall below 140°C. The sample mold is placed in the Marshall compactor and 75 strokes are applied on each side. The molds are allowed to cool for about 3 hours. The specimens are removed from the cooling mold with the help of a jack, named and marked. The samples are kept at room temperature for one day. Height measurements are taken from the specimens with the help of calipers. For this, a measurement should be taken from 3 different parts of the sample at approximately 120°. The weights of the samples in air and water are measured. The samples are kept in a water bath at 60°C for 30-40 minutes. The samples removed from the water bath are placed in the Marshall device. The device is started and the jaws of the device apply a load to the sample at a speed of 51 mm/min. As a result, the flow and strength values of the specimen are obtained. The results are recorded in terms of height, weight in air and water, Marshall stability and flow values

### **Determination of aggregate mixing ratios and gradation**

The aggregate to be used in the construction of bituminous hot mix layers should consist of a mixture of at least three different grain groups (coarse, medium, fine). In the crusher, the crushing of aggregate groups should be done in such a way as to ensure the gradation of the layer in which the aggregate will be used. In order to determine whether the appropriate mixture gradation will be obtained with the aggregates produced, it is necessary to calculate the aggregate mixture ratios from the day production starts. The proportions required to mix three or more aggregate groups to give the desired specification gradation can be determined by different methods.

### **Determination of optimum bitumen content (OBC)**

Optimum bitumen content (OBC) is used to determine the optimum amount of bitumen to be used in a bituminous mixture in order to mix bituminous material with graded aggregates. OBC is directly dependent

on the type and capillary structure of aggregate. When determining the OBC, aggregates with a certain gradation are mixed with bituminous material at a rate of 0.5% incrementation between 3.0% and 7.0%. Three samples of each percentage of bituminous material should be prepared.

Samples of all contents should be at the appropriate mixing and compaction temperature. The mixed samples are homogeneously placed in the Marshall mold and compressed by 75 strokes on both sides. The compacted bituminous mixtures are removed from the molds after reaching ambient temperature. With the results obtained according to the Marshall Table, "Bitumen ratio-Practical specific gravity", "Bitumen ratio-Marshall strength", "Bitumen ratio-air void ratio" and "Bitumen ratio - Voids filled with asphalt ratio" graphs are drawn. According to these graphs; the bitumen ratio corresponding to the maximum practical specific gravity value, the maximum Marshall strength value, 4% air void ratio value and 70% voids filled with asphalt ratio are determined. The optimum bitumen ratio for the mixture is found by averaging these four bitumen ratios. In order to determine whether the optimum bitumen content found is within the specification limits, the "Bitumen ratio - Flow" graph is drawn. If the optimum bitumen ratio meets the flow limits, it is accepted.

## **2.7. Modified Lottman test (AASHTO T 283)**

Flexible pavement practitioners have been aware since the 1920s that the adhesion between asphalt and aggregate weakens (stripping occurs) when water is present. In addition, the deterioration of cohesion in the asphalt binder with the presence of water has also been observed since that time. Therefore, attempts have been made to develop a reliable laboratory test method that can simulate the sensitivity of asphalt pavement to moisture in the field [32]. Lottman (1978) introduced to the industry a well-established laboratory test method for predicting moisture-induced damage of asphalt concrete [33]. This procedure developed by Lottman was later modified and standardized by AASHTO as Test Procedure T283 [32].

In this study, Modified Lottman (AASHTO T283) tests have been applied to determine the moisture susceptibility of bituminous mixtures. This test (AASHTO T283, TS EN 12697-12), which is applied to determine the resistance of mixtures to water-induced damage, includes the measurement of the indirect tensile strength (ITS) in the diameter plane due to accelerated water effects of compacted bituminous mixtures prepared in the laboratory. The bituminous mixtures prepared using PVC modified and base bitumen with the addition of hydrated lime were placed in an aluminum container and cooled at room temperature for 2 hours (Figure 12).



**Figure 12.** Loose Mixture During The Cooling Process for 2Hours

Then the mixtures were kept in an oven at 60°C for 16 hours for curing. After curing, the mixtures were kept in an oven at 135°C for 2 hours, then kept in an oven at 165°C for another 20 minutes and compressed using a Marshall Compactor at an air void of  $7 \pm 1.0\%$ . Three specimens are designated as dry (i.e. unconditioned) and tested without moisture exposure. In addition to the unconditioned specimens, three additional specimens are selected and subjected to conditioning (moisture conditioning). These samples are placed in a cylindrical metal pycnometer and then water absorption was applied to the sample by adding pure water at 25°C. The

plastic bag containing the sample was placed in a freezer cabinet at -18 °C for 16 hours and then cured in a 60 °C water bath for 24 hours. After this treatment, the samples were placed in a water bath set at 25 °C for 2 hours. The ITS values in the diameter plane of the dry specimens and the conditioned specimens as above were determined. The water-induced damage is determined by the ratio of ITS values of dry to conditioned specimens. This ratio should be minimum 80 % according to the General Directorate of Highways Technical Specifications. The flowchart of this experiment is given in Figure 13.

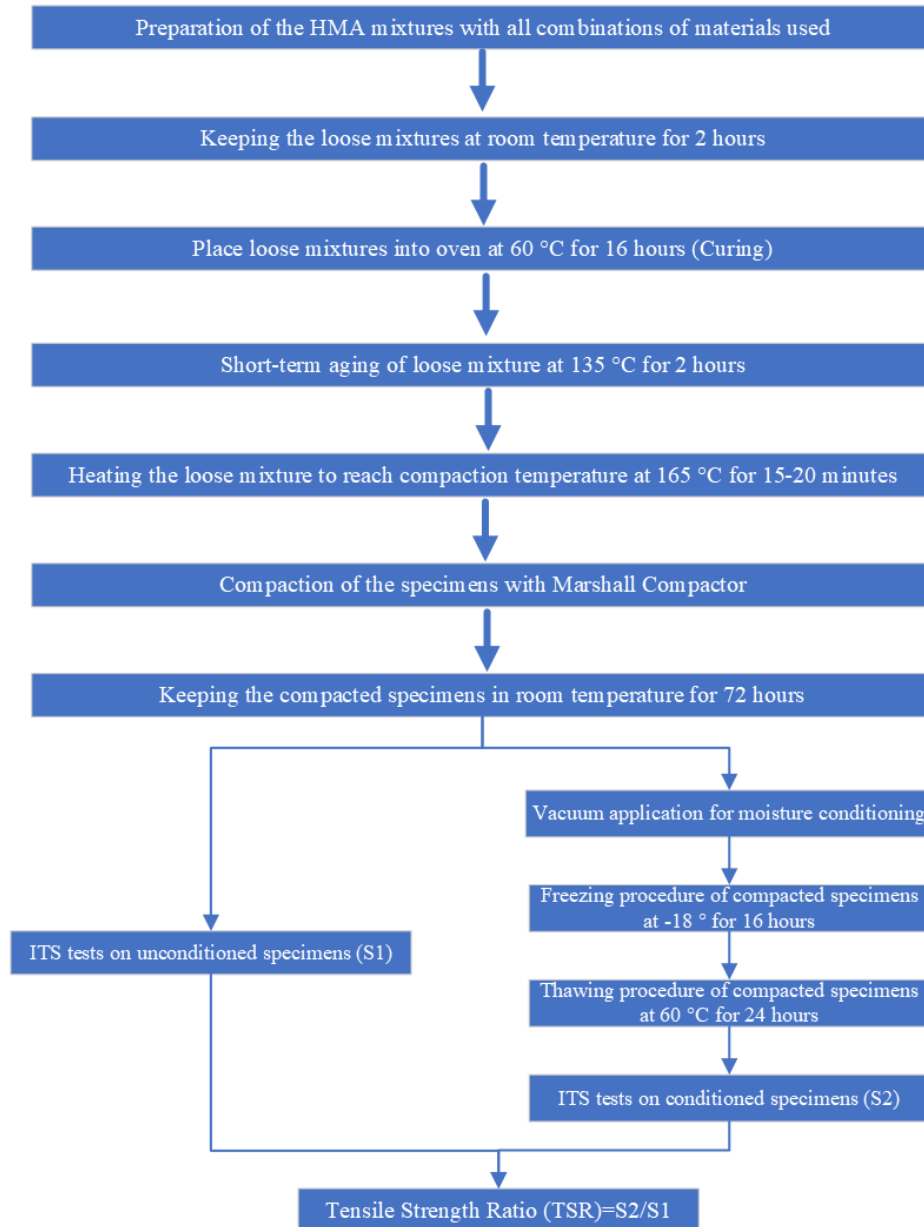


Figure 13. Modified Lottman (AASHTO T283, TS EN 12697-12) Test Flow Chart

### 3. Results and Discussion

With environmental factors, oxidation occurs in bituminous mixtures over time. This oxidation causes aging of bituminous materials, leading to a stiffer and more brittle structure of flexible pavements. Polar molecules in construction materials used in road building react with environmental factors to break down and cause deformation in flexible pavements. Adding hydrated lime to bituminous materials reduces the oxidation and aging rate of the flexible pavement. Since a chemical reaction occurs between the calcium hydroxide and the

highly polar molecules in the bituminous material, the pavement is not affected by environmental conditions. As a result, the bituminous material remains flexible and protected from crack occurrence for many years.

Stripping is caused by the presence of moisture in the bond between the bituminous material and the aggregate. In regions where the moisture in the air or ambient water is high, water entering between the bituminous material and the aggregate causes the binder to separate from the aggregate. Certain types of material, such as aggregates obtained from igneous rocks, are particularly susceptible to stripping. Severe environmental conditions such as heat, heavy rains, freeze/thaw cycles and traffic loads are significant contributors to peeling. Hydrated lime is one of the most effective and economical anti-peel admixtures available.

Hydrated lime reacts with highly polar molecules that can form water-soluble soaps that cause stripping in bituminous mixtures. As a result, they form hydrophobic insoluble salts. Furthermore, the homogeneous distribution of hydrated lime throughout the mix makes the mixture stiffer and more durable, reducing the likelihood of mechanical breakage of the bond between the bituminous material and the aggregate.

In many studies, it has been observed that the addition of 1-1.5% hydrated lime to bituminous mixtures increases the crushing strength by 77%. At low temperatures, the solidification effect of hydrated lime in bituminous mixtures is not significantly higher than that of other additives. The reason for this is that hydrated lime is more effective due to the strong interactions of the components that make up the bituminous mixtures [34].

Within the scope of the study, the reusability of waste Polyvinyl Chloride (PVC) materials as asphalt binder was investigated and it was aimed to improve the mechanical properties of the road pavement material as well as positive results in terms of environmental and economic aspects. In other words, it was aimed to reduce bitumen consumption and recycle waste PVC by mixing PVC waste into bitumen used as a binder in flexible pavements. In this context, the properties of modified bitumen with waste PVC powder were investigated and Marshall specimens were prepared with PVC-modified bitumen. Prepared specimens were subjected to Marshall Stability and Flow tests and stability flow and values were determined.

The performance of PVC addition to bituminous material in heavy rainfall areas was also evaluated. For this purpose, hydrated lime was added directly to aggregates as specified in the Turkish General Directorate of Highways, Highways Technical Specification Type-1 gradation for wearing course at the rate recommended in previous studies and its resistance to water-induced deterioration was determined. At this point, the use of hydrated lime was evaluated on a single-ingredient basis, as the focus was primarily on the performance of PVC in bituminous mixtures.

### **3.1. Test results of bituminous material prepared with PVC wastes with different contents**

Bitumen modification has been made by mixing 0.5%, 1% and 1.5% of waste PVC powder into bitumen. Firstly, the viscosity of pure and modified bitumen was measured with a rotational viscometer to determine the mixing and compaction temperatures. A set of specimens were prepared using asphalt cement at 4.0%, 4.5%, 5.0% and 5.5% then Marshall stability and flow tests were performed on these specimens to determine the optimum bitumen percentage. Using the optimum bitumen percentage of 4.22%, Marshall stability and flow tests were performed on the specimens prepared by using waste PVC powder at varying rates of 0.5%, 1% and 1.5% by weight of bitumen.

The Rotational Viscometer (RV) test is applied to determine the flow properties of bituminous materials at high temperatures. Viscosity values are obtained by the resistance to rotation of a shaft rotating at 20 rpm in the binder (Figure 14).



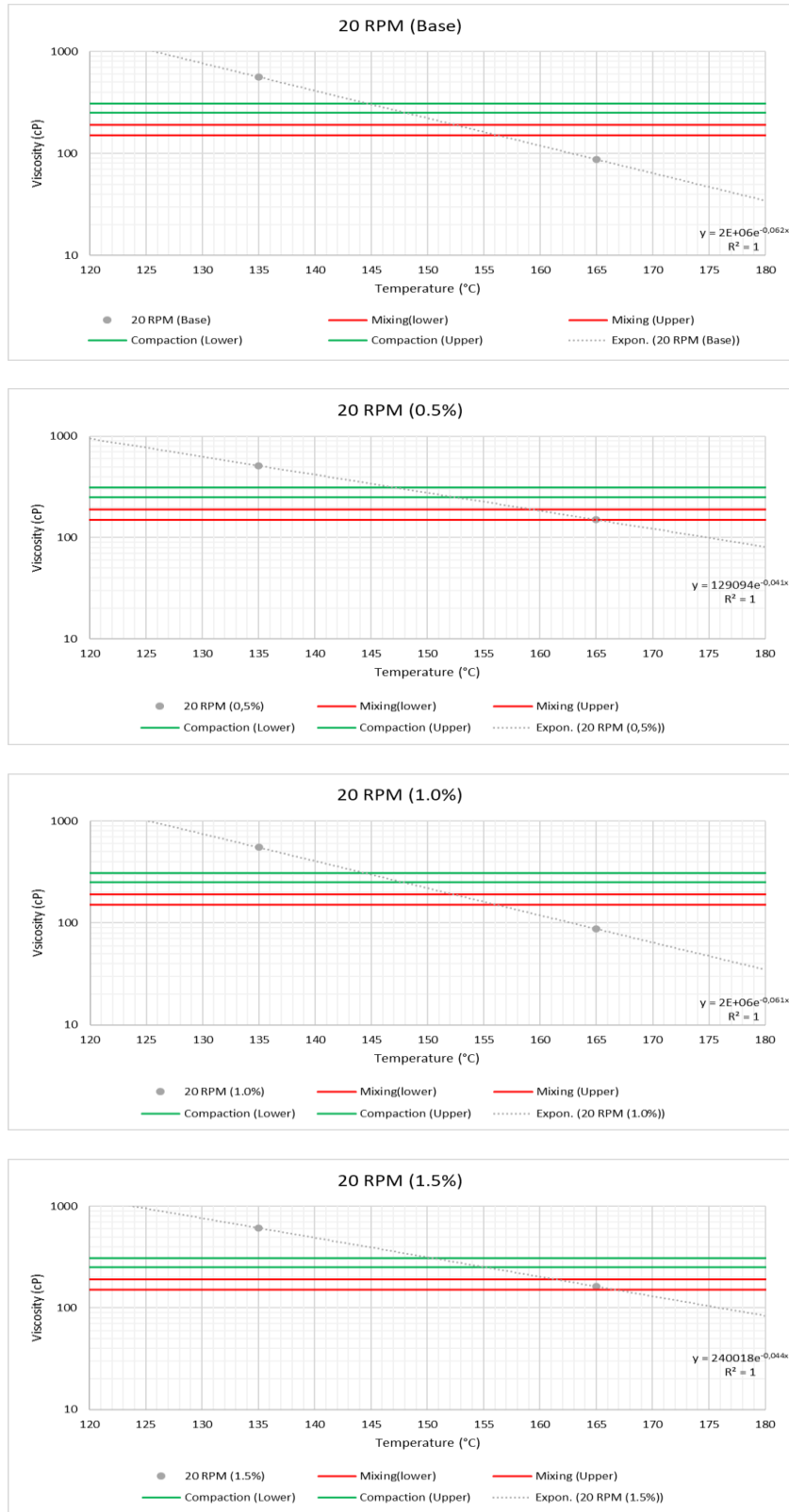


**Figure 14.** Brookfield Viscosity Test Device

For the experiment, a sample of about 30 grams of binder is taken and heated in an oven with a temperature of less than 150°C. About 11 grams is filled into the sample chamber and placed in a temperature-controlled container kept at a constant temperature. The sample is kept at a constant temperature for 15 minutes and then the experiment is performed. Viscosity readings are taken for temperatures of 135°C and 165°C. In this study, the viscosity of unmodified and modified bitumen was measured using a rotational viscometer. Measured viscosity results are given in Table 4 and the graphs for the determination of mixing and compression temperatures are shown in Figure 15.

**Table 4.** Viscosity values

PVC additive (%)	Viscosity value (cP)	
	135 °C	165 °C
0.0	562.5	87.5
0.5	572.5	150
1.0	550	87.5
1.5	612.5	162.5



**Figure 15.** Mixing and compaction temperatures

Mixing and compaction temperature ranges determined according to viscosity values are given in Table 5.

**Table 5.** Mixing and compaction temperatures

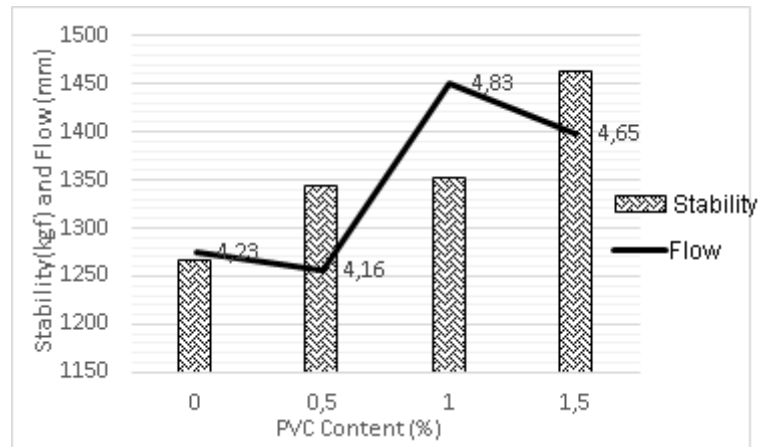
PVC additive (%)	Mixing temp. (°C)	Compaction temp. (°C)
0	152-156	144-148
0.5	159-165	147-153
1.0	152-156	144-148
1.5	161-167	150-156

### 3.2. Results of the Marshall stability and flow test of bituminous mixtures containing hydrated lime, prepared with different contents of PVC waste

Marshall stability and flow tests were performed on these samples and the optimum bitumen percentage was found to be 4.22%. According to the optimum bitumen percentage, 48.5 grams of bitumen was used for each Marshall sample. The bitumen to be used for the samples was then modified. For each percentage (for 0.5%, 1% and 1.5%), the relevant amount was added to the bitumen and mixed at 1500 rpm at 150°C for 1 hour. The specimens were placed in Marshall molds and compacted by applying 75 strokes on both surfaces of the specimens as specified in the Directorate of Highways Technical Specifications. Marshall samples were then prepared according to the relevant procedure. The prepared samples were allowed to cool and the physical properties of the samples were measured after 24 hours.

The specimens were cured in a water bath at 60°C for 30-40 minutes before conducting the Marshall strength and flow test. Following this curing period, the specimens were placed in Marshall test apparatus. At the end of the test, the stability, ITS and TSR values given in Figure 16 and Figure 17 respectively were obtained. Marshall mix design was performed on bituminous mixtures containing hydrated lime. The reason for this is to evaluate the effect of moisture sensitivity on the mixture in bituminous materials prepared with different contents of waste PVC additives.

According to the Turkish General Directorate of Highways, Highways Technical Specification, the minimum stability value (TS EN 12697-34) for wearing course Type-1 is 900kgf and the flow (TS EN 12697-34) is between 2-4 mm.



**Figure 16.** Marshall Stability and Flow test results

When Figure 16 is evaluated in terms of stability values, all of the bituminous mixture samples containing hydrated lime and prepared with different contents of PVC waste are well above the minimum value. It is seen that as the content of waste PVC additive to the bituminous mixture samples increases, the stability values of the specimens also increase.

During the production phase of the bituminous mixture, short-term aging occurs in asphalt plants due to bitumen oxidation and loss of the evaporated component. Long-term aging of bituminous materials is caused by the evaporation of the bituminous material in the asphalt mixture during mixing and paving [35].

The main factors in the aging of bituminous materials are parameters such as oxidation, evaporation, aging within the structure (thixotropy), which depend on environmental conditions such as time, temperature and oxygen [36]. It is thought that the bituminous material used in the study may have aged to some extent due to the distance of the source from which the material was brought and the fact that it was repeatedly heated and exposed to excessive temperatures during use. Indeed, when Figure 16 is examined, the flow values obtained from the bituminous mixtures are above the 2-4 mm values specified in the Technical Specifications of Highways. However, the evaluations made in the light of the obtained results were made by taking this situation into consideration and considered proportionally in order to eliminate the error situation.

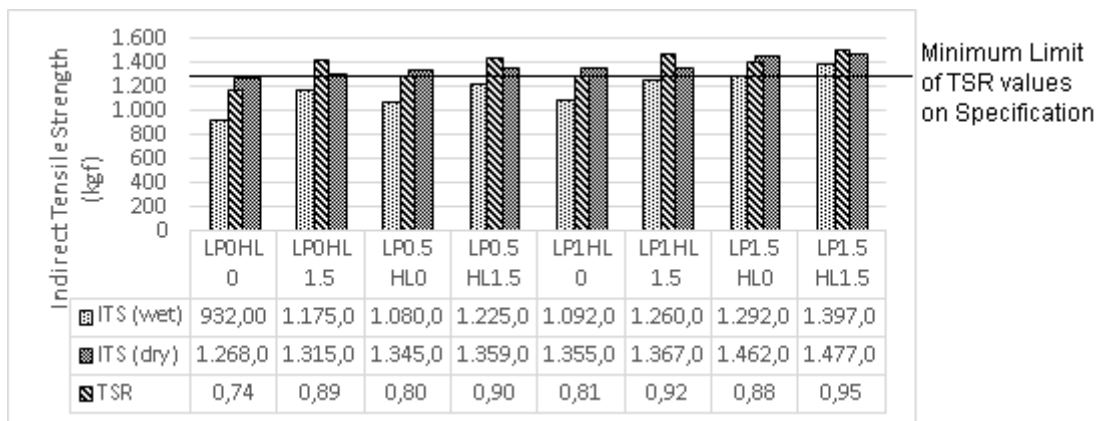
In this study, Marshall mix designs for the experiments on bituminous mixtures were made with samples containing 1.5% hydrated lime. The results shown in Figure 16 were obtained by mixing dry hydrated lime with wet aggregate gradation and adding different proportions of PVC containing bituminous materials to the aggregate mixtures. At this stage, the proportions of PVC added to the bituminous material were varied from 0% to 1.5%, while the hydrated lime containing 1.5% mixed with the aggregate was kept constant.

When the figure mentioned above is examined, it is understood that the flow values increase up to the samples prepared with 1.5% PVC waste and decrease from this content onwards. The reason for this decrease in the samples containing 1.5% PVC waste is thought to be that the additive used during mixing could not be distributed homogeneously in the bituminous material.

The classification of the samples used in the experimental studies is given in Table 6.

**Table 6.** Contents of the samples in accordance with the specimen nomenclature

Name of Specimen	Type of Aggregate	Type of Additive	Additive Content (%)
LP0HL0	Limestone	Unconditioned	0% for both
LP0HL1.5	Limestone	Hydrated lime	1.5% Hydrated lime
LP0.5HL0	Limestone	PVC	0.5% PVC
LP0.5HL1.5	Limestone	PVC and Hydrated lime	0.5% PVC, 1.5%hydrated lime
LP1HL0	Limestone	PVC	1.0% PVC
LP1HL1.5	Limestone	PVC and Hydrated lime	1.0% PVC, 1.5%hydrated lime
LP1.5HL0	Limestone	PVC	1.5% PVC
LP1.5HL1.5	Limestone	PVC and Hydrated lime	1.5% PVC, 1.5%hydrated lime



**Figure 17.** Indirect tensile strength (ITS) and tensile strength ratio (TSR) results

PVC waste is a synthetic material made from petroleum and salt that is used industrially to make doors and windows. The PVC used in the study is the material that accumulates as waste around the looms during production.

According to Figure 17, when the hydrated lime additive ratios were kept constant, a slight increase in TSR values was observed with increasing PVC waste content. It can be concluded that the addition of PVC waste to the bituminous material slightly increases the adhesion between bitumen and aggregate. The main increase was observed when 1.5% PVC waste was added to the specimens.

On the other hand, Figure 17 shows that the addition of hydrated lime to the bituminous mixture samples has higher TSR values than the samples prepared with PVC wastes. It can be concluded that the addition of hydrated lime to bituminous mixtures is significantly more effective than the addition of PVC waste in reducing the sensitivity of the mixtures to moisture. The reason why PVC wastes did not have as much effect on the stripping strength as hydrated lime is that PVC did not show sufficient binding properties at the current mixing temperature [37].

In the corresponding figure, the TSR values of bituminous mixtures without any additives were below the minimum value specified in the Highways Technical Specifications. The reason for this may be that the bituminous material used in the study may have aged due to uncontrollable environmental factors during the experimental studies. However, ignoring this possible factor, the use of both PVC waste and hydrated lime admixture in bituminous mixtures reduced the moisture sensitivity of the test specimens.

#### **4. Conclusion**

In the light of the data obtained within the scope of the study, the following conclusions were reached:

The stability of the mixture increased with the increase in the proportion of waste PVC added to the bituminous material. This can be attributed to the homogeneous distribution of polyvinyl chloride (PVC) in the bitumen without melting. In particular, the addition of 1.5% PVC to the bitumen significantly improved the stability values. Additionally, it is considered that the direct addition of PVC waste to bituminous materials with higher contents would have positive results in terms of increasing the strength of bituminous mixtures.

According to the Marshall Stability and Flow Test results, a significant increase in the flow rate was observed in bituminous mixtures with 1% additive content when PVC waste was added directly to the bituminous materials. On the other hand, the flow ratios of the samples prepared with higher additive content showed a tendency to decrease. The reason for this, especially for the flow values, can be attributed to the rigid nature of the PVC waste and its homogeneous distribution within the bituminous material without melting, thereby reducing the lateral flexibility. Although the flow values measured for all ingredients exceeded the upper limit of 4 mm, they are not far beyond the limit values and are at acceptable levels.

When the bituminous mixture samples prepared with all PVC contents were evaluated among themselves, the most significant increase in flow ratio was observed in the samples prepared with 1% PVC content, which showed the best resistance to lateral deflection without deformation. Accordingly, it is thought that the best resistance to lateral deflection without permanent deformation may also contribute positively to the workability of the flexible pavement during paving in the construction area.

Again, according to the results of Marshall stability and flow test, increasing the waste PVC content added to the bituminous materials increased the stability values, while decreasing the flow rates after 1% PVC content. This indicates that bituminous mixtures prepared with higher PVC contents will have limited lateral deflection on roads with high levels of heavy tonnage vehicle traffic, and thus their durability values will be better. According to this, the service life of the flexible pavement will be extended, and the service life will increase.

The effective recycling of PVC waste by using it as a binder modifier in asphalt pavements makes a very positive environmental contribution. In addition, by reducing the amount of bitumen used in asphalt pavements, almost all of which is imported, by approximately 16%, waste PVC will be transformed into an economic value. Thus, while decreasing environmental pollution, it will provide an economic contribution by reducing the amount of used bitumen that is imported.

When the bituminous mixture samples prepared with all PVC additive contents were analyzed, it was found that the addition of hydrated lime significantly reduced the sensitivity to moisture.

As the PVC content increased, it was seen more clearly that hydrated lime reduced the sensitivity to moisture, thus it was determined that the use of hydrated lime improved the water-induced damage of bituminous mixture samples prepared with waste PVC.

The study was conducted to evaluate the moisture sensitivity of adding PVC waste at different percentages to bituminous mixtures containing hydrated lime. The type of aggregate used in the experiments is limestone. Limestone is known to have a low silica content compared to other igneous rocks used in highway construction. The study can be extended to different igneous rocks with particularly high stability values and high moisture susceptibility. In addition, the interactions of different anti-stripping additives with PVC waste should also be evaluated.

## **5. Acknowledgement**

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## **6. Author Contribution Statement**

All authors contributed equally to this work

## **7. Ethics Committee Approval and Conflict of Interest**

The authors have no conflicts of interest to declare. All co-authors have seen and agree with the contents of the manuscript and there is no financial interest to report. We certify that the submission is original work and is not under review at any other publisher.

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