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Examination of the Properties of 70/100 Bitumen Modified with Activated Carbon

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Abstract

The volatile organic compounds are dispersed from asphalt fume to air during the construction of the pavement. These volatile organic compounds negatively affect the environment and the health of workers. Activated carbon is utilized to enhance the properties of bitumen and mitigate the impact of volatile organic compounds. In this study, the impacts of commercial activated carbon on the physical properties' of 70/100 bitumen were examined. 70/100 bitumen was modified for 1 hour at 2000 rpm and 150 ˚C with commercial activated carbon (0.5, 1, 2, 3 and 4%) using a high-speed mixer. Conventional bitumen tests (penetration, softening point and rotational viscometer) were performed on bitumen modified with activated carbon. Penetration index of modified bitumen was calculated. The findings indicate that the incorporation of activated carbon into 70/100 bitumen results in a decline in penetration values of all modified bitumen, accompanied by an increase in softening point and viscometer values. The storage stability test was conducted to determine the separation that may occur in the bitumen-commercial activated carbon phase and to determine its storability at high temperatures in modified bitumen tankers. Since activated carbon, added to 70/100 bitumen has little effect on the physical properties of modified bitumen, it is thought that activated carbon ratios in bitumen modification should be increased.

Keywords: Activated carbon, bitumen modification, conventional bitumen tests, storage stability.

1. Introduction

Activated carbon, defined as an absorbent material with large internal surface area and porous volume with the activated method applied to substances which have high carbon content, is obtained. Activated carbon has properties such as large surface area, high surface reactivity, large porous volume, appropriate porous distribution, and mechanical durability [1]. Since activated carbon contains a high amount of volatile content (approximately 50%), it is easily converted from solids or liquids to vapor [2]. Due to these properties of activated carbon, it is used in different areas (water purification, domestic, industrial wastewater, municipal wastewater treatment, desalination, refining and separating gases, odor and pollutant removal). Activated carbon is obtained from waste materials such as different waste products from various industrial by-products, municipal waste (waste plastic and tires, paper, garbage) and agricultural waste. The two methods for the preparation of activated carbon are physical activation and chemical activation. Activation method is an important factor affecting the porous structure of activated carbon. While chemical activation is carried out in one stage, physical activation is carried out in two stages. Firstly, after the carbonaceous materials are carbonized, the resulting char is activated with activation additives (CO_2, steam, air) at high temperatures. In chemical activation, the carbonaceous materials are impregnated with chemical activating agents (alkali and alkali metal-containing substances and some acids (H_3PO_4, KOH, NaOH, $ZnCI_2$, $CaCI_2$, H_2SO_4) for dehydrating purposes. KOH is the most widely used chemical activator and the activated carbon which has the best properties in chemical activation is obtained. The aim of both activation methods is to optimize the absorption properties of activated carbon used at different applications (environmental remediation, water purification, air treatment). It is evident that chemical activation is a more economical process than physical activation. The lower activation

temperatures, shorter processing times, larger surface areas and higher carbon efficiency that characterize chemical activation are key factors in this regard. Also, development of porous structure is better due to low temperatures used in chemical activation [3-10]. The surface morphology of activated carbon obtained from different biomass waste is affected by physical and chemical activation. Activated carbon from biomass flax fibre by both physical and chemical activation was obtained. It was observed that the activated carbon obtained by the physical activation a nodular and pitted surface morphology, while the activated carbon obtained by the chemical activation a smooth surface morphology [11]. In the study carried out by Demiral and Demiral [12], activated carbon, have high porosity and desired pore size distribution was obtained from cow dung using KOH and $ZnCI_2$ as chemical activation agents in chemical activation process. This study demonstrates that high surface area activated carbons can be prepared from the chemical activation of animal waste. Activated carbon for commercial purposes is generally obtained from different materials containing carbon such as peat, coal, lignite [13], nut shells, pitches, cokes [14], coconut, some commercial polymers, and wood [15]. In addition to this, activated carbon is obtained from biomass (wood sawdust, date stone [16], coconut shell [17], oleaster [18], elaeagnus and phoenix dactylifera seeds [19] and industrial waste which have high carbon content. In the study carried out by Akyıldız [1], the use of pomace waste material which occurs in our country where olive and olive oil production is high, as raw material in the production of activated carbon was investigated. Microporous activated carbon from coffee grounds and olive stones with physical (CO_2) and chemical (K_2CO_3) activation were prepared. It was determined that properties of activated carbon obtained from coffee grounds with the chemical activation were similar to properties of commercial activated carbon [20]. In the studies carried out by Çuhadar [21], Şen [22] and Sayın et al. [23], the properties of activated carbon from hazelnut shells were examined. Sayın et al. [23] stated that hazelnut shells obtained from Trabzon province in our country are a potential raw material source that can be used in activated carbon production.

During production, transportation and construction processes of bitumen, volatile organic compounds (VOCs) that are harmful to the environment and the health (skin, respiratory and nervous system) of workers are formed. VOCs that are known to be toxic and carcinogenic are dispersed from asphalt fumes to the air during pavement construction [24, 25]. The formation of VOCs is affected by factors such as asphalt temperature, asphalt oxidation and air humidity [26]. The concentration of VOCs which workers are exposed is between 0 and 139.97 mg/m^3 is. These VOCs also negatively affect the behavior of bituminous materials. Therefore, many techniques are investigated to create environmentally friendly asphalt pavement. Activated carbon is used as a modifier in bitumen to reduce the release of VOCs from bitumen. The porous structure of activated carbons absorbs the VOCs released from the bitumen material [27-29]. Xiao et al. [30] stated that when bitumen was modified with activated carbon, the weight of VOCs released from modified bitumen decreased by up to 30%. Also, it is known that the use of carbon-based modifiers such as activated carbon in bitumen modification improves properties of bitumen such as physical, rheological and strength. Carbon-based modifiers form a good chemical relationship with bitumen and thus the modifiers in the bitumen during bitumen modification are distributed homogeneously [31]. Bostancioğlu and Oruç [32] examined the effects of furan resin and activated carbon obtained from hazelnut shells on the performance of asphalt mixtures. They observed that the physical and mechanical properties of asphalt mixtures prepared by using bitumen modified with activated carbon at the ratios of 10, 15 and 20% and furan resin at the ratios of 4, 5 and 6% increased. Ani et al. (2024) [33] investigated the rutting and indirect tensile strength performance of asphalt mixtures prepared asphalt modified with powered activated carbon. The properties of asphalt modified wit powered activated carbon all temperatures have improved. Additionally, the rutting resistance of modified asphalt mixtures increased. Mamat et al. (2024) [34] modified 80/100 penetration grade bitumen with coconut shell activated carbon produced by the chemical activation of coconut shell. The rutting resistance, fatigue cracking, and stiffness of modified asphalt are improved. The use of 15% coconut shell activated carbon to improve the properties of 80/100 bitumen in bitumen modification has been suggested. Yalçın et al. (2025) [35] investigated the fatigue and fracture performance of asphalt mixtures containing activated carbon obtained from the olive waste pomace and slurry samples and styrene-butadiene-styrene (SBS). The fatigue performance of asphalt mixtures containing activated carbon obtained from waste materials increased significantly.

Ideal bitumen should be soft to resist thermal stress at low temperatures, hard to resist deformation at high temperatures, and resistant to fatigue cracking at moderate temperatures. The aim of this study is to investigate the effect of commercial activated carbon to reduce the consistency and improve the performance of 70/100 bitumen which is soft. Firstly, bitumen which has 70/100 penetration grade was modified with commercial activated carbon obtained from coconut shell. Commercial activated carbon has properties such as high surface energy, specific large surface area and porous structure. Physical properties (penetration, softening point, rotational viscometer) of bitumen modified with activated carbon at the ratios of 0.5, 1, 2, 3, and 4% were investigated. Furthermore, penetration index (PI) was calculated to ascertain temperature

sensitivity of all modified bitumen. Additionally, storage stability was carried out to examine the performance of bitumen modified with activated carbon at high temperature. The Kruskal-Wallis and Mann-Whitney U tests were performed to determine whether there was a statistically significant difference in test results.

2. Materials and Methods

In this section, the materials (70/100 bitumen and commercial activated carbon) and experimental methods used in the study are given.

2.1. 70/100 Bitumen and Commercial Activated Carbon

Bitumen, which has 70/100 penetration grade, was used in this study. Physical properties of 70/100 bitumen were determined with the help of penetration (TS EN 1426), softening point (TS EN 1427), specific gravity (TS EN 15326+A1) and rotational viscometer (ASTM D4402) tests.

Table 1. Physical properties of 70/100 bitumen.

Properties	70/100 Bitumen	
Penetration (25 ˚C)	87	
Softening point (˚C)	42.2	
Specific gravity (g/cm^3)	1.012	
Rotational viscometer (cP)	135 ˚C 282.5	165 ˚C 86.46

Commercial activated carbon obtained from coconut shell used in this study was obtained from Overwater Water Treatment Systems Industry and Trade Limited Company. The surface area of activated carbon is 1000-1500 m^2/g. Commercial activated carbon is microporous and pH neutral. Activated carbon which has a size of 0.6 – 2.6 mm was passed through No.200 sieve for use in bitumen modification. Bostancioğlu and Oruç [36] stated that use of activated carbon obtained from hazelnut shells smaller than 0.063 mm in bitumen modification was effective. According to this study, activated carbon (Figure 1) which has a size smaller than 0.075 mm at the ratios of 0.5, 1, 2, 3 and 4% was used to prevent the agglomeration of activated carbon in 70/100 bitumen.



Figure 1. Activated carbon.

2.2. Parameters of Bitumen Modification

The parameters used in the modification of bitumen with activated carbon were determined taking to account the studies occurred by Bostancioğlu and Oruç [31, 35], Mamat et al. (2024) [34] and Karimi et al. (2018) [37]. The parameters of bitumen modification are presented in Table 2.

Table 2. Parameters of bitumen modification.

Parameters used in bitumen modification	
Penetration grade of bitumen	70/100 bitumen
Modifier	Activated carbon
Mixing method	Wet mixing method
Mixing device	High-speed mixer
Mixing speed (rpm)	2000
Mixing temperature (˚C)	150
Mixing time (dk)	60
Ratios of modifier (%)	0.5, 1, 2, 3 and 4

2.3. Conventional Bitumen Tests

A series of tests were conducted to ascertain the physical properties of bitumen modified with activated carbon. These tests included determination of penetration, softening point and the use of a rotational viscometer. (Figure 2).



Figure 2. Conventional bitumen tests performed on bitumen modified with activated carbon.

The PI was calculated to determine temperature sensitivity of modified bitumen. PI values were calculated with the help of Equation (2.1).

$$PI = \frac{1952-500\log(Pen_{25})-20SP}{50\log(Pen_{25})-SP-120} \quad (2.1)$$

Where Pen_{25}, the penetration value of bitumen; *SP*, the softening point of bitumen.

Also, storage stability tests (TS EN 13399) were performed in order to determine the separation that may occur in the bitumen-commercial activated carbon phase and to determine its storability at high temperatures in modified bitumen tankers (Figure 3). According to these conventional bitumen test results, the compatibility of 70/100 bitumen and activated carbon will be determined.



Figure 3. Appearance of the top and bottom parts after conditioning at high temperature for storage stability.

2.4. Statistical Analysis

The Kruskal-Wallis test was preferred to statistically interpret the test results. Kruskal-Wallis test (non-parametric) compares two or more groups on a dependent variable based upon the non-normally distributed data. The Kruskal-Wallis test may be considered an extension of the two-group Mann-Whitney U test [38]. In this study, the effect of activated carbon on physical properties of 70/100 bitumen was investigated by considering one dependent (penetration and softening point results) and one independent variable (activated carbon ratios) of Kruskal-Wallis test. Following the determination of the difference between the groups through the implementation of Kruskal-Wallis test, the Mann-Whitney U test was performed to ascertain which groups exhibited a significant difference.

3. Results and Discussion

In this section, test results of bitumen modified with activated carbon at different ratios are given and obtained results are evaluated.

3.1. Results of Bitumen Tests

The penetration and softening point tests conducted within the scope of this study were performed on three samples for each ratio. A total of 36 samples were used for these two tests. According to penetration and softening point test results which are given in Figure 4, as the ratio of activated carbon added to 70/100 bitumen increased, penetration values of modified bitumen decreased, while softening point values increased.

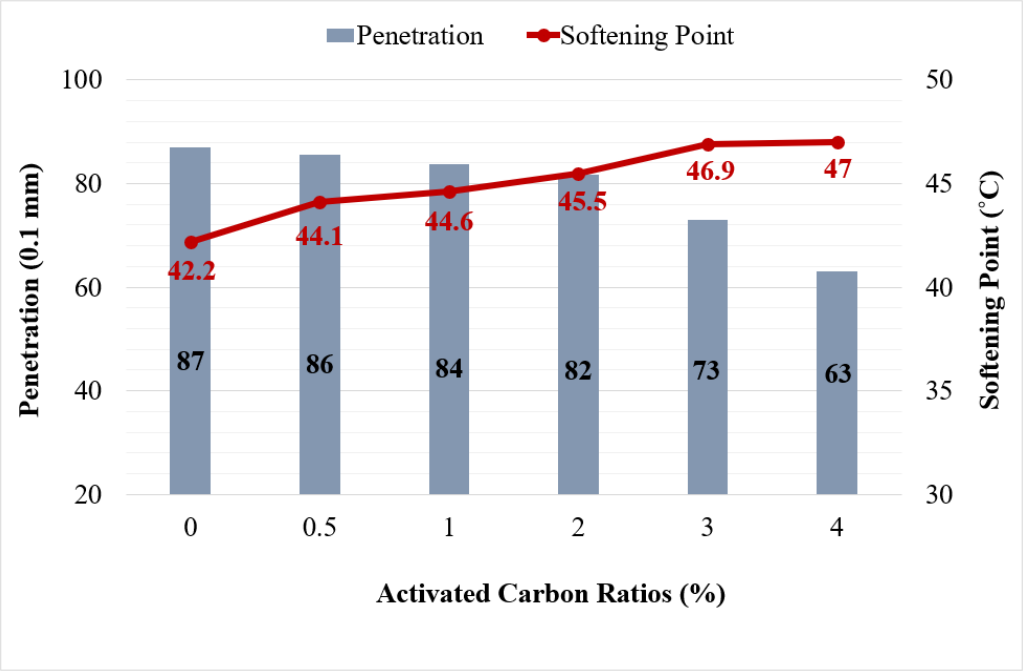


Figure 4. Penetration and softening point test results of all modified bitumen.

Compared to 70/100 bitumen, the penetration value of modified bitumen decreased by 1.15%, 3.45%, 5.75%, 16.09%, and 27.59% as the activated carbon ratio added to 70/100 bitumen increased, respectively. In the

softening point test results, the softening point of the modified bitumen increased by 4.50%, 5.69%, 7.82%, 11.14%, and 11.37% as the activated carbon ratio added to 70/100 bitumen increased, respectively. Recently, graphene oxide (GO), which is used as a carbon-based modifier to improve the performance of bitumen, is preferred because it has hydrogen bonding potential and high surface area, which can improve the functional behavior of bitumen [39,40]. Similar to the obtained results in our study, as the graphene oxide (GO) ratio added to the bitumen increased, the softening point values of the modified bitumen increased, while the penetration values decreased [41-43]. Seyrek et al. [44] determined that the consistency and stiffness of bitumen modified with activated carbon obtained from agricultural waste such as vinasse and marc increased. The decrease in the penetration value of the modified bitumen increases resistance to temperature degradation by increasing performance and durability modified bitumen during service life. The increase in the consistency and hardness of modified bitumen increases the resistance to rutting of modified bitumen [45]. In order to increase the high temperature performance of the bitumen, modified bitumen must have a high softening point and a low penetration value [46].

In addition, PI values of samples modified with activated carbon can be seen in Figure 5. When Figure 5 is examined, it is seen that according to the penetration index of 70/100 bitumen, the penetration index values of modified bitumen increased. The increase in the softening point, the decrease in the penetration value and the increase in the penetration index of the modified bitumen indicate that the modified bitumen is more resistant to thermal cracking at low temperatures and to deformation under traffic loads at high temperatures [47]. However, when PI values of bitumen modified with activated carbon are examined, PI value of bitumen modified with activated carbon at ratio of 4% decreased. Activated carbon which is added to 70/100 bitumen decreased the temperature sensitivity of all modified bitumen. Bostancioğlu and Oruç [36] stated that the temperature sensitivity of bitumen modified with activated carbon decreased according to reference bitumen. Also, they explained that size 1 and 2% additive ratio on the temperature sensitivity of modified bitumen are the most effective factors.

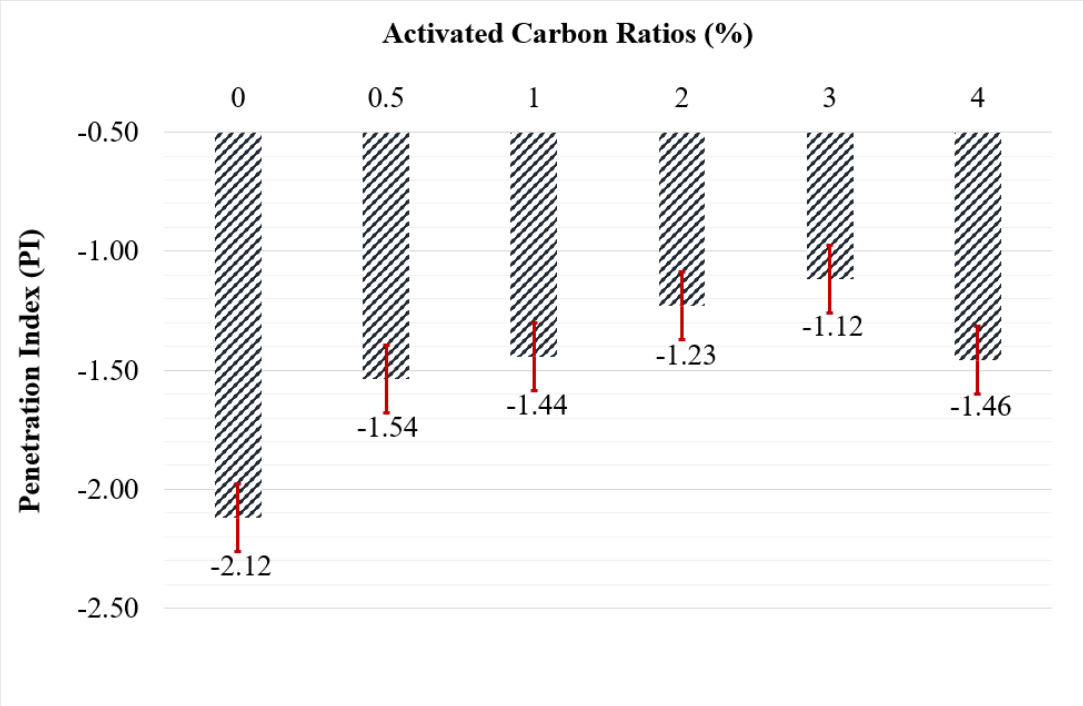


Figure 5. Penetration index values of all modified bitumen.

The rotational viscometer tests were conducted to ascertain mixing and compaction temperatures of modified bitumen samples. A total of 18 samples were prepared for the viscometer test, which was carried out at two temperatures (135°C and 165°C) on three samples for each ratio. The results of these tests are seen in Figure 6. Results obtained showed that as the ratio of activated carbon added to 70/100 bitumen increased, viscosity of modified bitumen increased. According to the results of the viscometer test carried out at 135°C temperature, the viscometer value of the modified bitumen increased by 9.13%, 11.96%, 19.04%, 26.62%, and 31.33% as the activated carbon ratio added to 70/100 bitumen increased, respectively. In the viscometer test carried out at 165°C, the viscometer value of the modified bitumen increased by 5.54%, 7.28%, 13.50%, 20.06%, and 22.95% as the activated carbon ratio added to 70/100 bitumen increased,

respectively. Similar to the obtained results, the viscometer values of bitumen modified with graphene oxide which has high carbon content such as activated carbon increased [42,48]. The increase in the viscosity value of the modified bitumen increases the performance and adhesion of its at high temperatures [49]. Bostancioğlu and Oruç [36] stated that the viscometer values of bitumen modified with activated carbon increased and the workability of modified bitumen decreased.

Mixing and compaction temperature ranges of modified bitumen are given Table 3. It was established that as the ratio of activated carbon added to 70/100 bitumen increased, the ranges of mixing and compaction temperature of the modified bitumen also increased. The ratios of activated carbon added to the 70/100 bitumen showed little difference in physical properties of the modified bitumen. It can thus be posited that the ratio of activated carbon added to bitumen should be increased. Storage stability determines whether modified bitumen can be stored safely in tanks at storage and distribution terminals before being transported to asphalt production facilities. In addition, it is determined whether there are significant changes in the physical and chemical properties of the modified bitumen during storage and transportation [50]. Within the scope of this study, a total of 24 samples were prepared to determine the softening point of the top and bottom site samples for each ratio. Results of storage stability test are given in Table 4. The softening point temperature difference between the top side and bottom side of the tubes which are used in the storage stability test should be 5 ˚C. The findings indicate that bitumen modified with activated carbon is capable of storage at elevated temperatures.

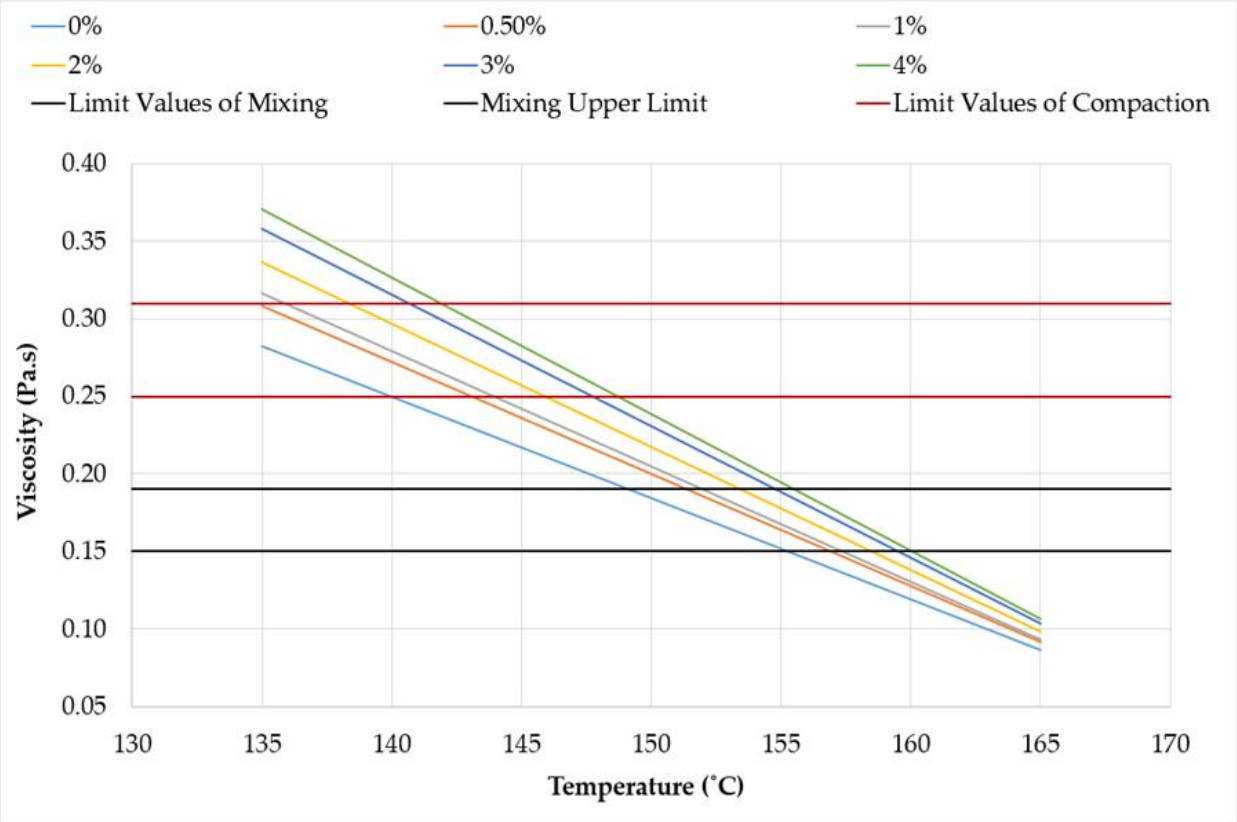


Figure 6. Rotational viscometer test results of all modified bitumen.

Table 3. Values of viscometer of bitumen modified with activated carbon.

Samples	Viscometer (cP) 135 ˚C	165 ˚C	Mixing Temperature Range (˚C)	Compaction Temperature Range (˚C)
0%	282.5	86.46	149.2 – 152.2	130.8 – 135.4
0.5%	308.3	91.25	151.4 – 154.1	134.8 – 138.9
1%	316.3	92.75	151.9 – 154.6	135.8 – 139.9
2%	336.3	98.13	153.4 – 155.9	138.3 – 142.1
3%	357.7	103.8	154.8 – 157.2	143.6 – 144.2
4%	371.0	106.3	155.8 – 157.8	141.9 – 145.3

Table 4. Results of storage stability test of all modified bitumen.

Ratio of Activated Carbon	Softening Point (˚C)		Difference
	Top Side (T)	Bottom Side (B)	
0%	46.7	45.9	0.8
0.5%	44.0	43.6	0.4
1%	45.4	44.9	0.5
2%	45.2	45.3	0.1
3%	45.9	45.9	0.0
4%	47.6	46.6	1.0

3.2. Results of Statistical Analysis

The Kruskal-Wallis test was performed to ascertain whether there was a statistically significant difference between activated carbon modified bitumen according to both penetration and softening point tests. The values obtained from these tests were interpreted in relation to various activated carbon ratios, with a p-value of less than 0.05 meaning level.

The Mann-Whitney U test was conducted to ascertain the significance between group variables and results are given in Table 5. When Table 5 was examined, 70/100 bitumen modified with 4% activated carbon showed a statistically significant difference between 70/100 bitumen according to both penetration and softening point test results. Thus, the addition of more than 4% activated carbon to 70/100 bitumen should be investigated.

Table 5. Results of statistical analysis.

Group Variables	The number of replicate (n)	Dependent Variables	Kruskal-Wallis Test p-values
4% - 0%	3	Penetration	0.022*
4% - 0.5%			0.099
4% - 1%			0.529
4% - 2%			1.000
4% - 3%			1.000
3% - 0%			0.193
3% - 0.5%			0.637
3% - 1%			1.000
3% - 2%			1.000
2% - 0%			1.000
2% - 0.5%			1.000
2% - 1%			1.000
1% - 0%			1.000
1% - 0.5%			1.000
0.5% - 0%			1.000
0% - 0.5%	3	Softening Point	1.000
0% - 1%			1.000
0% - 2%			0.569
0% - 3%			0.067
0% - 4%			0.011*
0.5% - 1%			1.000
0.5% - 2%			1.000
0.5% - 3%			0.471
0.5% - 4%			0.107
1% - 2%			1.000
1% - 3%			1.000
1% - 4%			0.685
2% - 3%			1.000
2% - 4%			1.000
3% - 4%			1.000

* Meaningful at 0.05 significance level

4. Conclusion

In this study, 70/100 bitumen was modified with commercial activated carbon at different ratios. Conventional bitumen tests were performed on all modified bitumen. The findings indicate that the incorporation of activated carbon into 70/100 bitumen results in a decline in penetration values of all modified bitumen, accompanied by an increase in softening point and viscometer values. Activated carbon which is added to 70/100 bitumen increased stiffness of the bitumen. Also, PI values were calculated by using penetration and softening point results. Calculations demonstrated that the addition of activated carbon results in a reduction of the temperature sensitivity of 70/100 bitumen. Storage stability tests were conducted in order to ascertain the performance of all modified bitumen at elevated temperatures. The findings indicate that bitumen modified with activated carbon is capable of storage at elevated temperatures. The Kruskal-Wallis test was conducted in line with the findings of the penetration and softening point tests. When penetration and softening point test results are taken into consideration, 4% activated carbon added to 70/100 bitumen created statistically significant difference on performance of modified bitumen.

In this study, the availability of between 70/100 bitumen and commercial activated carbon was determined. The ratios (0.5, 1, 2, 3, and 4%) of activated carbon added to the 70/100 bitumen showed little difference in terms of physical properties. According to the experimental results, it is recommended to investigate the use of activated carbon at higher ratios. Also, scanning electron microscopy (SEM) for the morphology, energy dispersive x-ray spectroscopy (EDS) for elemental analysis, x-ray diffraction (XRD) to determine the amorphous or crystalline structure, and Fourier transform infrared spectroscopy (FTIR) analysis to examine the chemical bonds of bitumen modified with activated carbon will be performed.

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Author’s Contributions

Öznur Karadağ: Performed the experimental studies, statistical analysis and result analysis, drafted and wrote the manuscript.

Gizem Kaçaroğlu: Assisted the experimental studies, statistical analysis and result analysis, helped in manuscript preparation.

Mehmet Saltan: Supervised the experimental study progress, result interpretation and helped in manuscript preparation.

Ethics

There are no ethical issues after the publication of this manuscript.

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