Research Article

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Rheological investigation and optimization of crumb rubber-modified bitumen production conditions in the plant and laboratory

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Abstract: Among the various bitumen additives, crumb rubber (CR) derived from waste tires stands out due to its economic and environmental advantages. The effectiveness of CR modification is influenced by several factors, including temperature, mixing speed, duration, and particle size. Excessive mixing speed and prolonged mixing in laboratory conditions can lead to the depolymerization of CR, which may adversely affect the elastic properties of the bitumen. In contrast, plant-scale production typically follows a more rapid and simplified process. In this study, bitumen containing 8% CR, modified under plant and laboratory conditions with varying mixing speeds and durations, was rheologically analyzed. Temperature and frequency sweep tests were conducted using a dynamic shear rheometer, and master curves were generated to evaluate the bitumen's viscoelastic behavior through various rheological models. The findings indicate that higher mixing speeds and extended mixing durations in laboratory conditions increase bitumen stiffness; however, excessive mixing leads to depolymerization, thereby reducing its elastic properties. The laboratory-produced CR modification demonstrates significantly superior performance compared to that generated in the plant. Furthermore, response surface methodology optimization analysis results indicate that the ideal conditions for mixing are a speed range of 3,000–4,000 rpm and a duration of 45–60 min.

Keywords: bitumen, rheology, crumb rubber, mixing conditions, master curve

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1 Introduction

Bituminous mixtures created with conventional bitumen are inadequate in withstanding the damaging effects of environmental factors and vehicular traffic, resulting in pavement degradation occurring more rapidly than expected [1]. The incorporation of materials such as recycled plastics and waste polymers promotes both economic viability and environmental sustainability. Crumb rubber (CR) derived from discarded tires is one of the most notable materials discussed in the literature in this context. CR significantly improves the rutting and fatigue properties of the mixture in bituminous hot mixes. Additionally, it has a beneficial effect on creep stiffness and thermal stability [2–4]. The addition of CR further enhances the bitumen's ability to resist cracking and boosts its ductility at lower temperatures [5]. It was noted that the rubber powder swells in the bitumen and fills with free volume due to heating, thus increasing the internal friction generated during intermolecular motion and resulting in the improvement of the high-temperature properties and aging resistance of CR-modified bitumen [6,7]. To avoid excessive viscosity and thus high production temperatures, it was recommended to use a maximum of 10% CR by weight of bitumen, and stone mastic asphalt (SMA) pavement produced at this ratio showed better resistance to permanent deformation and fatigue cracking compared to conventional SMA [8].

CR modification is usually performed in the laboratory in a high-shear speed mixer with different shear heads offering homogenization, particle reduction, emulsification, dissolution, and disintegration [9]. Four key parameters influence the characteristics of rubber asphalt: temperature, mixing speed, mixing duration, and particle dimensions. An examination of prior research indicates that the mixing temperature typically falls within the range of 170–190°C, with 180°C being the most frequently selected option. Additionally, the mixing speed is observed to vary significantly, spanning from 1,000 to 8,000 rpm, while the duration of mixing can range from 40 to 180 min [10–13]. According to road authorities of different countries, mixing time of 30–60 min, mixing temperature of 177–220°C, and CR size of 1.18–2.36 mm are recommended for CR modification [14]. CR have the capacity to expand to three to five times their initial volume as a result of absorbing the maltene fraction of the bitumen. This process results in a greater concentration of asphaltenes within the bitumen, consequently enhancing its viscosity [15]. Elevated mixing temperatures can enhance the expansion of rubber particles. Nevertheless, excessively high temperatures and extended mixing times may lead to the depolymerization of the rubber, causing the bitumen to harden undesirably. It is advisable to maintain mixing temperatures within the range of 160–180°C for rubber particles sized between 30 and 40 mesh [16]. Research indicated that conducting mixing at temperatures between 160 and 180°C for a period of 30–60 min enhances viscosity, elasticity, and resistance to permanent deformation. Nonetheless, this approach may result in phase separation during storage, attributed to the insufficient digestion of rubber particles [17–19]. Conversely, increasing the mixing speed to 8,000 rpm and subjecting the mixture to higher temperatures ranging from 200 to 260°C for prolonged periods exceeding 2 h can enhance the homogeneity and storage stability of the bitumen. However, these extreme blending conditions may result in the depolymerization of the rubber, which could adversely impact the properties of the bitumen [20]. To achieve high desulfurization and thermal degradation of the CR particles and thus improve the stability and processability of the bitumen, high preparation temperatures (220°C) and longer shear times (6 h), so-called terminal blending, were used in the modification and it was found that these conditions lead to CR particle breakdown and depolymerization of the rubber molecule and improve processability and stability up to a rubber content of about 40%, offering excellent low-temperature performance and fatigue resistance [21]. Under these severe processing conditions, the sulfur cross-linking bonds present in the CR particles may fracture and undergo depolymerization. This phenomenon results in the dissolution of CR in asphalt, ultimately producing a product that is significantly more homogeneous and stable than the modification at lower mixing temperature and speed [22].

In contrast to the wide variety of mixing options in the laboratory, simple and fast production is done in the modified asphalt plant. In the modified asphalt plant, hot bitumen is transferred from the storage silo to tank P1. In this tank, an additive is introduced in a quantity that corresponds to the weight of the bitumen. The resulting mixture of bitumen and additive is then pumped to tank P2, passing through a high-shear mixer located between tanks P1 and P2. If the modified bitumen is utilized immediately after this process, it is referred to as a single-pass system. Conversely, if the modified bitumen is redirected from tank P2 back to tank P1 via the high-shear mixer, it is classified as a two-pass system. In practical applications, a single-pass system is typically employed due to its high production efficiency. This research involved the production of bitumen samples sourced from a modified plant utilizing a single-pass system.

This research investigates how varying laboratory conditions for the preparation of modified bitumen influence its rheological properties. Specifically, the study compares laboratory-prepared CR modification, produced using a high-shear mixer under diverse conditions, with modifications conducted at a plant. The formulation of bituminous mixtures relies on the findings from laboratory experiments. Should the laboratory modifications yield superior results compared to those from the plant, it could lead to suboptimal mixture designs. Therefore, it is essential to establish laboratory conditions that replicate those of the plant for precise design outcomes. In this study, models derived from rheological tests were utilized to facilitate a comparison between CR-modified bitumen produced in both settings, ultimately identifying the optimal mixing conditions.

2 Materials and methods

2.1 Materials

In this research, the B 50/70 penetration grade bitumen sourced from the TÜPRAŞ Batman refinery, with its characteristics detailed in Table 1, was utilized. The CR employed in this investigation had a diameter of 0.6 mm and was produced through mechanical fragmentation. The chemical composition of CR is as follows; C: 68.3%, H: 7.1%, S: 1.7%, O: 22.9%, ash: 3%, and moisture: 0.5%. Both the CR and the bitumen were procured from the Turkish General Directorate of Highways.

Table 1: Properties of the pure bitumen

Properties	Standard	B 50/70
Penetration (0.1 mm), 100 g, 5 s	ASTM D5 [23]	64.00
Softening point (°C)	ASTM D36 [24]	55.00
Penetration index		0.60
Viscosity (cP, 135°C)	ASTM D4402 [25]	700.00
Viscosity (cP, 165°C)	ASTM D4402	200.00
Specific gravity	ASTM D70 [26]	1.024

2.2 Preparation of the modified bitumen

In the CR modification produced in the modified asphalt plant, CR was used at 8% by weight of bitumen. As per the data obtained from the site, the CR modification process is executed at a temperature of 170°C in a single pass utilizing a double tank system equipped with a mixer (Figure 1). The modified bitumen produced on-site was subsequently transported to the laboratory for rheological analysis.

The CR modification in the laboratory was carried out at 170°C under the same conditions as in the field. Modification was carried out in an insulated cylindrical container using a high-shear mixer (Figure 2) to ensure a homogeneous distribution of the materials. To investigate the effects of laboratory mixing conditions on the modified bitumen and to compare it with the bitumen obtained from the plant, bitumens were prepared at different mixing times and speeds. The modified bitumens were prepared by selecting stirring speeds of 1,000, 2,000, and 4,000 rpm and stirring times of 10, 30, 60, and 90 min. Prior to modification, the bitumen was heated to 170°C and then the CR additive was slowly added into the insulated container.

In the plant modification process, bitumen transferred from the storage tank was subjected to weighing and subsequently heated to a temperature of 170°C. Following this, 8% of CR was introduced into the mixing tank, where it underwent rapid mixing before being conveyed to the modified bitumen storage tank via a milling process. The resultant bitumen from this procedure is referred to as “plant.” Conversely, bitumens produced in the laboratory are designated by a two-number system, with the first number denoting the mixing speed and the second indicating the mixing duration. Figure 3 illustrates the nomenclature of bitumens along with the corresponding analytical methods.

Figure 1: Modified asphalt plant.

Figure 2: Laboratory type high shear mixer.

2.3 Methods

2.3.1 Temperature sweep test via dynamic shear rheometer (DSR)

In this study, rheological tests were carried out using a Bohlin DSRII rheometer under controlled-stress conditions to assess the viscoelastic behavior of bitumens. The tests were conducted over a temperature range of 52–76°C, ensuring a comprehensive evaluation of the bitumens’ high-temperature performance. A constant frequency of 10 rad/s was applied throughout the experiments, with a 25 mm diameter parallel plate geometry and a 1 mm gap opening to maintain standardized testing conditions (AASHTO T315). To preserve the integrity of the results, the stress amplitude for each bitumen was carefully adjusted to remain within the linear viscoelastic (LVE) region, preventing non-linear deformations that could distort the rheological measurements. This approach ensures that the obtained data accurately reflects the intrinsic material properties rather than artifacts introduced by excessive loading.

2.3.2 Frequency sweep test via DSR

The frequency sweep test is particularly valuable as it allows for the examination of the material’s behavior across a range of frequencies, simulating the conditions that asphalt pavements experience under traffic loads [27]. In conducting the frequency sweep test, bitumen samples are subjected to oscillatory shear loading at different frequencies, typically ranging from 0.01 to 10 Hz, while maintaining a constant temperature. The temperature is

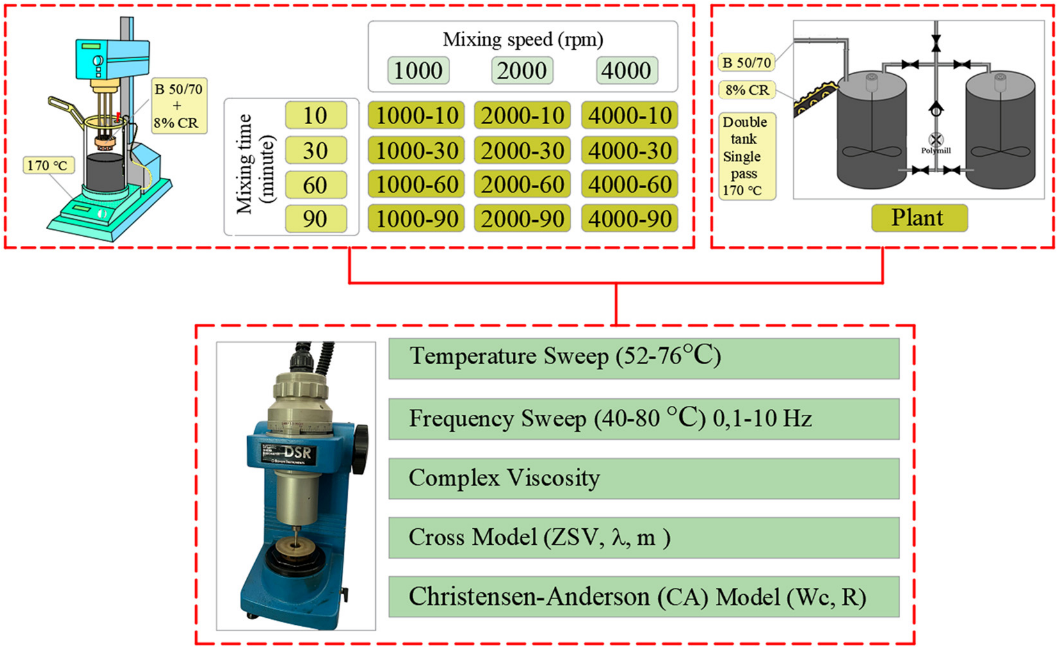

Figure 3: Nomenclature of bitumens and analytical methods.

often set at elevated levels, such as 40, 50, 60, 70, and 80°C, to replicate the high-temperature conditions that asphalt pavements encounter during service [28]. With this test, the speed of a vehicle traveling on the asphalt pavement is simulated and it is assumed that a loading frequency of 10 Hz corresponds to a speed of 60–65 km/h [29]. Frequency-sweep test was conducted within the LVE range of the bitumen. The experiment was performed in the range of 0.01–10 Hz with 10°C increments between 40 and 80°C.

Master curves were generated at a reference temperature of 40°C by applying the time-temperature superposition principle (TTSP), utilizing complex modulus values obtained from Frequency Sweep tests. The construction of master curves for asphalt bitumens offers significant advantages in comprehending the material's behavior in relation to temperature and loading frequency [30,31]. These curves facilitate the prediction of the rheological characteristics of bitumen across an extensive temperature spectrum and varying loading scenarios. In addition, master curves allow a more systematic comparison of aging effects, the effect of modifications on asphalt performance, and comparisons between different bitumen types.

2.3.3 Christensen-Anderson (CA) model

The analysis of frequency sweep master curves, particularly those constructed using the TTSP, allows for a comprehensive understanding of the rheological behavior of asphalt bitumens [32,33]. These master curves can then be analyzed using various rheological models, which provide insights into the material's performance characteristics under different loading and temperature conditions [34]. One such model is the CA model, which is widely used for characterizing the viscoelastic properties of bitumens [35]. The CA model is based on the assumption that the complex shear modulus (G^*) can be expressed as a function of frequency (ω) and temperature (T) (Figure 4). The model incorporates parameters that account for the material's behavior in both the linear and nonlinear viscoelastic regions, making it suitable for predicting the performance of asphalt under various conditions [36,37]. In this model, presented in equation (1), the rheological behavior is described in terms of G^* values as a function of the frequency applied to the bitumen. Numerous studies have been carried out with the CA model [37–39]. Following the analysis of the CA model, values for glass modulus (G_g), crossover frequency (ω_c), and rheological index (R) have been determined for both pure and modified bitumen (Figure 4).

G_g refers to the basic stiffness of the asphalt at low frequencies (long-term loading); a high G_g value indicates that the bitumen has high stiffness and resistance to deformation. ω_c represents the frequency at which the viscous and elastic modulus values are the same. It is also the point where the viscous asymptote and the glassy asymptote overlap. ω_c characterizes the overall hardness of the bitumen. A low ω_c value indicates that the bitumen can maintain its elastic properties even at low speeds and is more resistant to

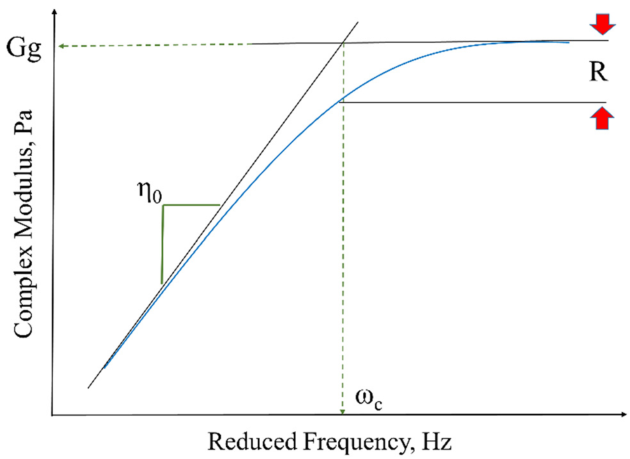

Figure 4: Definition of CA model.

sudden loading. R is defined as the difference between the complex modulus at ω_c and the intercept asymptotes. It is also called the shape factor. An increase in R indicates a decrease in the viscous properties and an improvement in the elastic properties of the bitumen at intermediate loading times and temperatures and gives the idea that it will show wider relaxation spectra.

$$|G^*| = G_g\left[1 + \left(\frac{\omega_c}{\omega}\right)^{\frac{\log 2}{R}}\right]^{\frac{-R}{\log 2}}. \quad (1)$$

2.3.4 Cross model

Theoretically, the complex viscosity of bitumens is related to the complex shear modulus, as expressed in equation (2) [40,41]. From this relationship, the magnitude of complex viscosity can be determined as the ratio of dynamic shear viscosity to angular frequency, as illustrated in equation (3).

$$\eta^*(\omega) = \frac{G^*(w)}{i\omega}, \quad (2)$$

$$|\eta^*(\omega)| = \frac{|G^*(w)|}{\omega}. \quad (3)$$

Here, $\eta^*(\omega)$ is the complex viscosity (Pa s); i is the imaginary part ($i^2 = -1$), and $|\eta^*(\omega)|$ is the magnitude of the complex viscosity (Pa s).

Complex viscosity, one of the key outputs of the frequency sweep test, is a critical parameter that reflects the material's resistance to flow. This property significantly influences the workability of asphalt mixtures [42]. The dynamically measured viscosity is termed "complex viscosity." Complex viscosity values are analogous to shear viscosity and can be modeled using various nonlinear flow models. Among these, the generalized Newton model effectively describes the nonlinear shear viscosity behavior and shear-thinning properties of bitumen [43].

The cross model is extensively utilized for its ability to capture the shear-thinning behavior of non-Newtonian fluids, including bitumens. This model, defined by equation (4), incorporates key parameters such as the infinite-shear viscosity (η_∞), zero-shear viscosity (η_0), time constant (λ), and a dimensionless exponent (m) [43,44].

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (\lambda\cdot\dot{\gamma})^m}. \quad (4)$$

Here, n is the viscosity value and $\dot{\gamma}$ is the shear rate. η_∞ and η_0 (Pa s) represent infinite and zero-shear viscosities. In other words, they are peak and trough viscosity values. λ (lamda) is the time constant and m is the dimensionless exponent. The parameter η_0 represents the viscosity of asphalt at zero shear rate and expresses the stiffness of the bitumen at high temperatures. The parameters λ and m determine how the bitumen responds to shear rate; a low value of λ indicates that the viscosity starts to decrease at lower speeds and the bitumen is more sensitive to shear thinning.

2.3.5 Response surface methodology (RSM)

RSM is a robust statistical and mathematical technique widely employed in experimental design and optimization studies [45,46]. RSM employs second-order polynomial models derived from empirical data to determine optimal response conditions, making it an invaluable tool in refining process parameters. One of the key advantages of RSM is its capacity to reduce experimental effort by minimizing the number of required trials, thus conserving time and resources [47]. Additionally, it is particularly effective in modeling non-linear relationships, allowing for the identification of optimal operating conditions with greater accuracy. By enabling systematic and data-driven decision-making, RSM contributes significantly to cost reduction, process enhancement, and overall system performance optimization.

3 Results and discussion

3.1 Complex modulus (G^*) master curves

The G^* values obtained from the frequency sweep test were used to calculate the master curves based on the

TTSP. Master curves were obtained at a reference temperature of 40°C and the results are shown in Figure 5.

The master curve graph presented in Figure 5 compares the rheological behavior of 8% CR-modified bitumens produced using different mixing speeds (1,000, 2,000, 4,000 rpm) and mixing times (10, 30, 60, 90 min). According to the results, the increase in mixing time and speed resulted in a generally increasing trend in complex modulus (G^*) values. The lowest G^* value was observed in the sample mixed for the shortest time (10 min) and at the lowest speed (1,000 rpm) (1,000-10), while the highest G value was obtained in the sample mixed for the longest time (90 min) and at the highest speed (4,000 rpm) (4,000-90). This indicates that the interaction and dispersion of CR with bitumen increases with mixing time and speed. Longer duration and higher speed mixing allow the rubber particles to swell more, interact better with the bitumen phase, and form a homogeneous network structure [48–50]. This interaction increases the elastic modulus of the bitumen, resulting in higher temperature resistance and stiffness.

On the other hand, excessively long mixing times or high mixing speeds may cause chain breaks in the polymer structures, resulting in possible deterioration of the rheological properties of the bitumen [51]. However, the results showed that the mixing time and speed under the conditions tested in the study improved the rheological performance of the bitumen up to a certain point. The bitumen from the plant exhibited a performance close to that of the sample mixed at 2,000 rpm for 30 min in the laboratory but showed slight differences depending on the modification process in the field conditions. This suggests that the variability between field and laboratory productions should be taken into account in terms of controlling the modification process. Furthermore, it is clearly seen that the master curves given in Figure 7 are quite smooth and thermorheologically simple.

3.2 CA model results

The master curves obtained for all bitumens were analyzed according to the CA model given in equation (1) and the CA model fit curve obtained for bitumen 1,000-10

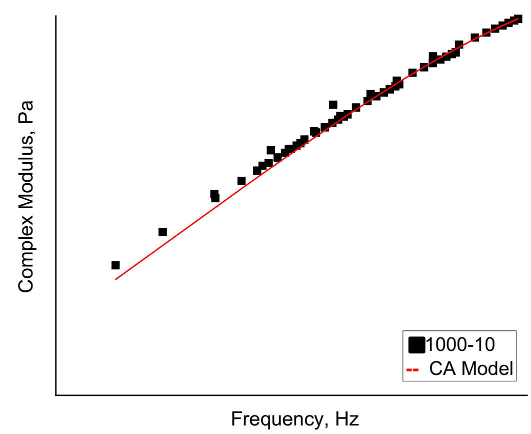

Figure 6: CA model fit of 1,000-10 bitumen.

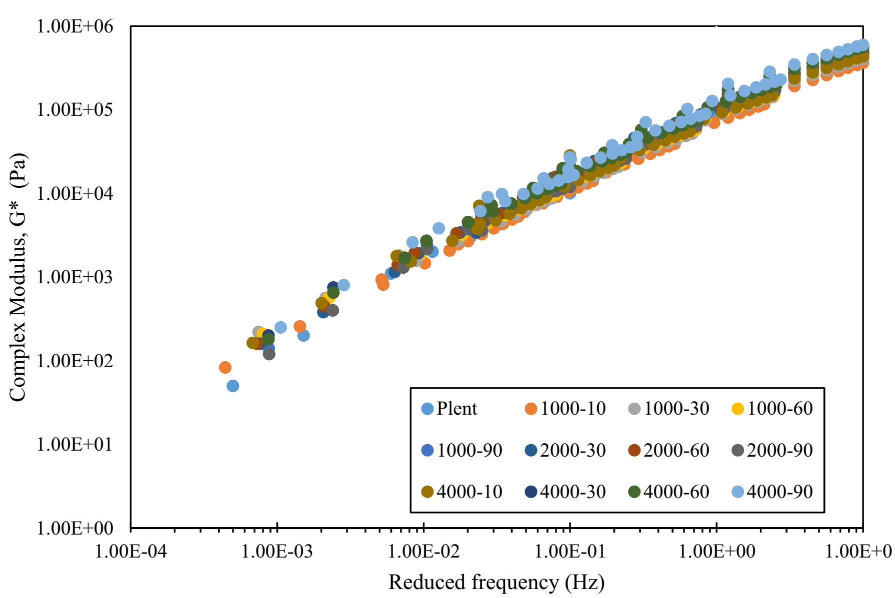

Figure 5: Complex modulus master curves of the bitumens.

is given in Figure 6. The CA model parameters are presented in Figure 7.

Figure 7 shows that the CA model was successfully applied to the experimentally obtained frequency versus complex viscosity values with high accuracy and low margin of error (R^2 > 0.99). Various studies have been carried out on the glass modulus (G_g) of 1 GPa in shear and most bitumens have been found to offer this value. Previous studies have suggested fixing the glass modulus (G_g) value to 10^9 and most bitumens have been found to have a G_g value of 10^9 [52]. In this study, the value of G_g was fixed at 10^9 and the other variables (ω_c and R) were left free. Upon examining Figure 7(a), it is evident that the 4,000-90 bitumen exhibited the lowest ω_c value, whereas the 1,000-10 bitumen demonstrated the highest ω_c value. Across all mixing speeds, a consistent decline in ω_c values was noted as mixing time increased. This reduction in ω_c values corresponds to a decrease in the frequency at which the elastic and viscous modulus values converge. The convergence of these two rheological parameters at lower frequencies suggests a transition of the material's primary curve toward the elastic domain. Furthermore, the combination of low mixing speed and duration adversely impacted the relationship between the CR and bitumen, which can be attributed to inadequate polymer swelling and insufficient modification [53]. In the case of 4,000-90 bitumen, increasing the mixing time and speed decreased the ω_c values and improved the elastic behavior. However, it should be kept in mind that too long time and excessively high speed may cause fragmentation in polymer structures after a certain point and the bitumen may become brittle by over-hardening.

"*R*," defined as the rheological index, refers to the shape of the master curve and is related to the width of the relaxation spectrum [54]. This parameter is also a very useful tool because of its sensitivity to changes in bitumen hardness in terms of loading time/frequency. Even small changes in bitumen hardness due to aging and chemical changes can cause significant changes in R values [55]. The increase in R value indicates the broadening of the

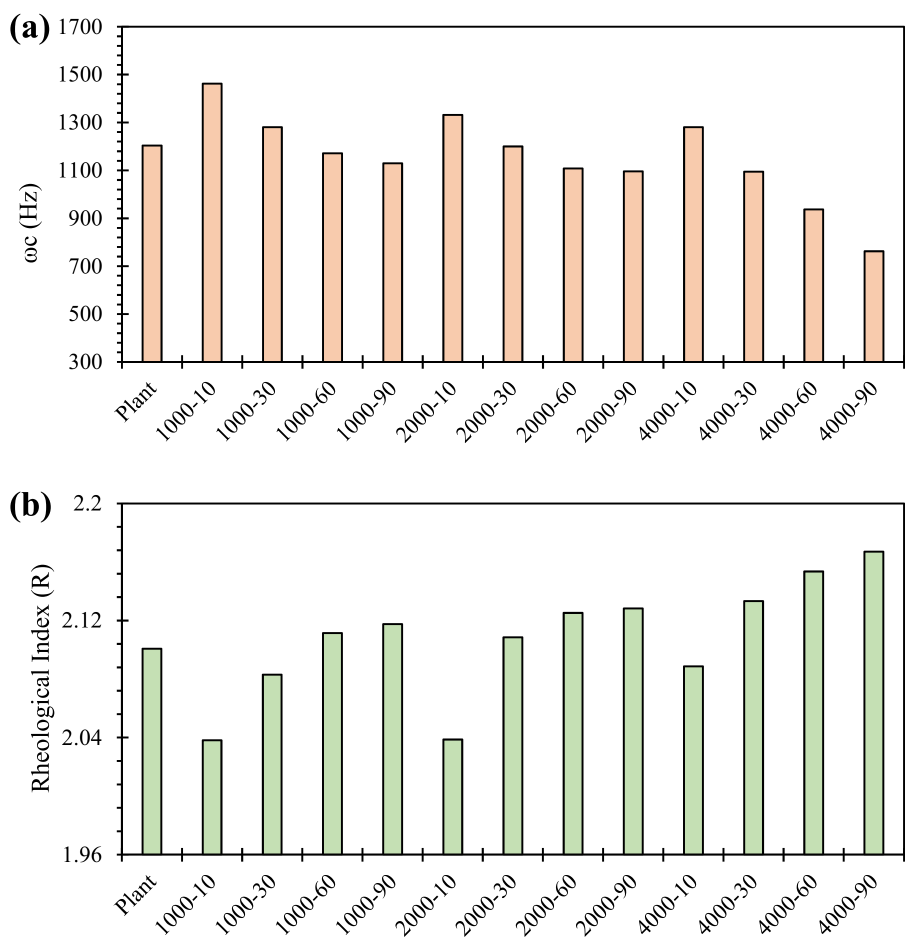

Figure 7: CA model parameters, (a) crossover frequency (ω_c) and (b) rheological index (R).

Table 2: ANOVA results for CA model analysis

Bitumen	Sum of squares	Mean square	*F* value	Prob > *F*
Plant	8.91×10^{11}	4.45×10^{11}	10254.52	6.90×10^{-64}
1,000-10	6.88×10^{11}	3.44×10^{11}	10716.87	2.40×10^{-64}
1,000-30	1.74×10^{12}	8.71×10^{11}	4547.296	1.92×10^{-55}
1,000-60	2.08×10^{12}	1.04×10^{12}	3127.476	1.45×10^{-51}
1,000-90	8.97×10^{11}	4.49×10^{11}	5887.595	4.02×10^{-58}
2,000-30	1.04×10^{12}	5.22×10^{11}	4749.128	6.82×10^{-56}
2,000-60	1.35×10^{12}	6.76×10^{11}	4155.148	1.66×10^{-54}
2,000-90	1.30×10^{12}	6.49×10^{11}	5538.332	1.73×10^{-57}
4,000-10	1.30×10^{12}	6.51×10^{11}	4741.042	7.10×10^{-56}
4,000-30	1.18×10^{12}	5.88×10^{11}	6024.105	2.32×10^{-58}
4,000-60	1.02×10^{12}	5.11×10^{11}	5735.463	7.51×10^{-58}
4,000-90	1.74×10^{12}	8.71×10^{11}	4548.131	1.92×10^{-55}

relaxation spectrum and is associated with increased stiffness and less viscous behavior. According to Figure 7(b), the lowest *R* value was observed in 1,000-10 bitumen and the highest *R* value was observed in 4,000-90 bitumen. Increasing the mixing speed and mixing time significantly increased the *R* values. Short time and low-speed mixing resulted in incomplete polymer-bitumen interaction and weak rheological behavior of the bitumen. The “*R*” value of the bitumen from the plant was closer to that of the bitumens mixed at 2,000 rpm. Analysis of variance (ANOVA) results for the CA model are given in Table 2.

The ANOVA statistically evaluates the effects of different mixing speeds and times on the rheological behavior of CR-modified bitumens (Table 2). High *F*-values and extremely low *p*-values were observed between the different groups, indicating a statistically significant effect of mixing time and speed on the rheological properties of the bitumen. Mean square values show that the variability in the bitumen increases with increasing mixing time and speed and has a systematic effect.

3.3 Cross model test results

The complex viscosity curves of the CR-modified bitumen were obtained at different mixing speeds and times and the bitumen obtained from the plant was obtained using a DSR device. With the complex viscosity curves obtained, the master curves given in Figure 8 were created based on the TTSP. While obtaining the master curves, the reference temperature was set as 60°C. The complex viscosity master curves of the plant and laboratory samples were fit to the Cross model as shown in Figure 9 to characterize the flow properties. Cross model parameters of all bitumens are given in Table 3.

Complex viscosity master curves were obtained to see the viscosity changes of bitumens at very low and very high frequencies at different temperatures in a single curve. As it is known, the preferred temperature for viscosity measurements and evaluations of bituminous materials is generally 60°C and therefore the reference temperature of the master curves was set as 60°C [56,57]. In one study, an experimental frequency sweep temperature of 60°C was reported to represent the high temperature of the road surface [58]. Upon analyzing Figure 8, it becomes evident that at lower frequencies, the bitumens exhibit characteristics of a Newtonian fluid. However, as the

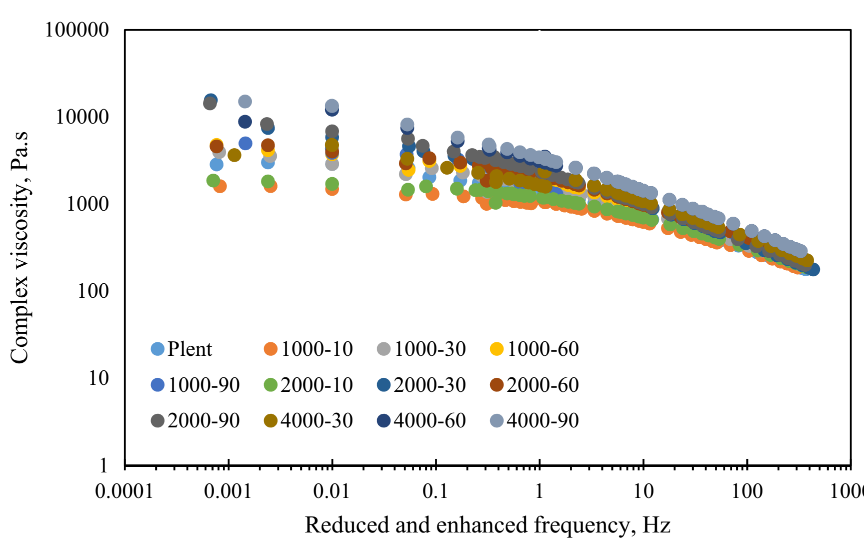

Figure 8: Complex viscosity master curves of bitumens.

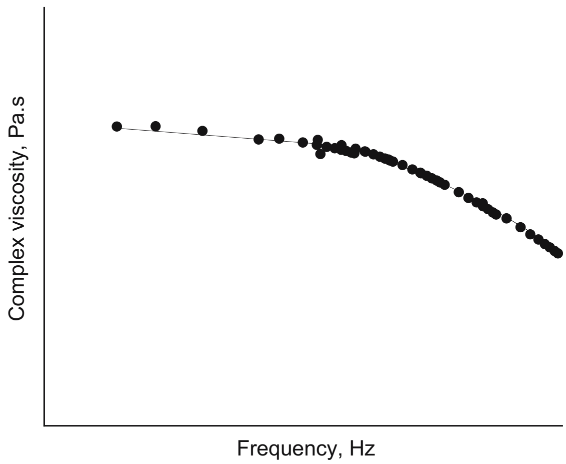

Figure 9: Cross model fit of bitumen complex viscosity master curve.

frequency escalates, their behavior transitions toward that of non-Newtonian fluids. A specific critical frequency marks the cessation of Newtonian behavior, after which a notable decline in bitumen viscosity occurs with increasing frequency. Additionally, both prolonged stirring time and higher stirring speeds resulted in elevated complex viscosity values. As frequency increased, the complex viscosity values converged. The bitumen labeled 4,000-90 demonstrated the highest viscosity, whereas the bitumen 1,000-10 exhibited the lowest viscosity. Notably, bitumens subjected to 10 min of mixing time showed the least viscosity values. These findings underscore the substantial impact of mixing duration on complex viscosity.

The variation of the parameters η_0 (zero shear viscosity [ZSV]), λ (characteristic time constant), and m (shear thinning index) obtained from the cross model analysis reveals the profound effects of mixing time and speed on the viscoelastic properties of the bitumen. ZSV is a parameter that reflects the quasi-static or Newtonian fluid-like behavior of the bitumen when the shear rate approaches zero. When the shear rate is very low, the effect of microstructure alignment due to flow is minimal and the intrinsic viscosity of the bitumen can be obtained through ZSV. ZSV is associated with permanent deformation resistance and therefore high ZSV values indicate improved rutting resistance. In other words, it is an indicator of a bitumen that is resistant to permanent deformation (rutting) that may occur in hot climates and under heavy traffic conditions [59–61]. The low ZSV values in bitumens produced with low mixing times and speeds indicate that the CR particles could not swell sufficiently and the polymer-bitumen interaction could not be fully achieved. This causes the bitumen to exhibit a more fluid behavior at low shear rates, while the viscous phase dominates the elastic phase, leading to a weaker resistance to deformation at high temperatures. However, a significant increase in ZSV values was observed with increasing mixing time and speed, and it was revealed that the bitumen

Table 3: Cross model parameters of bitumens

Bitumen	Parameter	Value	R^2	Bitumen	Parameter	Value	R^2
Plant	η_0	3246.713	0.97981	2,000-30	η_0	3409.388	0.96099
	λ	2.49748			Λ	3.05874	
	m	0.41182			M	0.42713	
1,000-10	η_0	1593.388	0.9879	2,000-60	η_0	4007.091	0.9871
	λ	0.27567			Λ	3.42313	
	m	0.26479			M	0.43935	
1,000-30	η_0	2341.806	0.94032	2,000-90	η_0	8347.991	0.95699
	λ	2.19266			Λ	3.61969	
	m	0.39276			m	0.45686	
1,000-60	η_0	4007.091	0.99289	4,000-10	η_0	2018.479	0.97806
	λ	3.11478			λ	2.30971	
	m	0.42965			m	0.3857	
1,000-90	η_0	4233.177	0.9696	4,000-30	η_0	19455.96	0.97806
	λ	3.12081			λ	3.77013	
	m	0.43633			m	0.45831	
2,000-10	η_0	1837.342	0.96911	4,000-60	η_0	23541	0.97251
	λ	0.29055			λ	3.78683	
	m	0.28513			m	0.47419	
				4,000-90	η_0	43080.47	0.9768
					λ	3.78013	
					m	0.51639	

became more rigid at low shear rates. This increase proves that the CR particles are better dispersed in bitumen, the polymer–bitumen interaction is strengthened and the structural stability of the modified bitumen at high temperatures is increased.

Similarly, the variation in λ values shows how the viscoelastic relaxation process of the bitumen is shaped. A low λ at low mixing times and speeds indicates that the bitumen reacts faster to the shear rate and the relaxation time is shorter. This suggests that the bitumen contains more viscous components due to the incomplete integration of the CR particles into the bitumen phase and its elastic response under load is poor. However, with increasing mixing time and speed, λ values increased and the viscoelastic equilibrium process of the bitumen shifted to longer time scales. This indicates that the relaxation process of the bitumen is prolonged and gains more pronounced elastic properties.

The high λ values obtained especially at high mixing speeds and for long periods of time prove that the CR particles are more homogeneously distributed in the bitumen and the modified bitumen becomes more durable at high temperatures. However, the saturation of this increase at a certain point at very long mixing times and extremely high speeds suggests that excessive hardening of the physical structure of the bitumen may occur and loss of flexibility may occur. If the samples were stirred at 4,000 rpm, the λ values reached the saturation point and the increase in λ values stopped significantly as the stirring time increased. This indicates that after a certain point, the mixing speed and duration trigger the aging of the material and disrupt the polymeric structure of the CR, leading to a decrease in the elastic properties of the bitumen [50,62].

The shear thinning index (m) is a critical parameter that defines the bitumen's sensitivity to frequency and shows a continuously increasing trend with the increase in mixing time and speed. The low values of the m index at low mixing speeds and short durations indicate that the bitumen is closer to Newtonian flow, meaning it exhibits a viscosity behavior independent of shear rate. This situation suggests that the CR modification has not fully occurred and that the bitumen does not exhibit sufficient shear thinning. However, with the increase in mixing time and speed, a significant rise in the m value was observed, indicating that the bitumen became more rigid at low shear rates and more fluid at high shear rates. This demonstrates that the bitumen's shear thinning behavior has improved and that it provides a more balanced viscoelastic response to deformation under load. Especially, the higher m values observed at high mixing speeds and long mixing durations indicate that the bitumen becomes more sensitive to shear rate due to the polymer–bitumen interaction and that the deformation resistance increases at high temperatures.

3.4 RSM results

Within the scope of the study, RSM analysis was conducted to reveal the relationships between the variables using the experimentally obtained data. RSM analysis was conducted for the $G^*/\sin\delta$ values given in Table 4. Table 5 provides the factor and response values. Additionally, the ANOVA results for the model determined in Table 6 are presented.

According to Table 6, the RSM analysis shows that the model is generally statistically significant (p < 0.0001) and has high explanatory power (R^2 = 0.9767, adjusted R^2 = 0.9712, predicted R^2 = 0.957). The effect of different factors and their interactions on the rheological properties of the bitumen was found to be statistically significant. Especially temperature (*A*), mixing speed (*B*), and mixing time (*C*), all showing significance at the p < 0.0001 level, exhibited strong effects on the rheological properties. Temperature has emerged as the most influential variable (F = 1179.92), followed by mixing time (F = 187.39) and mixing speed (F = 95.89). This situation indicates that temperature plays a critical role in altering the viscoelastic properties of CR-modified bitumen. Additionally, the binary interactions (*AB*, *AC*, and *BC*) and the second-order terms (A^2, B^2, C^2) were also found to be statistically significant, indicating that the interactions between the factors are important. The model's high Adeq Precision (51.0635) value indicates a strong signal-to-noise ratio and suggests that the model can make reliable predictions. The C.V. % (13.14) value indicates the variability of the model, showing that it has

Table 4: $G^*/\sin\delta$ values of all bitumens

Bitumen name	$G^*/\sin\delta$ (Pa)			
	58°C	64°C	70°C	76°C
1,000-10	12,288	6,092	3,020	1,538
1,000-30	21,590	11,066	5,732	2,985
1,000-60	23,456	12,090	6,682	3,560
1,000-90	24,364	13,167	7,173	3,881
2,000-10	12,804	6,180	3,177	1,642
2,000-30	21,931	11,212	5,831	3,144
2,000-60	24,539	13,175	6,921	3,657
2,000-90	26,895	14,187	7,595	4,083
4,000-10	20,083	10,435	5,401	2,829
4,000-30	27,198	14,885	8,003	4,320
4,000-60	31,515	16,788	9,517	5,103
4,000-90	42,164	23,427	13,131	7,461

Table 5: Factors and responses for RSM analysis

Name	Units	Minimum	Maximum	Coded low	Coded high	Mean	Std. dev.
Temperature (*A*)	°C	58	76	−1 ↔ 58	+1 ↔ 76.0	67.00	6.78
Mixing speed (*B*)	rpm	1,000	4,000	−1 ↔ 1,000	+1 ↔ 4,000	2333.3	1260.42
Mixing time (*C*)	min	10	90	−1 ↔ 10.00	+1 ↔ 90.0	47.50	30.63
Response	**Units**	**Minimum**	**Maximum**	**Mean**	**Std. dev.**	**Ratio**	
*G**	Pa	1,538	42,164	11831.6	9154.66	27.41	

a relatively acceptable margin of error. In conclusion, this analysis reveals that temperature, mixing time, and mixing speed significantly affect the rheological properties of the bitumen and that the model has the capacity to make reliable predictions. Equation (5) is a mathematical relationship that expresses the effect of temperature, mixing speed, and mixing time inputs on $G^*/\sin\delta$.

$$\begin{aligned} G^*/\sin\delta = {} & 9690.92 - 10464.6 \times A + 2699.08 \times B \\ & + 4089.84 \times C - 2040.75 \times AB - 2927.15 \times AC \\ & + 1397.23 \times BC + 4600.45 \times A^2 + 1375.99 \times C^2 \\ & + -1448.84 \times C^2. \end{aligned} \tag{5}$$

In Figure 10, RSM plots are presented, illustrating the effects of the variables temperature (*A*), mixing speed (*B*), and mixing time (*C*) on the complex modulus (*G**) of the CR-modified bitumen. Figure 10(a) presents the contour diagram showing the interaction between temperature and mixing speed. As the temperature increases, it is observed that the complex modulus decreases, while with the increase in mixing speed, the complex modulus rises. The same situation is observed in Figure 10(b) as well. Figure 10(b) and (c) are three-dimensional response surface graphs showing the interaction between temperature-mixing speed and temperature-mixing time, respectively, indicating that the complex modulus decreases as the temperature increases, but the complex modulus increases with the extension of the mixing time. Figure 10(d) analyzes the interaction of mixing time and mixing speed, clearly showing that the complex modulus increases with the rise of both factors. However, compared to the response graphs showing temperature-dependent changes, this effect is clearly weaker. Figure 10(e) analyzes the accuracy of the model by comparing the predicted and actual values. The data showing a distinct linear distribution proves that the model has high predictive power and that the RSM analysis provides reliable results. Finally, Figure 10(f) presents the model's results in a three-dimensional cube format, illustrating how the complex modulus values change with different combinations of variables. It is observed that the complex modulus decreases in high-temperature regions, but reaches its highest levels at low temperatures and high mixing speeds.

In this study, the effects of temperature, mixing time, and mixing speed on $G^*/\sin\delta$ values were analyzed using RSM. Within the framework of the experimental design,

Table 6: ANOVA results

Source	Sum of squares	df	Mean square	*F*-value	*p*-value	
Model	3.85×10^9	9	4.28×10^8	176.98	<0.0001	Significant
A – Temperature	2.85×10^9	1	2.85×10^9	1179.92	<0.0001	
B – Mixing speed	2.32×10^8	1	2.32×10^8	95.89	<0.0001	
C – Mixing time	4.53×10^8	1	4.53×10^8	187.39	<0.0001	
AB	7.68×10^7	1	7.68×10^7	31.79	<0.0001	
AC	1.31×10^8	1	1.31×10^8	54.32	<0.0001	
BC	3.72×10^7	1	3.72×10^7	15.4	0.0004	
A^2	2.01×10^8	1	2.01×10^8	83.08	<0.0001	
B^2	1.54×10^7	1	1.54×10^7	6.37	0.0159	
C^2	1.84×10^7	1	1.84×10^7	7.6	0.0089	
Residual	9.18×10^7	38	2.42×10^6			
Cor total	3.94×10^9	47				
Std. dev.	1554.15		R^2	0.9767		
Mean	11831.6		Adjusted R^2	0.9712		
C.V. %	13.14		Predicted R^2	0.957		
			Adeq Precision	51.0635		

different combinations of temperature, duration, and speed were evaluated, and the parameters providing the most suitable rheological properties were determined based on the obtained results. In the first stage, as a result of analyses conducted over a wide temperature range, the stabilization of 64°C was deemed appropriate, and the focus was on optimizing the mixing time and mixing speed at this temperature. To determine the optimal conditions, the numerical optimization module of the Design Expert software was used.

Numerical optimization is an approach aimed at finding the best combinations of factors that align with

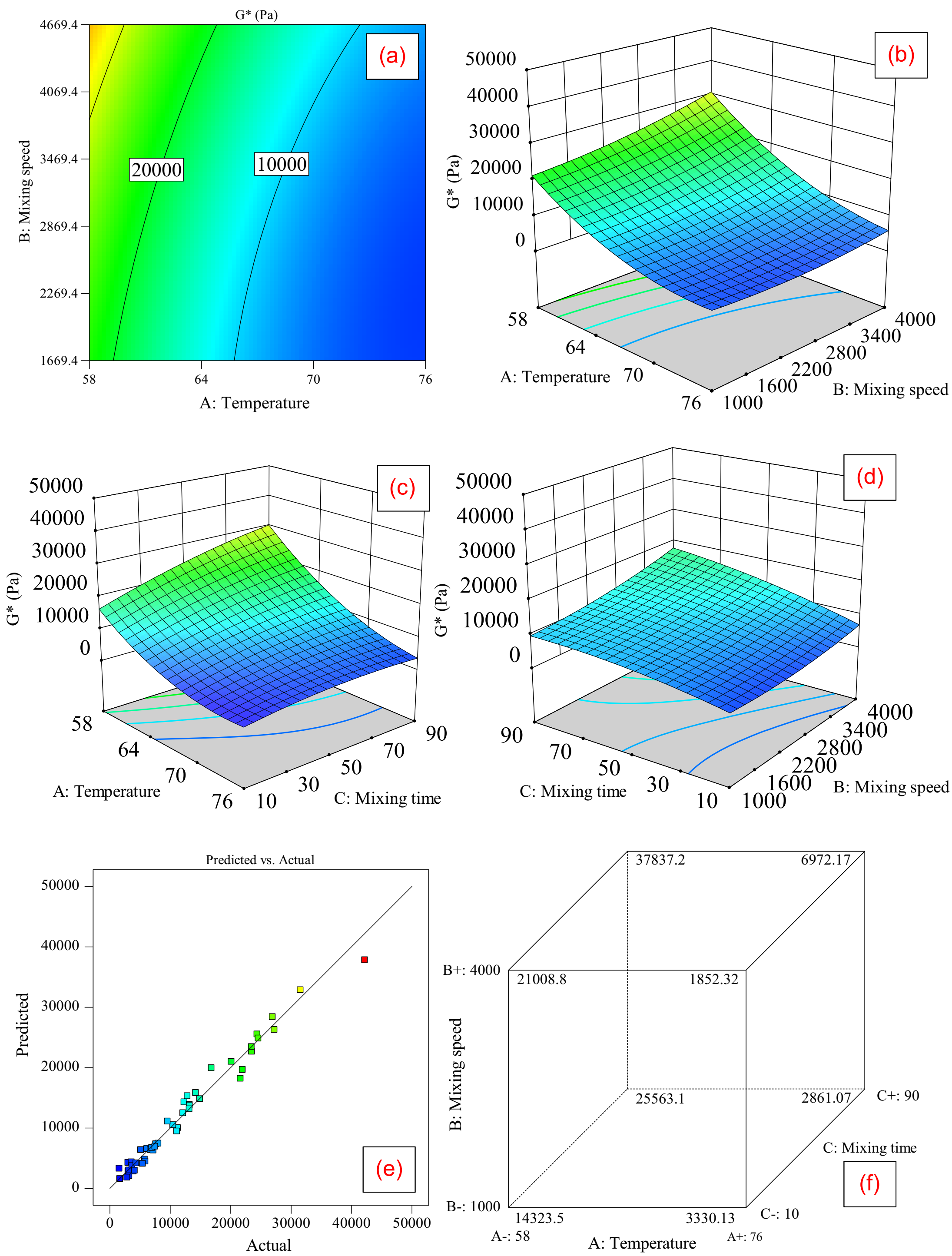

Figure 10: Effects of the variables temperature (a), mixing speed (b), and mixing time (c) on the complex modulus (G*), interaction of mixing time and speed (d), accuracy of the model (e), and model's results in a three-dimensional cube format (f).

Table 7: Optimization constraints for *G*/sin*δ*

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A: Temperature	Is equal to 64	58	76	1	1	*****
B: Mixing speed	Is in range	1,000	4,000	1	1	***
C: Mixing time	Is in range	10	90	1	1	***
*G**/sin*δ*	Is in range	6,000	24,000	1	1	***

the specified objectives. In this process, specific limits for mixing time and mixing speed were defined, with the aim of maximizing the *G*/sin*δ* value. The Design Expert software optimizes all response variables using the desirability function and determines the optimal levels for each factor. The optimization results obtained present the combinations of mixing time and mixing speed that will bring the material's rheological properties to the best level. Table 7 presents the constraints for the optimization process.

In Table 8, five solutions selected from different scenarios obtained as a result of the optimization are presented. When Table 8 is examined, there are scenarios for different mixing speeds and mixing durations, both relatively low and high. In previous studies, it has been stated that an increase in mixing time can lead to negative impacts on factors such as cost and environmental parameters (CO_2 emissions, etc.) [63,64]. In this context, it has been determined that solution number 4 would be more suitable based on the presented scenarios, and optimization ramp, desirability, and cubic graphs for this solution are provided in Figure 11.

Figure 11 presents the numerical optimization results conducted to achieve the ideal rheological performance of CR-modified bitumens. In the optimization process, considering the variables of temperature (*A*), mixing speed (*B*), and mixing time (*C*), the most suitable parameter combinations to idealize the complex modulus (*G*) value have been determined. The temperature value has been fixed at 64°C. Considering that lower temperatures cannot represent high-temperature values at the desired level for coatings and higher temperatures make the viscous components of the bitumen more dominant, it is thought that this temperature value will allow for a viscoelastic evaluation by reflecting both elastic and viscous properties. The most suitable value for the mixing speed has been determined to be 3,780 rpm. The increase in mixing speed enhances the dispersion of CR, thereby improving the mechanical stability of the bitumen. As a result of the optimization, the most suitable mixing time has been determined to be 45.8 min. In a previous study, it was determined that the ideal CR mixing time is between 45 and 60 min [53]. It can be said that these parameters are rational considering that the statistical significance of mixing speed on binding performance is higher than that of mixing time and that mixing time would also bring higher production costs. The desirability graph shows the extent to which the parameter combinations determined during the optimization process achieve the desired output. The desirability value for temperature, mixing speed, and mixing time has been determined to be 1.00, indicating that each parameter is within the most optimal ranges set during the optimization process and that the model provides reliable predictions. The overall desirability (combined) value has also been calculated as 1.00, indicating that the model has successfully identified the optimal points for all variables and that the obtained results are statistically reliable.

4 Conclusions

In this study, 8% CR-modified bitumen mixed in the laboratory at speeds of 1,000, 2,000, and 4,000 rpm for durations of 10, 30, 60, and 90 min, as well as bitumens directly taken

Table 8: Optimization solutions for ideal mixing speed and mixing time

Number	Temperature	Mixing speed	Mixing time	*G**	Desirability
1	64	3469.439	85.688	20620.96	1
2	64	3658.679	58.526	18365.633	1
3	64	2546.346	76.131	16515.1	1
4	***64***	***3780.092***	***45.825***	***16907.38***	***1***
5	64	2967.248	77.322	17957.8	1

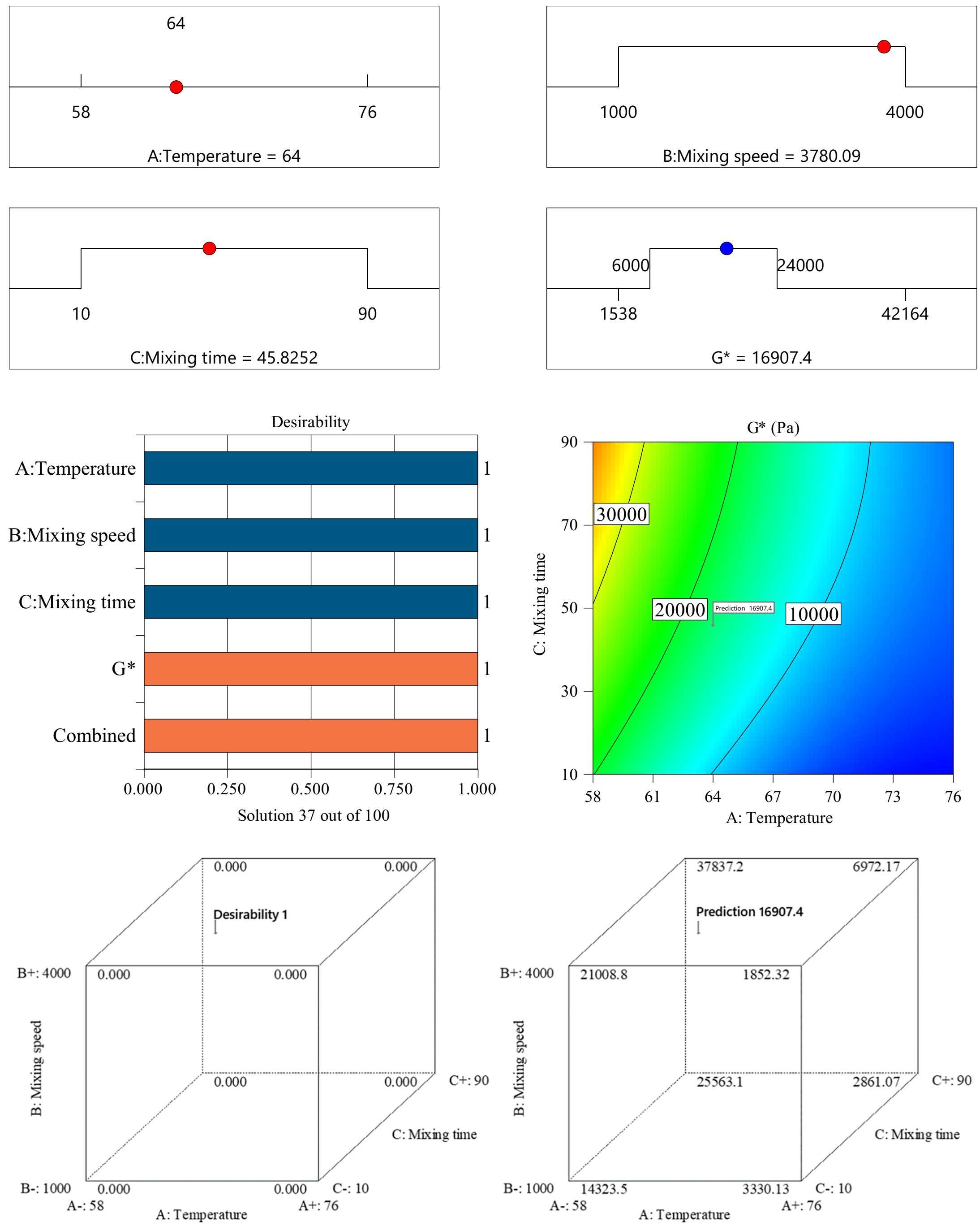

Figure 11: Numerical optimization results for different mixing speeds and mixing times.

from the modified plant in the field, was used. The results obtained in the study are compiled below:

- The master curves confirmed that mixing time and speed significantly influence the viscoelastic properties of CR-modified bitumens. An increase in both mixing speed and duration resulted in higher G^* values, indicating improved rubber-bitumen interaction, enhanced dispersion, and increased elastic response.
- The smoothness of the master curves suggests that the experimental data exhibit thermo-rheological simplicity, validating the quality and reliability of the measurements.
- According to CA model results, crossover frequency (ω_c) values decreased systematically with increasing mixing time and speed, demonstrating that the bitumen exhibited a more pronounced elastic behavior at lower frequencies. Higher mixing intensity enhances rubber

swelling and polymer dispersion, shifting the material behavior toward greater elasticity.
- The plant bitumen exhibited a rheological index (*R*) value similar to those of bitumens mixed at 2,000 rpm, reinforcing the importance of controlling field mixing conditions to match laboratory-produced materials.
- The CA model successfully predicted the experimental frequency-dependent complex viscosity values with high accuracy (R^2 > 0.99), indicating that the model is well-suited for characterizing the rheological behavior of these modified bitumens.
- The cross model parameters indicated that ZSV systematically increased with mixing speed and time, confirming that prolonged and high-speed mixing improved rubber-bitumen interaction.
- The relaxation time, λ, consistently increased with both mixing time and speed, indicating that the modified bitumen became more elastic and took longer to relax under stress. Especially in 4,000-60 and 4,000-90 bitumens, the increase in λ values reached the saturation point.
- The RSM analysis revealed that temperature had the most dominant effect on the rheological properties of CR-modified bitumens, followed by mixing time and mixing speed. Longer mixing times and higher speeds generally enhanced G^*, improving rubber dispersion and elasticity. Contour plots confirmed that optimizing mixing parameters is essential to maintain a balance between workability and performance.
- The optimization results identified 3,000–4,000 rpm mixing speeds and 45–60 min mixing times as the optimal range for maximizing bitumen elasticity and deformation resistance. The desirability function confirmed that process optimization is essential for balancing high-temperature stability with low-temperature cracking resistance, ensuring a durable and high-performance modified bitumen. In addition, it can be inferred that to achieve an effective modification of the CR, it is necessary to implement multiple passes within the plant, and the duration of mixing should be prolonged. Comparison of the performance of modified bitumen produced at different pass numbers and plants with those produced under laboratory conditions is an issue that can be addressed in future studies.

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