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Effect of Bitumen Production Process and Mix Heating Temperature on the Rheological Properties of Hot Recycled Mix Asphalt

Emiliano Prosperi [1], Edoardo Bocci [2,*] and Maurizio Bocci [1]

[1] Dipartimento di Ingegneria Civile Edile e Architettura, Università Politecnica delle Marche, Via Brecce Bianche, 60131 Ancona, Italy
[2] Faculty of Enginnering, Università degli Studi eCampus, 22060 Novedrate, Italy
* Correspondence: edoardo.bocci@uniecampus.it

Abstract: Heavy traffic loads require the replacement of damaged pavements, so a huge amount of reclaimed asphalt pavement (RAP) material is now available and must be recycled in order to avoid landfill and to achieve both environmental and economic benefits. The most common and profitable solution to reuse RAP is associated with the hot recycling technique, as it allows recovering both solid and binding components of RAP. Several factors influence the performance of hot recycled mix asphalt (HRMA). Among those, this paper focuses on the role played by the origin of the virgin bitumen, i.e. the oil-distillation process, and by the mixing temperature adopted during HRMA production. The objective was to evaluate the rheological properties of mixtures produced using a high amount of RAP (50%), two different rejuvenators, two mixing temperatures (140 °C or 170 °C) and two neat bitumen types derived from different distillation processes (visbreaker and straight-run). The results showed that the addition of RAP led to an increase in the dynamic modulus and a decrease in the phase angle, while the use of rejuvenators partly tended to rebalance these characteristics. The visbreaker bitumen showed a higher sensitivity to short-term aging than the straight-run, determining higher mix stiffness and lower viscous features. The higher mixing temperature also determined an increase in the complex modulus and a reduction in the phase angle as a result of the higher mobilization of the aged bitumen from the RAP.

Keywords: reclaimed asphalt; rejuvenator; 2S2P1D; mixing temperature; straight-run bitumen; visbreaker bitumen

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1. Introduction

Due to heavy traffic loads and environmental conditions, more and more asphalt pavements are failing prematurely, and their reconstruction has become a necessity [1]. This procedure involves the milling of the damaged layers, generating the by-product named reclaimed asphalt pavement (RAP). The question is: What can be done with the RAP? The alternative to landfill is to reuse it in the production of new asphalt pavements, achieving many economic and environmental benefits [2,3]. The use of high amounts of RAP in road construction is already a widespread practice in many countries. For instance, Germany, the United States, the Netherlands and Japan have started to manufacture mixtures with 100% RAP in asphalt plants: this represents the dawn of the concept of "total recycling" in pavement engineering [4].

The commonest solutions for RAP reuse involve cold and hot recycling techniques. Cold recycling consists in blending RAP with bitumen emulsion (or foamed bitumen), hydraulic binders (Portland cement, hydrated lime), water, and in some cases virgin aggregate [5]. Since RAP is not heated, it is typically supposed to behave as a "black" aggregate, meaning that the bitumen in the RAP does not have binding properties and is considered a solid. On the other hand, hot recycling consists in the inclusion of RAP in

new hot mix asphalt (HMA). Through heating or by contact with hot virgin aggregate, the bitumen in the RAP softens and can interact in some way with the other HMA components (virgin aggregates, virgin binder, and rejuvenator) [6]. This represents the most profitable reuse method, because it allows recovering both the solid and binding components of RAP. However, since RAP comes from old pavements and contains aged bitumen, there are many issues related to the performance of HMA that include hot recycled RAP (also called hot recycled mix asphalt—HRMA). In particular, the use of RAP leads to the production of a stiffer and more brittle mix [7]. Recent studies showed that in HRMA with 50% RAP, stiffness could increase by up to 25–60% compared to HMA with only virgin components, leading to cracking problems. On the other hand, rutting and moisture resistance are likely to be better or similar to those of conventional mixtures as the percentage of RAP increases [8].

In order to produce HMA with high amounts of RAP, it is necessary to use a rejuvenator [9]. The main purpose is to restore the properties of the aged asphalt binder to levels comparable with a virgin one [10]. Several additives have been used as rejuvenators, including bio-oils [11–15], waste minerals [16–19], or vegetable [20–23] oils. Since the amounts of RAP in HRMA are steadily increasing and many products have been proposed as rejuvenating agents, a deep understanding of the way in which they modify the properties of the aged bitumen is required, leading more and more researchers to investigate this aspect in detail in the last few years [24–26].

As well as the type and content of the rejuvenators [11,12], the mechanical properties of HRMA depend on other factors, such as the amount of RAP [27], the content, type, and aging degree of the bitumen contained in the RAP [28–30], the degree of binder activation and blending [31], the RAP heating procedure during HRMA production [32,33], and the type of virgin bitumen (neat or polymer-modified soft binders) [34]. However, two further variables, which are often neglected or underestimated, influence the performance of HRMA: the virgin bitumen production process and the HRMA manufacturing temperature.

Regarding the type of virgin binder used, a recent study shows that virgin bitumen with the same performance grade (PG) deriving from different oil sources will not always lead to mixtures with the same performance [35]. Moreover, in addition to the oil source (that can hardly be managed, even at refineries), the type of distillation process to which the crude oil is subjected can influence the characteristics of the HMA as well.

The single phases of crude oil are separated during distillation, due to the differences in boiling and condensing temperatures [36]. A typical distillation process involves a first step in which lighter components are separated, subjecting the crude oil to a temperature of about 350 °C at atmospheric pressure. The residue of the first step is then subjected to a higher temperature, around 350–425 °C, under a controlled pressure ranging from 1 kPa to 10 kPa. The residue of the second process is called straight-run bitumen [37]. Moreover, if the residue of this second process is subjected to another step of thermal distillation at temperatures between 455 °C and 510 °C, visbreaker bitumen is produced [38]. Visbreaking allows refineries to reduce the amount of the residue produced (i.e., the bitumen), as it allows a further recovery of lighter products, such as diesel and gas. This penalizes the quality of the resulting bitumen, which is more rigid, brittle, and susceptible to aging [39]. Some years ago, Giavarini [40] studied visbreaker (VB) and straight-run (SR) bitumen obtained from the same crude oil. He repeated tests, such as penetration and softening point, immediately and after 1 year, during which all bitumen was subjected to the same treatment (i.e., controlled heating to simulate aging) and stored under the same conditions in the laboratory. He found that the effect of aging was much more pronounced for the VB. In particular, starting from average penetration values of 175 [0.1 mm] and 155 [0.1 mm] for SR and VB bitumen, respectively, the average penetration decrease for the VB binder was more than 50% compared to 25% for the SR. Moreover, the penetration index, which was originally similar for both bitumen types, became appreciably lower for VB, indicating that the characteristics of VB products change significantly during storage.

Summarizing, the products derived from visbreaking show higher temperature susceptibility, lower oxidation resistance, and more rapid changes in physical properties. Considering the amount of VB bitumen that is marketed in Europe, more detailed and recent information is needed on the differences between such binders and straight-run ones and on the correlations between the severity of the visbreaking process and the stability of the bitumen. Very often, pavement technologists classify bitumen only according to penetration index or PG without considering the distillation process from which it derives, which greatly affects the characteristics of the HMA and its aging.

Temperature plays an essential role in bitumen aging, as it can accelerate chemical modifications [41]. For this reason, it is fundamental to avoid bitumen overheating by keeping temperatures low during HMA manufacturing. In fact, this can cause more severe short-term aging for both virgin and RAP binders [42], leading to stiffer and excessively brittle mixtures. In HMA plants, virgin aggregates are often overheated as a function of the target mix temperature and as a consequence of the introduction of RAP into the mix in relation to its temperature, humidity, and quantity. The stronger the aggregate overheating is, the more severe the thermal shock for the bitumen when it comes into contact with the aggregate particles. Moreover, the effectiveness of the rejuvenators can also be influenced by the temperature at which the HRMA is produced. On the other hand, a low mix temperature can result in poor workability and therefore in high air void content, leading to a higher risk of moisture damage, raveling, rutting, and cracking [43].

2. Objectives and Experimental Program

Considering the lack of papers concerning these aspects in the literature, and in order to provide a deeper comprehension of the influence of the variables highlighted before, this paper focused on the effects determined by the virgin bitumen distillation process and by the temperature during HRMA mixing on the material performance. In particular, the study evaluated the rheological properties of mixtures produced using a high amount of RAP (50%), two alternative kinds of rejuvenator (coded with the letters A and B), two mixing temperatures (140 °C and 170 °C), and two different neat bitumen types derived from visbreaking or straight-run distillation processes. Table 1 summarizes the mixtures tested, while Figure 1 shows the experimental program in a flowchart.

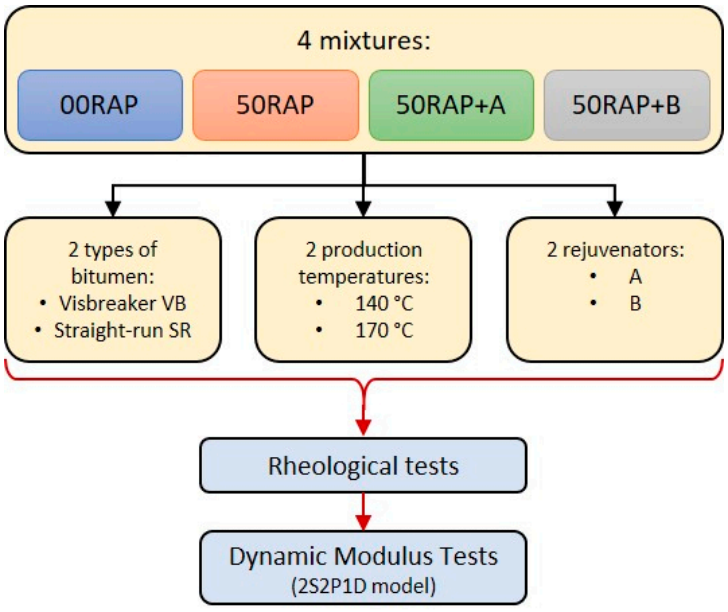


Figure 1. Experimental program.

Table 1. Investigated mixtures.

Mix Code	Virgin Bitumen	RAP Content [%]	Rejuvenating Agent	Mixing Temperature [°C]
00RAP_VB_170	Visbreaker	0	-	170
50RAP_VB_170		50	-	170
50RAP_VB_170 + A		50	A	170
50RAP_VB_170 + B		50	B	170
00RAP_VB_140		0	-	140
50RAP_VB_140		50	-	140
50RAP_VB_140 + A		50	A	140
50RAP_VB_140 + B		50	B	140
00RAP_SR_170	Straight-run	0		170
50RAP_SR_170		50	-	170
50RAP_SR_170 + A		50	A	170
50RAP_SR_170 + B		50	B	170
00RAP_SR_140		0	-	140
50RAP_SR_140		50	-	140
50RAP_SR_140 + A		50	A	140
50RAP_SR_140 + B		50	B	140

3. Materials and Specimen Preparation

Two bituminous mixtures, one without RAP (00RAP) and one containing 50% RAP by aggregate weight (50RAP), were designed in compliance with Italian specifications. Two fractions of RAP (0–8 and 8–16), two fractions of coarse limestone (12–16 and 6–12), limestone sand (0–6), and limestone filler were used. In order to determine the bitumen content, the "white" gradation curves of the two RAP fractions and the characteristics of the bitumen contained in the RAP, bitumen extraction using trichloroethylene (EN 12697-1) and rotary evaporation (EN 12697-3) were carried out. The 8–16 RAP fraction and the 0–8 RAP fraction showed 4.8% and 5.1% binder contents by RAP weight, respectively. The aged binder extracted from the RAP showed 13 [0.1 mm] penetration at 25 °C (EN 1426) and 77.1 °C softening point (EN 1427).

The aggregate fractions were proportioned to obtain mix gradations in compliance with the Italian specifications for a binder layer. The gradation curves of the mixtures with and without RAP (Figure 2) were similar and laid within the reference envelope provided by specifications.

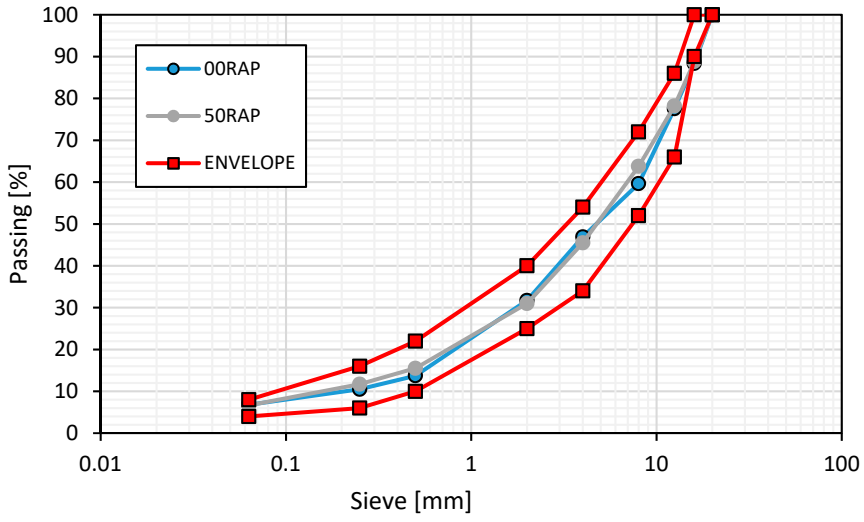


Figure 2. Gradation curve of the mixtures.

Two 50/70 neat (not polymer-modified) virgin bitumen types, typically used in Italy to produce bituminous mixtures, were selected: a bitumen obtained as the residue from a visbreaking process (VB) and a straight-run bitumen. The physical properties of the bitumens are presented in Table 2. As it can be noted, VB and SR bitumens are very similar in terms of penetration and softening point. Moreover, the same bitumens were investigated in previous studies [44,45] and showed a similar rheological behavior (almost superimposed G^* master curves).

Table 2. Physical properties of the bitumens.

ID	Penetration at T = 25 °C [0.1 mm]	Softening Point [°C]	Retained Penetration after RTFOT [%]
VB	62	50	>50
SR	63	49	>50

For all the mixtures, a total binder content of 5.2% by mix weight was fixed. The HRMA including 50% of RAP included a virgin bitumen content of 2.8% by mix weight. This value was calculated as the difference between the total binder content (5.2% by mix weight) and the RAP binder content (2.5% approximately). Even if this assumption corresponds to 100% reactivation of the RAP binder (very unlikely), it was made to avoid the execution of a complete mix design, which would have been very time and material consuming and could have led to different binder dosages (adding a further variable to the problem).

Two different rejuvenators were used (Table 3):

- Rejuvenator A: a mix of different chemicals and consists of modified polyamines and vegetal oils.
- Rejuvenator B: miscible crude tall oil derived from pine wood processing in the paper industry, and contains fatty acids and resin acids and is unsaponifiable.

Table 3. Physical properties of the rejuvenators.

ID	Density @ T = 20 °C [g/cm^3]	Flash Point [°C]	Kinematic Viscosity @ T = 25 °C [mPa × s]
A	0.80	>150	45
B	0.93	>295	98

The FTIR analysis was carried out on the pure rejuvenators. The absorbance spectra in Figure 3 shows that both the additives have a complex chemical structure, related to the presence of many peaks with wave numbers < 1200 cm^{-1}. Moreover, the two rejuvenators have unsaturated compounds (band at 3010 cm^{-1}) and ester C = O groups (band at 1742 cm^{-1}). In particular, it has to be remarked that the ester band at the wave-number 1742 cm^{-1} is highly marked for rejuvenator B. Differently, rejuvenator A has a smaller ester band, but presents peaks corresponding to N−H bends (1589 cm^{-1}) and N−H stretches (3400 cm^{-1}), denoting the presence of amines.

The additive content was fixed at 9% and 6% by RAP binder weight, respectively, for rejuvenators A and B, replicating the dosages adopted at the HMA plant where the raw materials were sampled. Two ways to add the additives into the mixtures were used out of the multiple possibilities [46], simulating the typical plant process for the selected rejuvenators. Additive A was blended for 10 min with the virgin binder at the same heating temperature of the aggregates (at 140 °C or 170 °C), using a mechanical mixer. Subsequently, the virgin binder containing the additive was kept in the oven for 30 min before the mix production, in order to restore the exact temperature. Differently, additive B was sprayed on the RAP at room temperature. Then, the RAP damped with the additive was placed in the oven together with the other virgin materials to reach the mixing temperature.

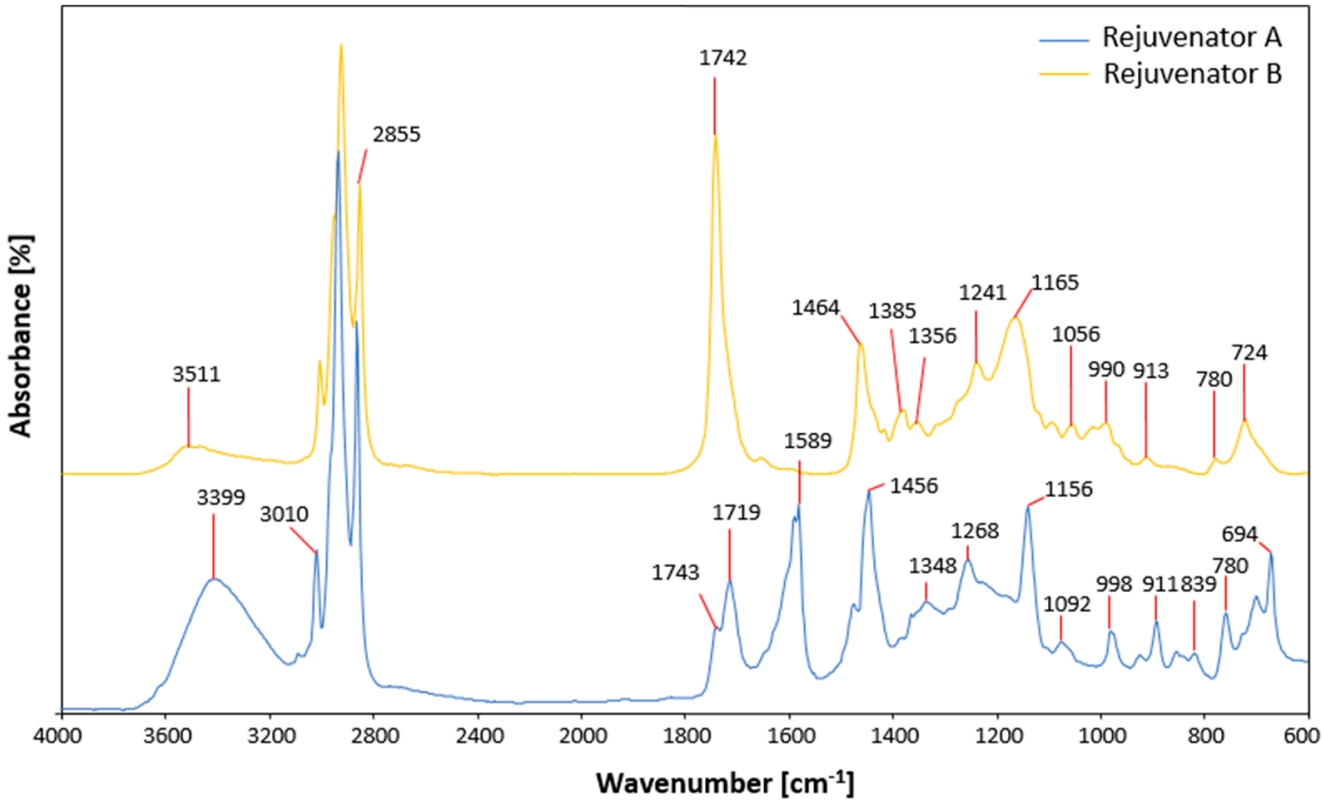
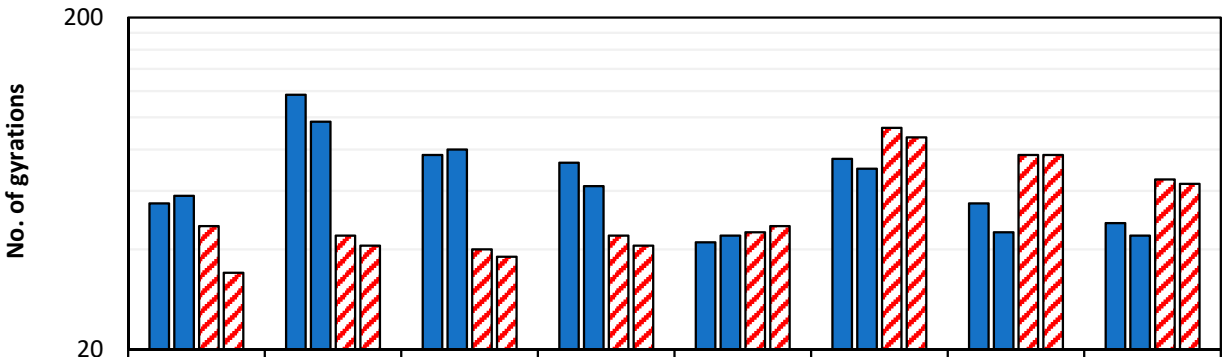


Figure 3. FTIR spectra of the two rejuvenators.

The following laboratory mixing procedure was followed: the aggregates and the RAP were heated in the oven, according to EN 12697-35, either at 140 °C or 170 °C for 3 h while the virgin binder was heated for 90 min. The materials were mixed by means of a mechanical mixer, initially placing only the coarse aggregates and the RAP, then adding the bitumen, and finally the filler. Then, the loose bituminous mixtures were kept in the oven for 30 min to reproduce the hauling time and equalize temperature within the mix. In the 30 min conditioning in the oven and the following compaction procedure, the mix temperature was again 140 °C or 170 °C. In the end, two cylindrical specimens for each mixture with 150 mm diameter were made using a Superpave gyratory compactor (EN 12697-31), fixing the final height of the specimens to 150 mm. The mass of the loose mix of each specimen (6250 g) was determined in order to obtain a final value of 4% for the air void content of the cored samples.

Figure 4 shows the number of gyrations applied to each specimen.

From the graph, it can be observed that the number of gyrations was always lower than 120 (the usual limit used by the Italian specifications), denoting a good mix workability for all the materials, even for high RAP contents and low mixing temperatures. In addition, it can be noted that the decrease of the mix temperature from 170 °C to 140 °C did not largely affect the workability. Indeed, the mixtures with VB showed a lower number of gyrations for the mixing temperature of 170 °C, while the mixtures with SR were slightly more compactable at 140 °C. In general, it was observed that the presence of RAP determined an increase in the number of gyrations, due to higher binder viscosity and thus lower workability, while the use of rejuvenators allowed decreasing the number of gyrations.



	00RAP_VB	50RAP_VB	50RAP_VB_A	50RAP_VB_B	00RAP_SR	50RAP_SR	50RAP_SR_A	50RAP_SR_B
140 °C_1	55	117	77	73	42	75	55	48
140 °C_2	58	97	80	62	44	70	45	44
170 °C_1	47	44	40	44	45	93	77	65
170 °C_2	34	41	38	41	47	87	77	63

Figure 4. Number of gyrations applied to each specimen.

After cooling them to room temperature, the specimens were cored to a diameter of 75 mm. Then, a thickness of 15 mm was cut from the top and the bottom of the cylinders. Each face was leveled by using a two-component resin in order to obtain a final height of about 120 mm and a perfectly smooth plane for testing. Finally, 3 pairs of strikers were glued on the lateral surface of the specimens in order to fix the linear variable displacement transducers (LVDT). The aspect of the specimen at each step is shown in Figure 5.



Figure 5. Preparation of each specimen for the test.

4. Test Methods

The complex modulus E^* of the specimens was measured through uniaxial cyclic compression tests. A servo-hydraulic universal testing machine (UTM-30) was used. The load cell was used to monitor axial stress, while the axial strain was measured on the middle part of the specimen (measuring base of 70 mm) using three LVDT placed 120° apart. A haversine compression loading was applied to obtain a target vertical strain amplitude of 50 microstrain (50×10^{-6} mm/mm). Four temperatures (5 °C, 20 °C, 35 °C, and 50 °C) and eight frequencies (ranging between 0.1 and 20 Hz) were investigated. Two samples of each mixture were tested.

The 2S2P1D rheological model (see Figure 6) was used to fit the experimental data [47]. The model consists of a series of a linear dashpot, two parabolic elements and a spring of stiffness $E_\infty - E_0$, assembled in parallel with a second spring (E_0) (Figure 5). The mathematical representation of the model can be described through the following expression:

$$E^*(i\omega t) = E_0 + \frac{E_\infty - E_0}{1 + \delta(i\omega\tau)^{-k} + (i\omega\tau)^{-h} + (i\omega\beta\tau)^{-1}} \quad (1)$$

where ω is the frequency, k and h are the parabolic element constants ($0 < k < h < 1$), E_0 is the static modulus when $\omega \to 0$, E_∞ is the glassy modulus when $\omega \to \infty$, δ is a dimensionless

shape factor, the parameter β is proportional to dashpot viscosity η ($\eta = G_\infty \times \beta \times \tau$), i is the unit imaginary number, and τ is characteristic time. Based on the time–temperature superposition principle (TTSP), τ can be determined as in Equation (2):

$$\tau(T) = a_T \times \tau_0 \quad (2)$$

where a_T is the shift factor at temperature T and $\tau_0 = \tau(T_0)$ is the characteristic time at reference temperature T_0 (equal to 20 °C).

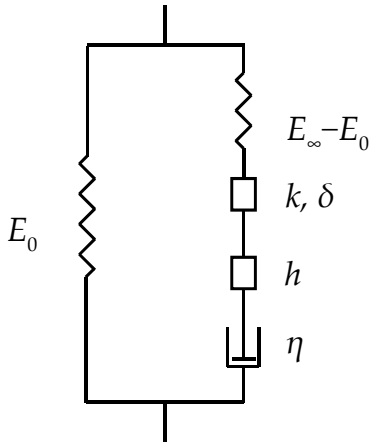


Figure 6. 2S2P1D model.

TTSP must be applied to fit this model with the experimental data. This principle states that the same rheological characteristics can be obtained at different temperatures by multiplying frequencies by a shift factor. This latter was defined using the William, Landel, and Ferry (WLF) equation [48]:

$$\log(a_T) = \frac{-C_1 \times (T - T_0)}{(C_2 + T - T_0)} \quad (3)$$

where C_1 and C_2 are constants, T is the temperature, and T_0 is the reference temperature.

Figure 7 highlights the correlation between the 2S2P1D model parameters and the shape of the master curve in the Cole–Cole plot: the static (E_0) and glassy (E_∞) shear moduli represent the intersection with the axis ($\phi = 0$), k and h are proportional to the angles that the curve generates with the real axis, and δ defines the height of the pinnacle point [49].

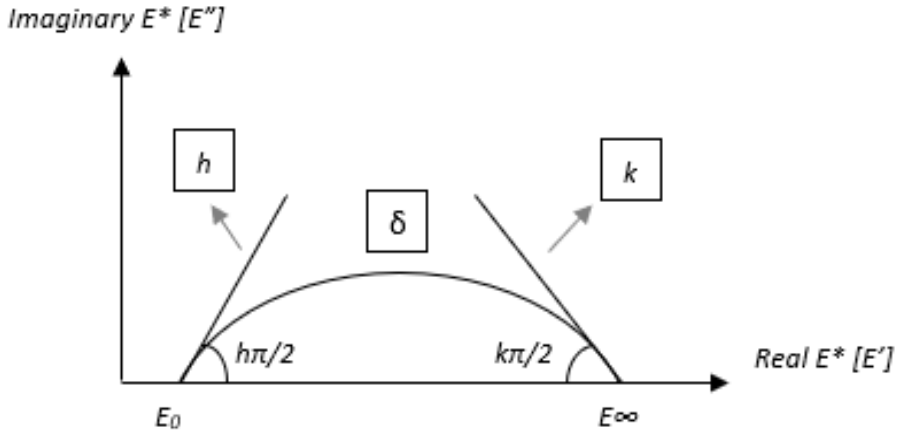


Figure 7. Definition of the 2S2P1D parameters.

With the aim of obtaining the 2S2P1D constants, the superposition of the experimental data to the model was achieved by minimizing the following sum of errors for each measured temperature and frequency:

$$Error = \left(\frac{|E^*|_c - |E^*|_m}{|E^*|_c}\right)^2 + \left(\frac{\phi_c - \phi_m}{\phi_c}\right)^2 \quad (4)$$

where $|E^*|_c$ and $|E^*|_m$ are respectively the calculated and measured norm of the complex modulus and ϕ_c and ϕ_m are respectively the calculated and measured phase angles.

Identical values of h, k, δ, β and τ_0 were assumed for the two specimens of the same mixtures, as they are commonly associated with the properties of the binder phase, thus exhibiting a small sample-to-sample variability [50].

Over the last few years, the Glover–Rowe (*G-R*) parameter has been used to evaluate the stiffness and relaxation characteristics of bitumen and correlated with the field performance of asphalt pavements [51–53]. This index is calculated from the values of $|G^*|$ and ϕ at a 15 °C temperature and at an angular frequency of 0.005 rad/s. Ogbo et al. [54] proposed using the mixture-based *G-R* parameter to assess the intermediate-temperature cracking resistance of the mixtures directly from the Black Space (ϕ vs $|E^*|$) data. Conversely, the temperature–frequency combination that is indicated in the original *G-R* formulation (15 °C and 0.005 rad/s, i.e., 0.0008 Hz) for the binders was considered unsuitable for the mixtures. In fact, E^* is not directly measured at these temperatures and frequencies. Moreover, this combination is in the high-temperature/low-frequency area in the phase angle master curve, where the aggregate skeleton has a great influence on the rheological behavior. For these reasons, mixture-based *G-R* parameter was formulated according to Equation (5):

$$G - R = |E^*|\frac{(\cos\phi)^2}{\sin\phi} \quad (5)$$

where $|E^*|$ and ϕ are the norm and phase angle of the complex modulus measured at 20 °C and 5 Hz.

5. Results and Discussion

5.1. Rheological Behavior

The Black (Figure 6) and Cole-Cole (Figure 7) diagrams show and validate the thermo-rheologically simple behavior of the mixtures. From the Black diagrams, the variability of $|E^*|$ and ϕ can be noted. In particular, $|E^*|$ approximately ranged between 150 MPa and 25'500 MPa for the mixtures with VB, between 50 MPa and 25'000 MPa for the mixtures with SR, independently from the mixing temperature (140 °C or 170 °C). The phase angle ϕ ranged between 4° and 35° for mixtures with VB, between 4° and 48° for mixtures with SR. In general, the maximum value of $|E^*|$ (measured at 5 °C and 20 Hz) was comparable among the different mixtures and ranged between 18'300 MPa (mix 00RAP_SR_170, specimen #2) and 25'500 MPa (50RAP_VB_170 mix, specimen #1). Conversely, some differences can be observed in the phase angle and in the stiffness modulus at high test temperatures (35 °C and 50 °C). In particular, the presence of RAP led to a global decrease in ϕ at all temperatures and an increase in the $|E^*|$ at high temperatures. These differences are more remarkable in correspondence of the use of the straight-run bitumen and of the higher mixing temperature.

The graphs in Figures 8 and 9 show that even if the data are slightly scattered and a certain sample-to-sample variability can be observed, the E^* values measured at different frequencies and temperatures are aligned on a single smooth curve for each specimen. Therefore, the thermo-rheological simplicity is confirmed for all the tested mixtures, and thus the TTSP can be applied.

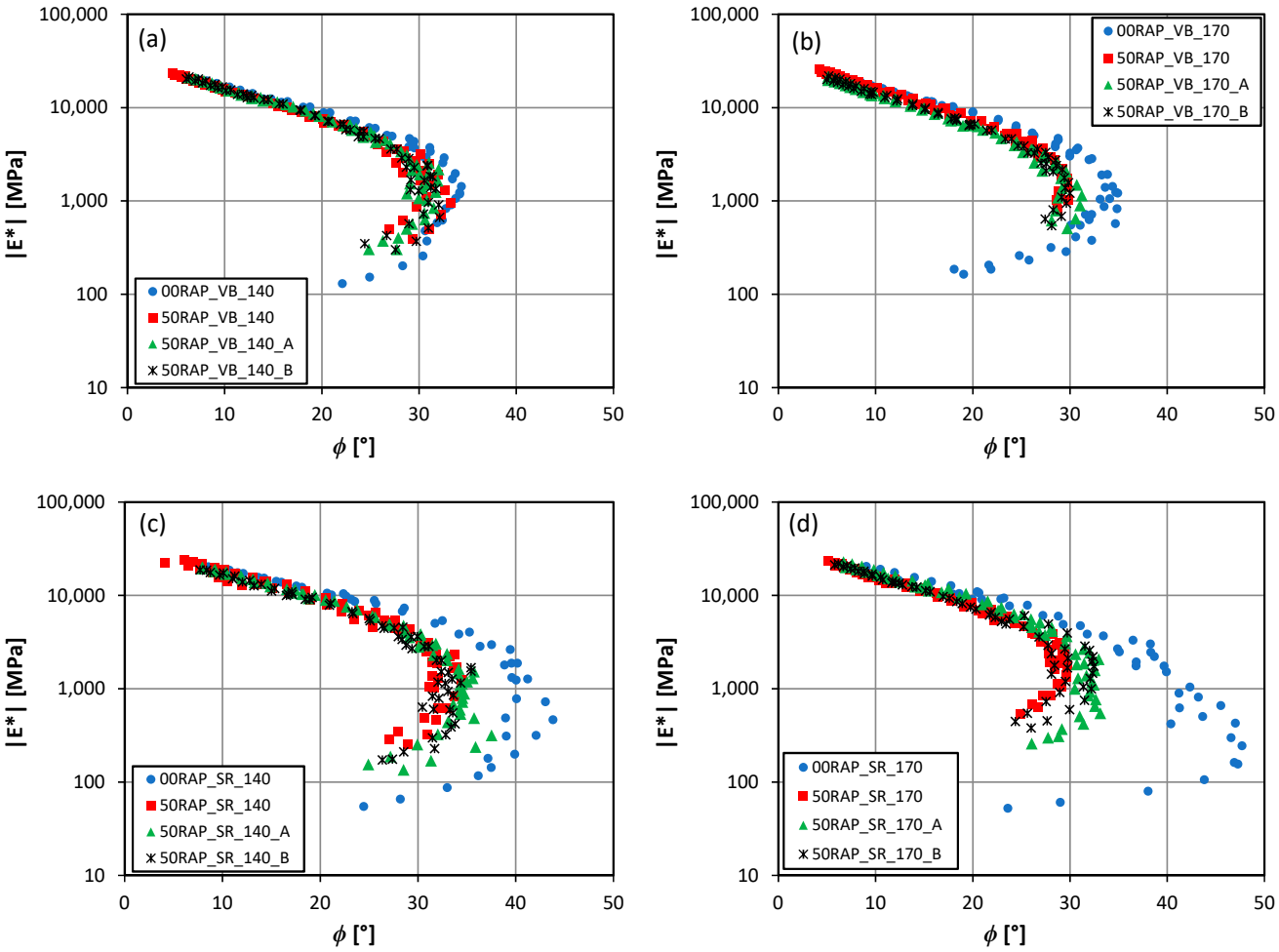


Figure 8. Black diagrams: (**a**) visbreaker bitumen, 140 °C; (**b**) visbreaker bitumen, 170 °C; (**c**) straight-run bitumen, 140 °C; (**d**) straight-run bitumen, 170 °C.

The Black and Cole–Cole diagrams were also used for the first estimation of glassy and static asymptotes to be fit into the 2S2P1D model before the error minimization (Equation (4). In particular, the Black diagram allowed estimating the glassy modulus (E_∞) as the intersection of the prolongation of the top data with the y-axis. For all the tested samples, the glassy modulus ranged between 42 and 48 GPa. Using the Cole–Cole diagram, the static modulus (E_0) can be obtained as the intersection of the data with the x-axis (enlarged plots on the upper right side of the graphs in Figure 9). The estimation of E_0 changed a lot depending on the RAP content, the rejuvenator A or B, the type of virgin bitumen, and the mixing temperature. In particular, E_0 ranged between 35 MPa and 195 MPa, respectively, obtained for a specimen of the mix 00RAP_SR_140 and a specimen on the mix 50RAP_VB_170.

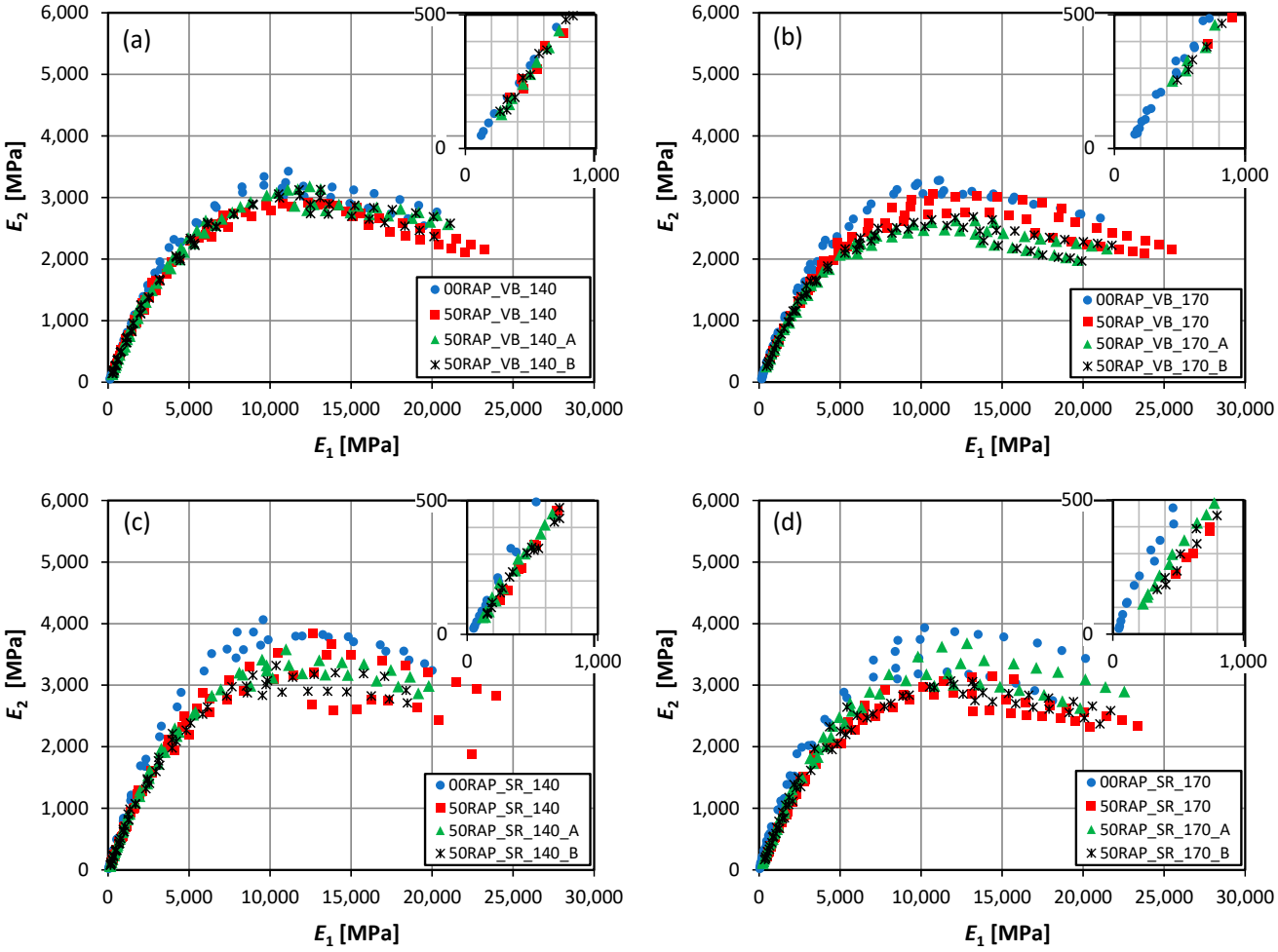


Figure 9. Cole–Cole diagrams: (**a**) visbreaker bitumen, 140 °C; (**b**) visbreaker bitumen, 170 °C; (**c**) straight-run bitumen, 140 °C; (**d**) straight-run bitumen, 170 °C.

5.2. Application of the 2S2P1D Model

After assessing the applicability of the TTSP, the temperature-shift factors were applied to the complex modulus data to obtain $|E^*|$ and ϕ master curves at the reference temperature of 20 °C. The shift factors were estimated through the closed-form shifting (CFS) algorithm, which provides the minimization of the area between two successive isothermal curves [55].

Figure 10 shows the superimposition of $|E^*|$ master curves on the 2S2P1D model. For all the tested mixtures, the addition of RAP induced an evident shift of the master curve upwards, while the rejuvenators were able to take it back downwards at an intermediate position between the two boundary cases. The mixing temperature had an amplifying effect on these shifts. In fact, the higher the mixing temperature, the more the distance between the 00RAP and the 50RAP mixtures. Moreover, it can be noted that for both mixing temperatures, the use of straight-run bitumen led to an increase in the gap between the mixes made with and without RAP. No remarkable differences were visible between the two rejuvenators, except when straight-run bitumen and a temperature of 170 °C were used: in this case, rejuvenator A seemed to be more efficient than rejuvenator B.

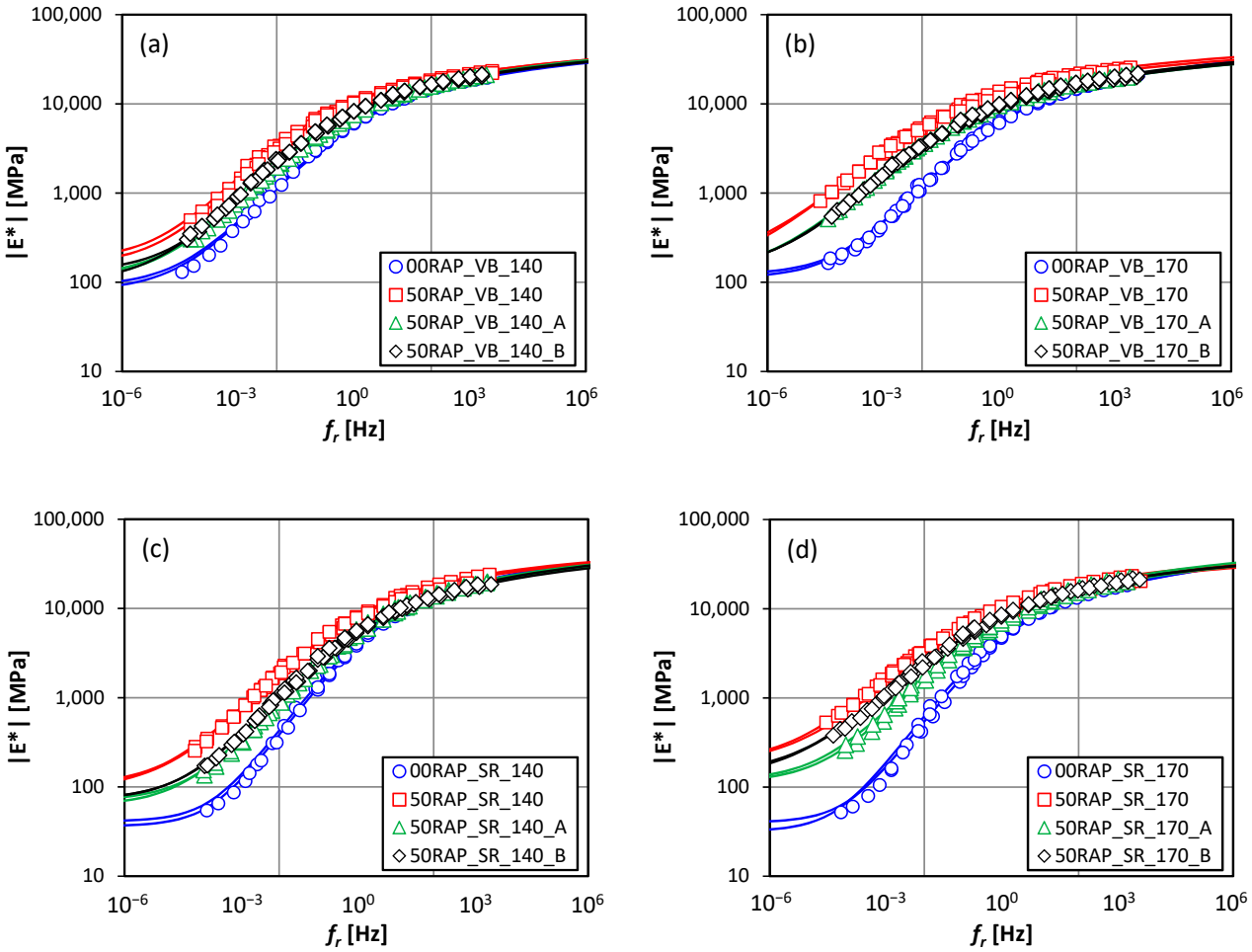


Figure 10. Master curves of $|E^*|$: (**a**) visbreaker bitumen, 140 °C; (**b**) visbreaker bitumen, 170 °C; (**c**) straight-run bitumen, 140 °C; (**d**) straight-run bitumen, 170 °C.

The phase angle is an indicator of the viscous properties of the material evaluated. For a purely elastic material, $\phi = 0°$ and for purely viscous material, $\phi = 90°$. Therefore, evaluating the variation of this parameter as a function of RAP content, rejuvenator type and mixing temperature is fundamental to achieve a better comprehension of the evolution of the rheological characteristics of HRMA compared to those of a mix made with only virgin materials. Coherently with the results obtained for the $|E^*|$ master curves, from Figure 11 it can be observed that the use of 50% RAP led to a downward flattening and a leftward shift of the phase angles. Therefore, the 50RAP was found to be less viscous than the 00RAP. As viscosity enhances the capacity of the material to dissipate the stress energy and relax, a decrease in viscosity entails an increase in brittleness. The addition of the rejuvenators almost did not alter the shape and position of the phase angle master curves (only a slight increase of the ϕ values can be noted), implying that the effect of the additive mainly consisted in the reduction of the stiffness of HRMA, without fully restoring the rheological characteristics. Moreover, the differences in the shape and position of the ϕ master curves between the mixtures made with and without RAP seemed to increase with mixing temperature and when straight-run bitumen was used.

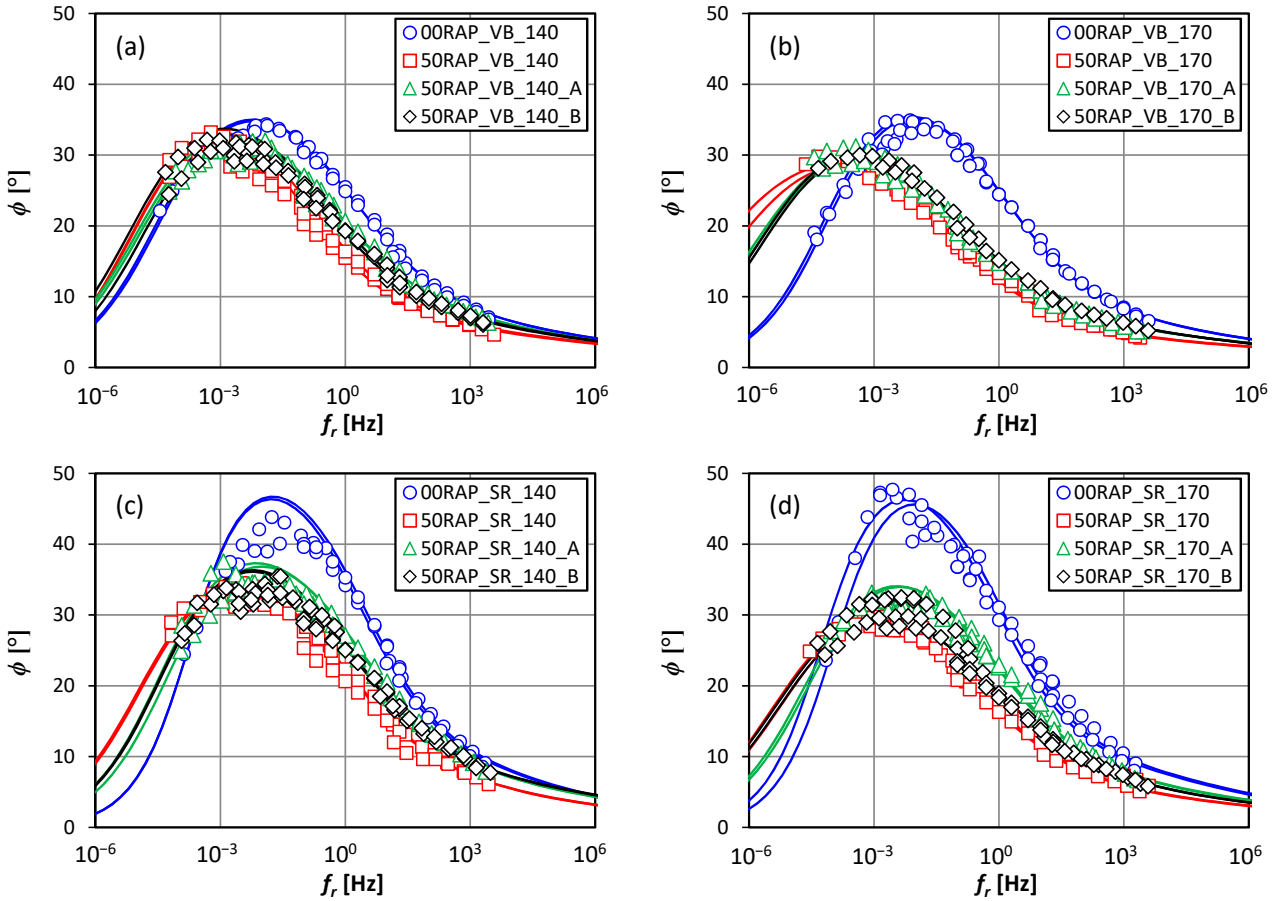


Figure 11. Master curves of ϕ: (**a**) visbreaker bitumen, 140 °C; (**b**) visbreaker bitumen, 170 °C; (**c**) straight-run bitumen, 140 °C; (**d**) straight-run bitumen, 170 °C.

For a clearer comprehension of the influence of the bitumen production process and the mixing temperature on the $|E^*|$ and ϕ master curves, these factors are represented by plotting the same mixtures (00RAP, 50RAP, 50RAP + A and 50RAP + B) in separated graphs (Figures 12 and 13). In this way, with the equivalent mix composition, the differences in the complex modulus can be directly related to the mixing temperature and to the type of virgin bitumen used. In Figures 12a and 13a, it can be noted that the mixtures with visbreaker bitumen had almost identical master curves of $|E^*|$ and ϕ for both 140 °C and 170 °C mixing temperatures. The master curves of the HMA with straight-run bitumen showed noticeably lower stiffness and higher phase angle in comparison with the HMA with VB, especially at low frequencies/high temperatures. Moreover, for the SR bitumen, a slight increase in $|E^*|$ and a decrease in ϕ were observed when increasing the mixing temperature from 140 °C to 170 °C (even if for one of the 00RAP_SR_170 specimens the peak of the phase angle was higher). In the result interpretation, it is important to consider that the rheological behavior of the unaged VB and SR bitumens, tested with the dynamic shear rheometer in a previous research [45], was almost identical. Therefore, it can be deduced that the VB bitumen had already suffered severe short-term aging at the mixing temperature of 140 °C and a temperature increase did not determine a further worsening of the performance. On the other hand, the SR bitumen showed a significantly lower aging sensitivity in the short term, but the increase in mixing temperature to 170 °C was slightly more detrimental.

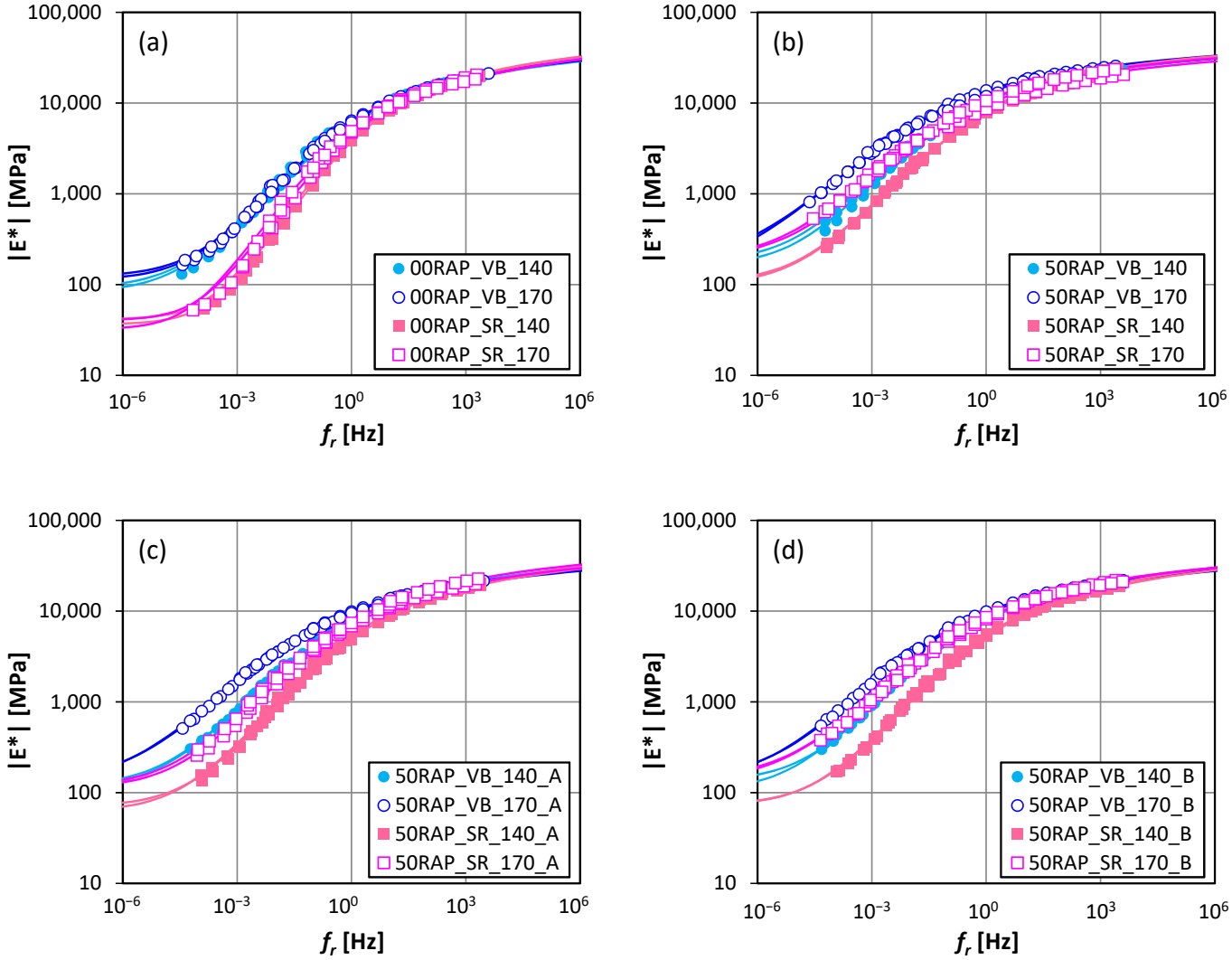


Figure 12. Master curves of $|E^*|$: (**a**) 00RAP; (**b**) 50RAP; (**c**) 50RAP + A; (**d**) 50RAP + B.

Figures 12b and 13b show the $|E^*|$ and ϕ master curves of the mixtures including 50% RAP. It can be observed that—comparing the behavior of the mixtures with different virgin binders—the SR bitumen allowed obtaining lower $|E^*|$ and higher ϕ with respect to VB. However, the difference between the performances of the mixtures with SR and with VB was lower in HRMA with 50RAP than for HMA without RAP. The most interesting result is related to the effect of the mixing temperature on the rheological properties of the specimens: mix stiffness grew significantly and the phase angle noticeably decreased when raising the mixing temperature from 140 °C to 170 °C, both in the case of VB and SR bitumens. These results are related to the different short-term aging of the virgin bitumen only in a small part, as the influence of the mixing temperature was low for the mixtures with no RAP. As such, they could be explained by hypothesizing that at the mixing temperature of 170 °C a higher percentage of RAP bitumen melted and blended with the virgin one. The higher aged/virgin bitumen ratio of the "active" binder in the mix can be considered responsible for the stiffer and less viscous behavior of the mixtures produced at 170 °C.

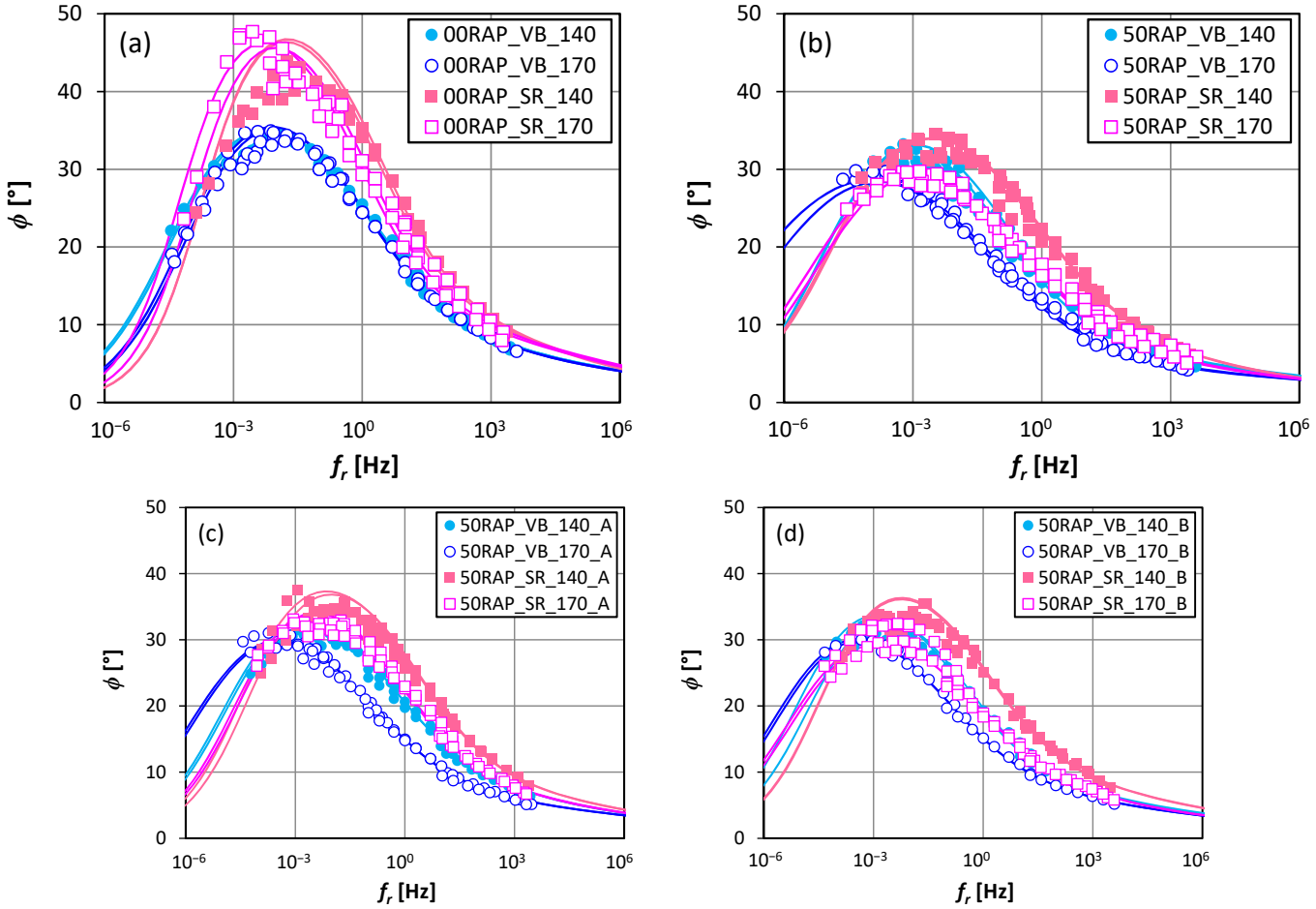


Figure 13. Master curves of ϕ: (**a**) 00RAP; (**b**) 50RAP; (**c**) 50RAP + A; (**d**) 50RAP + B.

This assumption was confirmed by the graphs in Figure 12c,d and Figure 13c,d, plotting the $|E^*|$ and ϕ master curves of the mixtures including the rejuvenator. Indeed, these mixtures containing 50% RAP also showed a significant sensitivity to the operational temperature, providing higher stiffness and lower phase angle values for the mixing temperature of 170 °C, regardless of the virgin bitumen and rejuvenator type. In general, for the mixtures including 50% RAP (with or without rejuvenator), the master curves of the specimens made at 170 °C with the SR bitumen were very close to those of the specimens made at 140 °C with the VB bitumen.

5.3. Analysis of Rheological Parameters

2S2P1D model parameters (E_∞, E_0, h, k, δ, β, τ_0) and Glover-Rowe (G-R) parameters are shown in Figure 14.

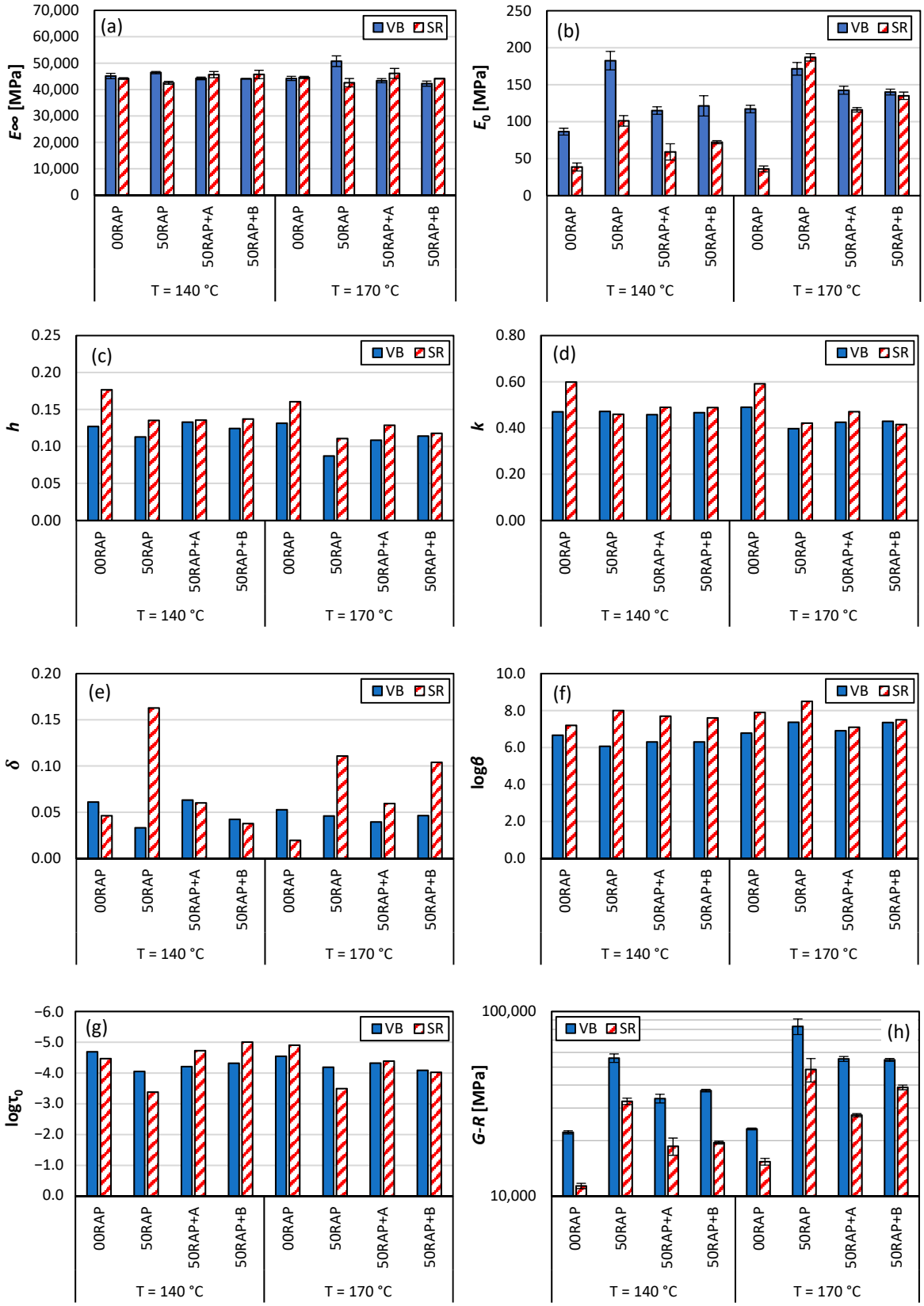


Figure 14. Rheological parameters changing the mixture composition, the temperature and the type of the virgin binder used: (**a**) $E\infty$ glassy modulus; (**b**) E_0 static modulus; (**c**) h; (**d**) k; (**e**) δ; (**f**) logβ linear dashpot parameter; (**g**) logτ_0 characteristic time; (**h**) *G-R* Glower-Rowe.

Figure 14a,b respectively report the values of the glassy and viscous asymptotes of the E^* master curves using the 2S2P1D model. From the graphs, it can be noted that comparable values of E_∞ were obtained for the different mixtures (approximately around 45 GPa) except for the mix 50RAP_VB_170 which provided an E_∞ value of about 50 GPa. Differently, E_0 was sensitive to the presence of RAP/rejuvenator, the virgin bitumen type and the production temperature. In particular, E_0 values were higher in the case of higher mixing temperature, presence of 50% RAP in the mix and use of visbreaker bitumen. The presence of the rejuvenator contributed to the decrease of the viscous asymptote value but did not reach the performance of the mixes without RAP. It must be highlighted that the experimental data did not allow reaching the asymptotic trend of the model at low reduced frequencies, as the "softer" mix condition investigated was T = 50 °C, f = 0.1 Hz, so the values of E_0 represent a mere estimation. However, a significant result and a good correlation with the physical behavior of the different materials were obtained. In addition, specimen-to-specimen variability was very low in the case of both E_∞ and E_0 approximation.

Figure 14c,d show the values of h and k, representing the parabolic elements in the 2S2P1D model. In particular, for both parameters, using RAP and increasing mixing temperature led to lower h and k, while there was a very slight increase in these parameters by adding rejuvenators. The decrease of h and k values corresponded to the flattening of the curves in the Cole-Cole plot, denoting more inhibited viscous characteristics. No remarkable changes were noted between the two rejuvenators. Moreover, it can be observed that the mentioned differences were less relevant between specimens with visbreaker bitumen.

The values of δ (Figure 14e) showed a low variability among the tested mixtures when VB bitumen was used, ranging between 0.03 and 0.06. Differently, for the mixtures including SR bitumen, the parameter δ slightly increased when 50% RAP was included in the mix (up to 0.165) and decreased with the addition of the rejuvenators.

The values of β (Figure 14f) did not provide a significant trend as a function of RAP/rejuvenator presence, bitumen type and mixing temperature. Moreover, it has to be remarked that the parameter β was very high ($> 10^6$) and a further increase did not entail either change in the master curve shape or reduction of the error between calculated and measured E^*. This suggests that the viscous damper in the 2S2P1D model was so viscous that it could be equated to a rigid element and the analogical model could be reduced to a Huet–Sayegh (2S2P) [56].

Figure 14g shows that the use of RAP produced higher values of $\log\tau_0$ (lower in absolute value), denoting lower relaxation ability. The addition of rejuvenator led to a decrease (increase in absolute value) of the characteristic time, reaching values even below those of the mixtures made without RAP. This trend was noted for both mixing temperatures, but it was more remarkable in presence of the SR bitumen. No differences between the two rejuvenators were found.

Finally, Figure 14h shows that the *G-R* parameter ranged between 11'000 MPa and 90'000 MPa for the different mixtures, resulting in about 5 orders higher than for binders. The addition of 50% RAP to the mixture led to a noticeable increase of the *G-R* parameter while the addition of rejuvenators allowed lowering the *G-R*, but never reaching those of the mixture produced without RAP. Moreover, for all the investigated mixtures, the straight-run bitumen showed lower *G-R* values with respect to the visbreaker. The increase of the mixing temperature induced a significant increase of the *G-R* for all the mixtures except for those produced without RAP using the visbreaker bitumen, confirming what was observed for the master curves (Figures 10 and 11). Rejuvenator A seemed to be slightly more effective in reducing the *G-R* values for both the bitumen types and both mix production temperatures.

6. Conclusions

The present study aimed to evaluate the influence of mixing temperature and of the origin of the neat bitumen (visbreaker vs straight-run) on the rheological characteristics of an HRMA made using 50% of RAP. The measured complex modulus data were interpolated using the 2S2P1D model in order to observe the variation of the master curves shape and the rheological parameters. Based on the results presented in this paper, the following conclusions can be drawn:

- All the tested specimens showed a thermo-rheologically simple behavior, which could be well simulated by the 2S2P1D model. In particular, the Huet–Sayegh model would have been as accurate as the 2S2P1D because the logβ was always higher than 6, so the dashpot was actually an infinitely rigid element.
- The addition of RAP in the mixtures induced a pronounced upwards shift on the $|E^*|$ master curve. The rejuvenators were able to take it back downwards in an intermediate position between the master curves of the 00RAP and 50RAP mixes. No remarkable differences were visible between the two rejuvenators, except when straight-run bitumen and a mixing temperature of 170°C were used, a scenario in which rejuvenator A seemed to be more efficient.
- When including 50% RAP, the phase angles master curves tended to flatten and shift leftward. The addition of rejuvenators left the shape and position of the ϕ master curves almost unchanged, meaning that the effect of the additive was mainly to reduce the stiffness of HRMA, without fully restoring its rheological characteristics.
- The master curves of the mixtures without RAP produced with VB or SR bitumen indicated a higher sensitivity to the short-term aging for VB than for SR. Also, mixtures with VB including RAP/rejuvenators showed higher stiffness and lower phase angle when compared to the analogous mixtures with SR.
- The mixing temperature poorly influenced the rheological behavior of the mixtures without RAP, while it had a high impact on the mixtures including 50% RAP. This denotes that the mixing temperature increase did not determine a significant worsening of the virgin bitumen short-term aging, but probably entailed the mobilization of a higher percentage of aged bitumen from the RAP.
- The parameters E_∞, δ and β of the 2S2P1D model showed a low significance in representing the evolution of the mix rheological properties as a function of the different variables (presence of RAP/rejuvenator, origin of virgin bitumen and mixing temperature). The parameters E_0, h, k and τ_0 had a more relevant variation when RAP and, afterwards, rejuvenators were used, but they varied less significantly as a function of bitumen type and mixing temperature. The Glover–Rowe parameter, in the formulation proposed by Ogbo et al. [54] for bituminous mixtures, proved to be effective in summarizing the changes in the complex modulus with the different factors.

In conclusion, the adoption of high mixing temperatures and the use of visbreaker bitumens proved to amplify the differences between 00RAP and 50RAP mixtures and limit the effectiveness of the rejuvenators. Therefore, using a reasonably low mixing temperature (enough to make the bitumen workable) and a good straight-run virgin bitumen plays a fundamental role in the production of an HRMA that achieves optimal rheological/mechanical characteristics and adequate durability, without being susceptible to cracking issues. Future investigations will aim at validating this conclusion through the characterization of the HRMA mixtures produced in the plant at different temperatures, with particular focus on the measurement of the pollutant emissions during production and the rheological, mechanical, and fatigue behavior of the mixtures.

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