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Research Article

Characterization of Cold Bituminous Mastics Prepared with Different Active Fillers at High and Intermediate Temperature

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Cold recycling of asphalt mixes is becoming a usual pavement rehabilitation technique. The use of active fillers in conjunction with bituminous emulsion changes the properties of the mixes, mainly on the mastic characteristics. The use of active filler can make the mastic more brittle, but limited data are available on this aspect. In this study, emulsion-active filler mastics were prepared with two different mixing methods. Two different active fillers, ordinary Portland cement and fly ash, were used at four different contents with a single emulsion and an inert filler. Once cured, the mastics were tested with a dynamic shear rheometer (DSR) to get the viscoelastic properties at high temperature and with the double-edge-notched tension (DENT) test to look at the cracking potential at intermediate temperature. The results show that the order that the materials are incorporated during the mixing of the mastics does have a significant effect on the properties. Also, in terms of stiffness, the optimum amount of active filler is different for the cement than for the fly ash. For the cracking resistance, the results have shown that the addition of both active fillers does have an impact on the cracking resistance, but in the amount tested, no brittle fracture was observed. The results have shown that the type and quantity of active filler are important since it changes the fracture performance and the viscoelastic properties of bituminous mastic.

1. Introduction

Cold in-place recycling (CIR) and full-depth reclamation (FDR) are pavement rehabilitation techniques used to correct the defects of bituminous pavement. These two techniques are recognized in road construction industries for their environmental, economic, and structural benefits [1]. CIR and FDR materials are composed of reclaimed asphalt pavement (RAP) in different quantities and often treated with a binding agent like cement, bituminous emulsion, or foamed asphalt. Like for hot mix asphalt, it is the mastic that gives the cohesion to the mix. Therefore, it is important to study the properties of the mastic of those mixes.

Filler has a major impact on cold mix asphalt. Usually, two types of filler can be used for the mastic with emulsion, properly called inert and active filler. By definition, an inert filler does not have a chemical reaction with water, and it generally comes from quarries like limestone and basalt [2]. To increase the structural capacity of the cold mix and to accelerate the curing process of the bitumen emulsion, active filler, like ordinary Portland cement (OPC), can be used [3]. The active filler reacts with the water in the cold mix (the added water and the water phase of the emulsion). Therefore, the hydration process of the OPC is affected by the presence of the asphalt droplet in the emulsion [4]. When a high quantity of cement is in contact with a little quantity of emulsion, the bitumen droplets will coat the cement particles and slow the curing process. However, in emulsion asphalt mixes, the right proportion of OPC speeds up the breaking of the emulsion. The hydration of the active filler depends on the curing condition of the mix (relative humidity and temperature). Using OPC improves both resistance to cracking and permanent deformation [5]. Active filler content and properties have a great influence on the performance of the cold mix asphalt [3, 6, 7].

Several studies have shown that some wasted material or by-product like hydrated lime (HL), ground granulated blast furnace slag (GGBS), lime kiln dust, fly ash (FA), silica fume (SF), and treated biomass fly ash (TBFA) offers good properties in cold mix asphalt, and it can be used as a substitute of OPC [3, 6–9]. The use of these by-products in cold mix asphalt significantly decreased the susceptibility to permanent deformation and water damage by producing a higher stiffness modulus [10] while lowering greenhouse gas (GHG) emissions and the price. For example, the TBFA has a high absorptive ability and cementitious properties, which improves the indirect tensile stiffness modulus by a factor of 5, with only 5% added to the mix [7, 11].

In cold mix asphalt, the percent passing 0.075 mm sieve by mass must be between 3 and 8% to respect the Indian standard [7, 12]. The addition of active or inert filler must be in this range. Cold mix asphalt (CMA) usually contains up to 2% of the total mass of the dry aggregates of cement and has about the same total bitumen content as HMA. Bitumen-stabilized materials (BSMs) and cement-bitumen-treated materials (CBTMs) have the same bitumen content but not the cement content. The cement content of the BSM is lower in order to have the viscoelastic properties of the asphalt and not the rigidity of concrete [13]. To better understand the behavior between active filler and bitumen emulsion, some studies did exceed the standard cement and bitumen content [11, 14, 15]. To represent a mastic with the properties of CMA, the active and total filler content should respect the Indian roads passing sieves specification [12]. It is important to look at not only the amount of filler, but also the ratio of active filler, like cement, and bitumen from the emulsions. Ratios from 0.2 up to 2.0 of cement/bitumen have been tested in different studies [16–18].

To characterize the stiffness of cold mix asphalt, indirect tensile strength modulus (ITSM) has been used to determine the viscoelastic properties of the mix in several studies [7, 13, 14]. But to characterize the viscous and elastic behavior of bituminous mastic, the dynamic shear rheometer (DSR) can be used. AASHTO gives the standard test method to define the high temperature of the PG grade by testing the material with at 10 rad/second [19, 20], 2016.

To evaluate the cracking performance of the asphalt binder, the double-edge-notched tension (DENT) has been developed by the Ministry of Transportation of Ontario, Canada. This test is now implemented for the acceptance of modified binders in different places. This test covers the determination of a bitumen resistance to the ductile failure using double-edge-notched tension equipment. It determines the essential and plastic works of failure by measuring the strength and toughness of the material [21]. The DENT proposes to use 3 different lengths of the notch and to break them in tension at a fixed displacement rate. With these values, we can obtain the critical crack tip opening displacement (CTOD), which gives us a measure of strain tolerance of the material in the ductile zone. The results of DENT can be used to evaluate the asphalt field performance such as premature and excessive cracking and low-temperature cracking.

The objectives of this research project are to elaborate a method to prepare and characterize cold mix bituminous mastic, and to evaluate the effect of different active filler types and concentration on the properties of the mastic. To achieve this goal, a laboratory study was performed. The methodology and the materials used are explained in the next sections.

2. Materials and Methods

To meet the objectives, eight different mastics were prepared with a single bitumen emulsion source, an inert filler, and two active fillers in different proportions. Two mastic preparation methods were also used before testing their properties. The selected tests were the DENT and the complex shear modulus.

3. Materials

The inert filler used in this study was baghouse fine (basalt) taken at the supreme asphalt plant in Navi Mumbai. The first type of active filler is a fly ash (FA) "Class F" generated by the combustion of pulverized coals in the thermal power plants. The two major chemical components in it are silica (SiO_2) and alumina (Al_2O_3), and it contains less than 10% of calcium oxide (CaO). The particles are spherical with a smooth surface and have pozzolanic properties [23]. The other active filler is an ordinary Portland cement (OPC).

One type of bitumen emulsion was used in this study, a slow-setting cationic emulsion called SS-1 from Hincol India [22]. Cationic slow-setting bitumen emulsions are the most commonly used emulsion for cold mix asphalt. Table 1 shows the properties of the active fillers, and Table 2 shows the properties of the bitumen emulsion. Figure 1 shows the particle size distribution of the fly ash used in this study and of a usual OPC.

It is important to note that the inert filler has been sieved through a 75 μm sieve to eliminate unwanted bigger particles.

3.1. Mastic Preparation Methods. Since the impact of the amount of active filler is bitumen emulsion, mastic is not well known, and different ratios of active/passive fillers were tested. However, because the interaction between each filler type and the emulsion is expected to differ, the methodology used to mix the filler with the emulsion is expected to also have an effect. Here are the two methods used.

Method 1: For the first method, the active and inert fillers were manually mixed together directly on a silicone mat to obtain a homogeneous mix. After 2 minutes of mixing, the bitumen emulsion was added and the mastic was mixed with a glass rod for another 3 minutes.

Method 2: For the second method, the inert filler was first manually mixed with the emulsion and then the active filler was gradually added while mixing with the glass rod.

After mixing, all the mastic on the silicon mat was collected to create a mastic ball. The mastic was spread with a clean glass rod by rolling it on the ball. The glass rod was rolled on the mastic ball until spread rates between 2.00 kg/m^2 and

Table 1: Properties of the active filers [23].

Chemical properties		
Chemical constituents	Fly ash (%)	Ordinary Portland cement (%)
Al_2O_3	26.82	4.4
BaO	0.07	0
CaO	1.13	63.1
Fe_2O_3	5.49	2.7
K_2O	0.94	0
MgO	0.96	2.1
MnO	0.03	0
P_2O_5	0.25	0
SiO_2	61.32	20.3
SO_3	0.08	3.4
SrO	0.06	0
TiO_2	1.65	0
LOI*	1.23	4
LOI*: Lost in oven		
Physical properties		
Specific gravity (ASTM C188)	2.15	3.15
Retaining on sieve 45 μm (%) (ASTM C430)	55	5
Blain-specific surface area (cm^2/g) (ASTM C204)	224	401

Table 2: Properties of the bitumen emulsion [22].

Hincol emulsion SS-1	
Residue on 600 μm IS sieve (% max)	0.05
Viscosity Saybolt Furol 25°C (sec)	20–100
Setting time (hrs)	Less than 48
Binder residue by evaporation (% min)	50
Coagulation at low temperature	Yes

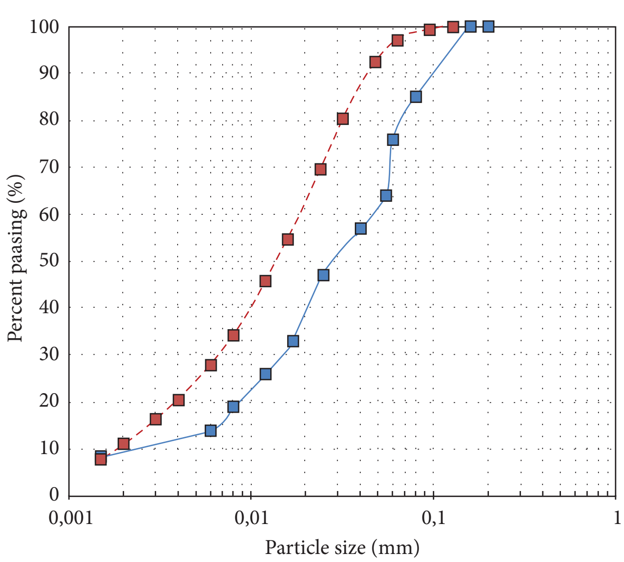


Figure 1: Particle size distribution of fly ash [23, 24].

3.00 kg/m^2 were obtained. After recording the spread rate, the samples were put on a flat surface in a room at 25°C for a first 24 hours curing and then in the oven at 60°C for 48 hours. The samples were taken out of the oven and then tested.

From the literature, the best proportion of emulsion in mastic is around 50% of the total weight of the mix [12]. To visually evaluate the workability of the material, different proportion emulsions and inert filler have been made. An evaluation was made based on the spreadability of the material and excess water content. Figure 2 shows three (3) different filler concentrations. To have a better hydration of the active filler and ease the manipulations, a proportion of 60% emulsion was selected and kept constant for all the tests. Table 3 shows the proportion used in the present study. All the mastics respect the Indian standards of active and inert filler content in the cold asphalt mixes [12, 13].

Based on the proportions shown in Table 3, the following acronyms are used for each filler: FA for fly ash, OPC for the cement, and BA for the inert filler. So, for example, the 15–85 mastic with fly ash is labelled 15%BA-85%FA.

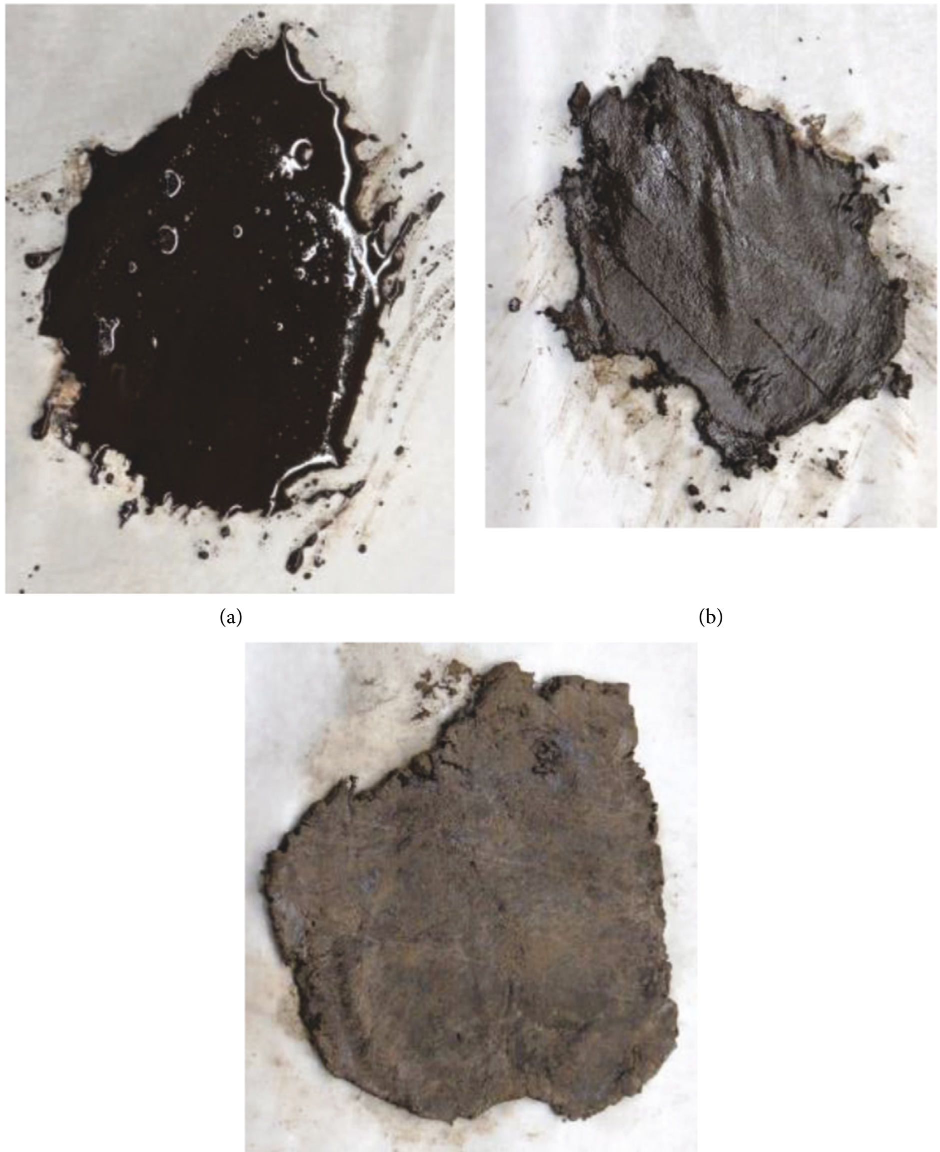
3.2. Testing Protocol. As previously mentioned, two tests were used to evaluate the mastic properties. First, the complex shear modulus (G∗) was used with the dynamic shear rheometer (DSR) at different temperatures and one frequency (1.59 Hz). G∗ allows evaluating the impact of the filler on the stiffness of the mastic. The second test is the DENT that was selected based on its capacity to measure the ductility of the mastic. The addition of active filler like OPC can make the mastic more brittle, which can result in a material that is prone to cracking. According to Wirtgen [26], a maximum of 1.5% of cement (by mass of total mix) can be used in cold recycling before brittle behavior is observed.

The total weight of the sample for DSR was 15 g, and the tested material was taken from the middle of this sample. The tested material was put on the bottom DSR plate and heated at 80°C for 1 minute. After that, the specimen was squeezed to its specific gap between the two DSR plates and put at 64°C for testing. For DSR, three repetitions for each mastic were done at 64, 70, 76, 82, and 88°C to evaluate the high-temperature stiffness.

The DENT consists in pulling on a notched (precracked) specimen with a constant strain rate, at the constant temperature, until failure. The test was done according to the Ontario (Canada) MTO LS299 methodology but with a slower speed of 25 mm/min. The standard speed of 50 mm/min has shown early breaking in the specimens. Two specimens were prepared for each mastic with dent sizes of 5, 10, and 15 mm.

After the oven curing of the mastics, the mastic was put in the DENT mold. It was observed that the mastic was too stiff and difficult to place properly in the mold. Because of this, the DENT molds with the mastic in it were put in the oven at 60°C for 4 more hours. Previous tests have shown that without this time in the oven, the sample was not useable. The samples were removed from the oven to cool down at room temperature for trimming with a hot knife. The DENT samples were then conditioning at 15°C in a water bath for 3 hours before the test.

For DENT, force-displacement curves of specimens with different ligament lengths (material left at the notch level)



(a) (b) (c)

Figure 2: Evaluation of the filler concentration. (a) 20% filler content. (b) 50% filler content. (c) 70% filler content.

Table 3: Mix proportions.

	Active—inert filler ratio by weight			
	15–85 (%)	30–70 (%)	45–55 (%)	60–40 (%)
Emulsion SS-1	60	60	60	60
Fly ash or OPC	6	12	18	24
Inert filler	34	28	22	16

are drawn and the total and essential work (W_e) at failure is calculated. With the essential work at failure, it is possible to calculate the critical crack tip opening (CTOD). CTOD and W_e are used to evaluate the ductile failure and the fatigue resistance of the tested material [25]. The essential work is basically a measurement of strength and toughness where CTOD is a measurement of strain tolerance. Figure 3 shows the flowchart of the experimental methodology of this study.

4. Results and Discussion

4.1. Dynamic Shear Rheometer. For the mixing method 1, the active and inert fillers were mixed together directly on the silicone mat to obtain a homogeneous mix. After 2 minutes of mixing, the bitumen emulsion was added and the mastic was mixed with a glass rod for another 3 minutes. Figures 4 and 5 show DSR results with ordinary Portland cement and fly ash, respectively, for mix method 1. The curves are plotted for an average of 3 sample results tested at 64°C, 72°C, 80°C, and 88°C, with a coefficient of variability below 10%.

It is interesting to note that the overall maximum stiffness observed for all the mixes produced with mixing method 1 is very similar, but it is achieved with 60% fly ash or with only 15% cement. For the fly ash mixes, higher

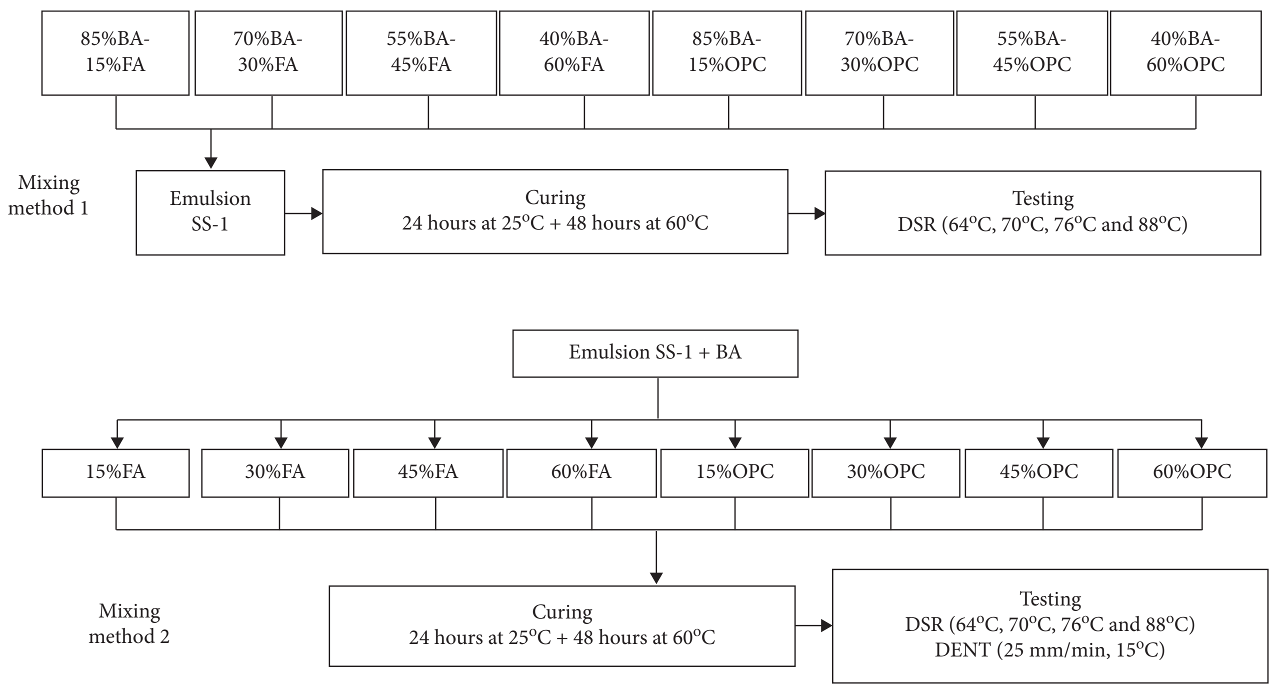


Figure 3: Research plan flowchart.

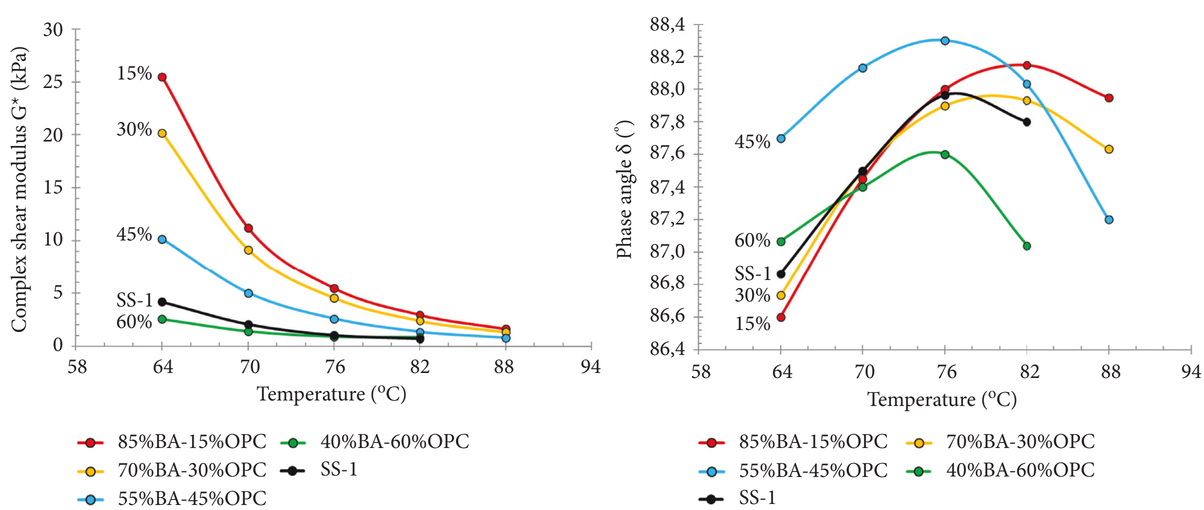


Figure 4: DSR results, shear modulus, and phase angle @ 1.59 Hz, of the residual bitumen from the emulsion (SS-1) and Portland cement mixes with mixing method 1.

content of FA means higher stiffness, but at least 30% of FA is required to have a noticeable difference from the pure emulsion (SS-1). However, for cement, the higher the portion of cement in the filler, the lower the stiffness, with the 60% OPC being similar to the emulsion alone (SS-1). The addition of OPC did not change the trend observed for the bitumen from the emulsion regarding the phase angle since an optimum value is observed at a given temperature. It is not the case for the FA mixes. However, for FA mixes, the highest stiffness (60% FA) is associated with the highest phase angle. So in this case, it seems that it is the inert filler that decreases the phase since the higher the content of the inert filler, the lower the phase angle. It is probable that there is not enough water available for the cement hydration in the mix, but the water is sufficient when fly ash is used. It would be interesting to redo those tests with fillers, inert and active, with different gradations and maximum size in order to understand the impact of the volumetric properties, like the surface area, on the viscoelastic properties of the mastics.

For the second mixing method, in which the inert filler was first mixed with the emulsion before adding the active filler, the DSR results are shown in Figures 6 and 7.

For all the mixes, the stiffness is higher when mixing the emulsion and the inert filler before adding the active filler. For the same amount of active filler, mixing method 2 offers higher stiffness. For both active fillers, a maximum stiffness

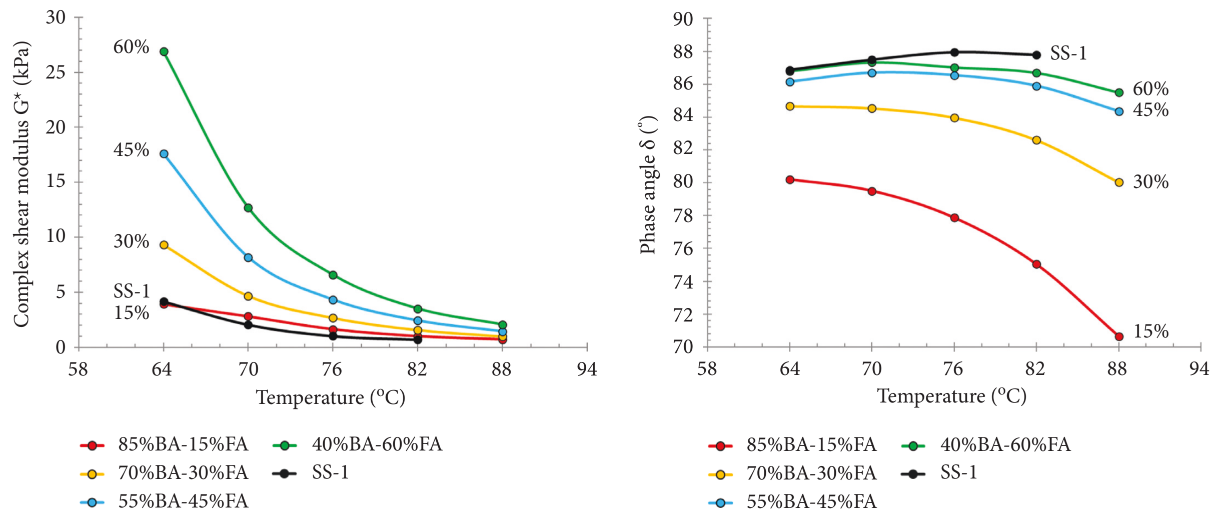


FIGURE 5: DSR results, shear modulus, and phase angle @ 1.59 Hz, of the residual bitumen from the emulsion (SS-1) and fly ash mixes with mixing method 1.

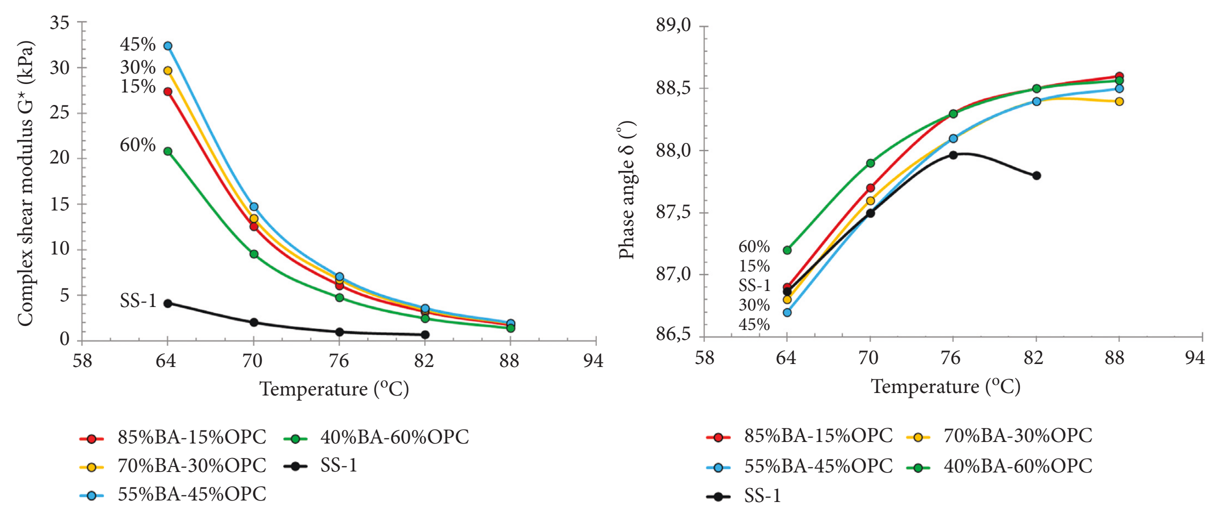


FIGURE 6: DSR results, shear modulus, and phase angle @ 1.59 Hz, of the residual bitumen from the emulsion (SS-1) and Portland cement mixes with mixing method 2.

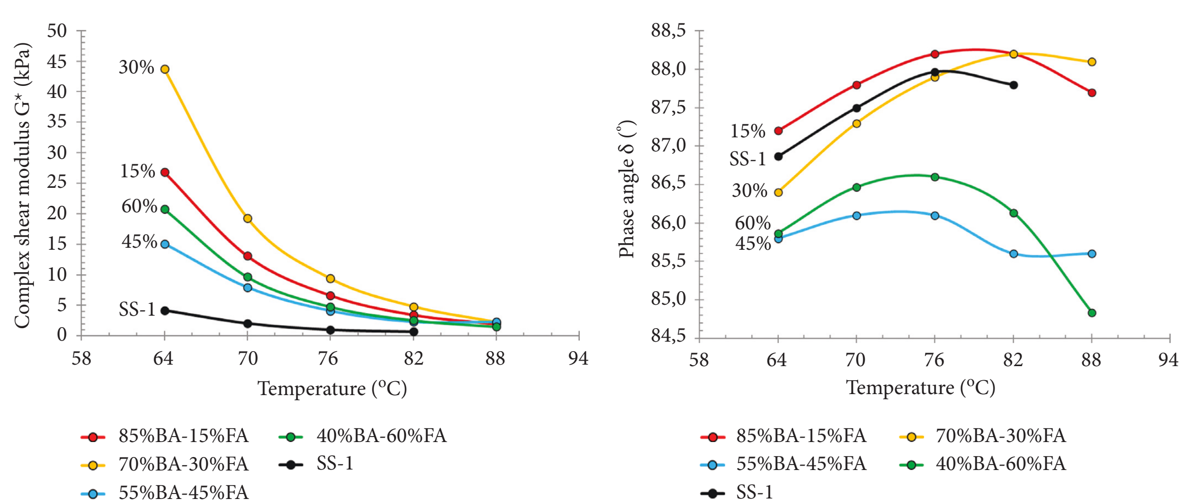


FIGURE 7: DSR results, shear modulus, and phase angle @ 1.59 Hz, of the residual bitumen from the emulsion (SS-1) and fly ash mixes with mixing method 2.

is reached on a certain quantity (45% of OPC and 30% for FA). Over this amount, stiffness gets lower with the increase of active filler. The impact of the active filler content is more noticeable with the fly ash. Unexpected results are obtained when looking at the phase angle. For OPC mixes, the biggest difference on phase angle is 0.50° (at 64°C) and 3.27° for FA mixes (at 88°C). It was hypothesized that there would be a relationship between the amount of active filler and the phase angle, but no clear trends are observed. More work is required to understand those results.

It is interesting to note that for most cases here, mixes with FA have higher stiffness than OPC mixes. This is unexpected since other studies suggest better performance with OPC than FA [27]. However, a study by Li et al. [28] has shown good fatigue performance of cold mixes with fly ash and it was shown in another study that the use of fly ash could result in higher fatigue life than mixes with cement [29]. The modulus ratio (mastic divided by emulsion) was calculated and plotted in Figure 8. The ratio is the average of the complex shear modulus ratio at all the tested temperature for a given active filler content. In Figure 8, it is clearly shown that an optimum active filler content is present when method 2 is used, but no optimum is seen for the active filler amount used in this study.

For method 1, the stiffness of all mixtures with fly ash increases with the concentration of active filler and the trend is the opposite for the mixture containing Portland cement. For both types of active filler, the slope of the curves presented is less pronounced at low active filler content and the slope increases from a dosage of 30% of active filler. Neither active filler content shows a plateau (optimum), but it seems to be above 60% for the mixtures with FA and below 15% for the mixtures with OPC. At 60% OPC, the modulus ratio is in average lower than the emulsion SS-1 alone.

For method 2, the fly ash mixes show the highest stiffness at 30% fly ash content and the OPC mix at 45%. The amount of fly ash used has a bigger impact on the stiffness of the mix than the amount of OPC. The modulus ratio of the OPC mix stays over 6 with a concentration of OPC of 15 to 40%. Over 40%, the stiffness goes down with the active filler concentration. For the fly ash mixes, the stiffness rises until 30% of active filler is used and then falls down with the concentration of fly ash used in mix. The slope of the change in the modulus according to the active filler concentration of both mixes is similar after reaching the optimum. The amount of water available, calculated as a ratio of water divided by the amount of active filler, cannot explain, by itself all the results observed. The volumetric ratio follows the same trend as the weight ratio. From those results, method 2 is better to evaluate the impact of the active filler on the stiffness of the emulsion base mastic.

4.2. Double-Edge-Notched Tension (DENT) Test. To obtain the resistance to the ductile failure, and the essential and the plastic works of failure, double-edge-notched tension (DENT) tests were made with the mixing method 2. Mix method 2 was chosen since it enabled the differentiation between mixes with the DSR. For each mix, three samples

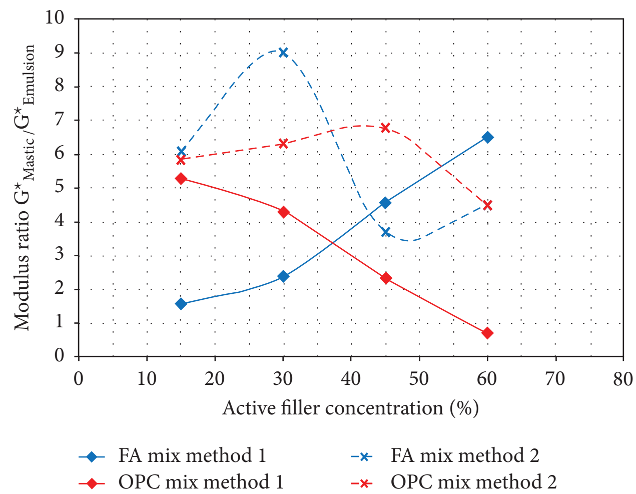


Figure 8: Average stiffness ratio for mixing methods 1 and 2 for OPC and FA mixes.

were mixed on silicone mold and cure was the same as for the DSR: 24 hours at 25°C, 24 hours at 60°C, and then 4 hours in the DENT mold at 60°C. The tests were made at 15°C with a loading speed of 25 mm/min. Previous tests have shown that 50 mm/min is too fast for the mastic, and the samples at 50 mm/min presented failure immediately after the test started. Figure 9 shows the load-displacement relationship for the fly ash mix for the samples of 10 mm. All the other results with fly ash show similar trends.

It is important to note that the DENT was developed to test bitumen only. The presence of filler, inert or active, does have a significant impact on the tensile resistance of the material. In fact, it is expected that the mastic gets stiffer with the increase of the filler content, but the maximum force at failure may be lower. In this study, the total amount of filler in all the mastics is constant, so it is only the amount of active filler that has an impact. However, even if the amount of filler is higher than the limit stated in the literature to observe brittleness [13] and also since the presence of filler may cause weak spots [30], no brittle behavior is observed. That being said, the load-displacement curves from DENT tests normally show higher area under the curve [31], more ductility, than what is observed in Figures 9 and 10. This could be due to the tests performed at a different loading rate than what is normally done or because of the presence of active filler.

As observed in Figure 10, the addition of fillers affects the performances of the mastic. For every notch length, the mix 55BA-45FA shows the best resistance and 85BA-15FA shows the worst results. The peak is increasing with the active filler concentration and going down after 45%. For every mix, the peak is increasing with the notch length. No specimen has shown fragile behavior due to the active filler. Figure 10 shows the load-displacement of the ordinary Portland cement mix for the sample of 10 mm in length. All the other results with ordinary Portland cement show similar trends.

As observed in Figure 10, DENT results of OPC mix behavior are different than fly ash mixes. The load peak is obtained at lower displacement raising the initial slope of the

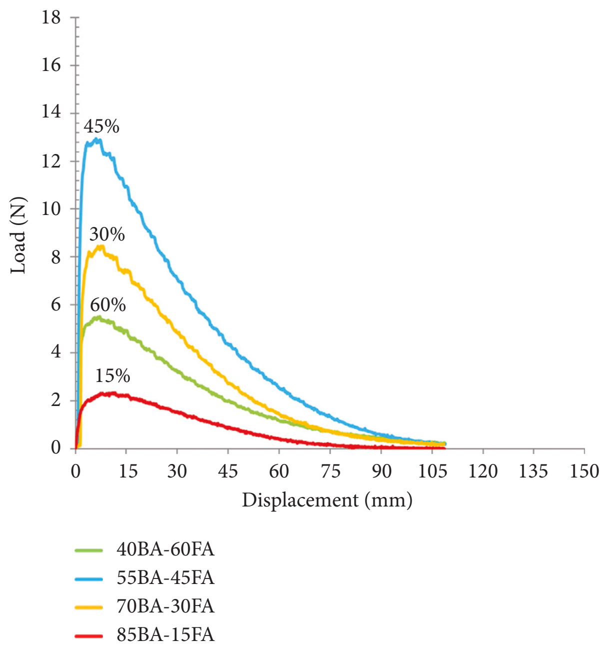


Figure 9: Load-displacement curve of DENT tests with doubled-edge notches of 10 mm for fly ash at 15°c and speed rate of 25 mm/min.

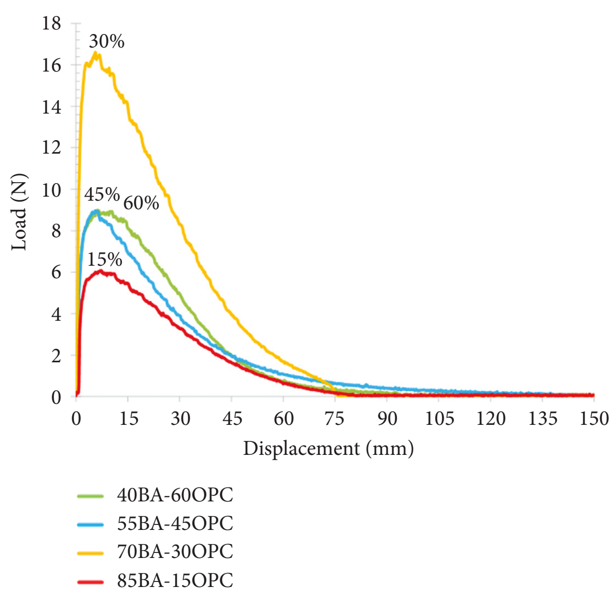


Figure 10: Load-displacement curve of DENT test with doubled-edge notches of 10 mm for OPC at 15°C and speed rate of 25 mm/min.

load-displacement curve. After the load peak, the mixes do not follow any trend. Almost all the slopes of the mixes are different. The results of fly ash and OPC mixes are summarized in Table 4.

As stated before, the DENT test was developed for bitumen only. According to the Ontario (Canada) standard OPSS 1101, the CTOD has to be greater than 6.0 to meet the criteria for a PG58-28 binder. For other bitumen, the requirements can

Table 4: Specific essential work and critical tip opening displacement for mixes with FA and OPC at 15°C and speed rate of 25 mm/min.

	FA				OPC			
	15	30	45	60	15	30	45	60
$\mathbf{W_e}$, specific essential work of fracture (**KJ/m²**)	0.82	1.92	3.67	2.61	1.59	4.48	3.80	1.42
$\boldsymbol{\delta}$, critical tip opening displacement CTOD (**mm**)	29.8	23.9	17.1	30.8	19.2	20.9	15.3	14.6

increase to 18.0 mm (PG58-40). The results in Table 4 show that all the mastic tested meets the highest requirement, meaning that the mastics have higher fatigue life than the bitumen alone. When looking at the results shown in Table 4, it can be observed that there is no good relationship between the essential work and the CTOD even if, in general, the CTOD decreases when the W_e increases. For the FA mixes, it can be observed that the specific essential work of fracture W_e is the highest for mix 55BA-45FA indicating that it has the highest strain tolerance to resist fatigue cracking. W_e is increasing linearly with the active filler concentration up to 45% and goes down after reaching its optimum. The CTOD provides a measure of strain tolerance in the ductile state under conditions of severe confinement. It can be used to determine a high correlation with cracking distress. In Table 4, it shows that 55BA-45FA has the lowest CTOD means that it has the lowest strain tolerance in the ductile state, which can be translated to the lowest fatigue life. 85BA-15FA and 40BA-60FA have about the same value and show the best performance for strain tolerance in the ductile state. Linearity can be observed with the fly ash content. The CTOD value decreases until 45% and raises with a two times higher slope.

As for the OPC mixes, the highest strain tolerance to resist fatigue cracking is obtained with mix 70BA-30OPC by observing the specific essential work of fracture W_e. W_e is increasing linearly with the OPC concentration up to 30% and goes down linearly after reaching its maximum. 40BA-60OPC mix has the lowest CTOD which means that it has the lowest strain tolerance in the ductile state. 85BA-15OPC and 70BA-30OPC have about the same value and show the best performance for strain tolerance in the ductile state. No linearity can be observed with the ordinary Portland cement content. Its value increases until 30% and decreases after it.

Fatigue resistance should never be analyzed by itself since the stiffness of the material controls the strain level in the material. So, this means that if a given mix has a lower fatigue or cracking resistance but it is stiff enough to remain below a given threshold where it will crack, it could mean a higher capacity to resist to cracking under loads (traffic). For example, if we calculate a ratio of CTOD and the maximum stiffness measured at 64°C, the mastics with 45% FA and 30% OPC are equivalent which means that they would probably have similar durability. Precise calculation at the pavement structure is required to have a solid conclusion here, but the mixes with 15% FA and 60% OPC would be the most durable mastics.

5. Conclusions

The use of cold recycled materials treated with bitumen and an active filler, like cement or fly ash, is usual in parts of the world. However, even if it is known that the addition of an active filler changes the ductility and the behavior of the mixes, there is little information available to quantify the amount of active filler that should be used and the effect of the different types of active fillers. The present study examined the properties of mastic made from bituminous emulsion and a combination of inert and active filler. The first part of the work is on the mastic preparation before testing the prepared mastic with a DSR to obtain the viscoelastic properties and with the DENT test to study the ductile failure. The mastics were prepared with a single emulsion, one source of the inert filler and different concentrations (15, 30, 45, and 60%) of active filler (Portland cement or fly ash). The main conclusions are as follows:

(i) The use of the shear complex modulus and the DENT test allows differentiating the behavior of the mastics with different amounts of active filler.

(ii) The methodology to prepare the mastic does have an influence on the behavior of the mastic. It was observed here that first mixing the emulsion with the inert filler before adding the active filler better shows the impact of the active filler amount.

(iii) Depending on the mixing methodology, the relationship between the stiffness and the amount of active filler can be very different. With the second mixing method used here, an optimum amount of OPC and FA was found, but those optimums are different. It is probable that if more water was available to interact with the active fillers, the optimum would be different. It is also important to note that if a different curing time was used, the results may change.

(iv) The addition of active filler does change the ductility of the mastics. The results show that in the mastics studied here, the mix with 30% OPC would be the toughest, but the 15% FA and 60% FA mastics have the strain tolerance, which can be translated in the highest fatigue resistance. Actual fatigue resistance tests are required to verify this conclusion.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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