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EXPERIMENTAL STUDY ON THE ENHANCEMENT OF MECHANICAL PROPERTIES OF BITUMINOUS MASTICS AT HIGH STRAINS

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Abstract

The paper presents the results of a laboratory investigation aimed at evaluating the effects of filler on the rheological properties of bituminous mastics. More specifically, the mechanical behaviour of mixtures made with bitumen and inorganic aggregate is analyzed: selected fines from Municipal Solid Waste Incinerator (MSWI) ashes were used. The interaction between binder and aggregate determines a change in the rheological behaviour. Experimental data show a viscoelastic behaviour in the usual operating temperatures range and in a dynamic stress configuration characterized by high strains. It can also be observed that the thermal susceptibility of the mastic markedly depends on the content of filler, which is responsible for the variations of stiffness and resilience, measured in terms of complex modulus and phase angle. In order to examine the rheological effect of inorganic filler, tests were performed with the objective of checking the mastic behaviour at the edge of the linear viscoelastic region. Above the linear viscoelastic threshold, the bituminous mastic is significantly sensitive to the presence of filler and a statistically robust protocol is required. The experimental data were obtained by means of a dynamic shear rheometer (DSR) and synthesized with a viscoelastic model. For the interpretation of the results, master curves of the main performance indices were calculated, being related to the filler content. As expected, the results confirm that artificial aggregate from MSW incinerators can be used in order to improve mastic properties.

Keywords: bituminous mastics, inorganic aggregate, marginal material, rheology

1 Introduction

In recent years, marginal and industrial waste materials have been widely used in various civil and industrial engineering sectors. These materials have demonstrated a good capacity of integration with the traditional materials and an ability to substitute aggregates of natural origin, contributing to a saving of natural aggregates extracted from quarries, and therefore to environmental conservation. In particular, the diffusion of incinerators has made residues available from the combustion of municipal solid waste (MSW) in the form of slags, which are worthwhile studying for their use in road pavements [1-8]. The MSW slags, at one time disposed of in dumps, cannot be used as they are, because they often contain unsuitable elements (metal residues like nails, bolts, cutlery) and tend to release substances (heavy metals or very fine particles) that are potentially polluting for soils and groundwater. The material may also contain components incompatible with the bitumen under the electro-chemical profile, which impede the mixing or performances of the asphalt concretes. The slags are therefore utilizable only after a selection process. It should be remembered that the major strong point of this waste material is its low cost. For this reason, economic fractionation methods or selection processes (vitrification) of the aggregate should be used to provide a suitable aggregate for use in construction.

In this paper it was decided to use only the finest fraction (the filler, D < 0.075 mm) of the MSW slags to study the variation of the mechanical properties of the bitumen and the interaction with the filler. The hot mixing of these two elements, filler and bitumen, gives a semisolid mastic, which influences the performances of the asphalts produced. In particular, it was demonstrated that the presence of the filler in the bitumen improves the durability of the mix, mechanical resistance and pavement performances: an increase of the filler content increases the stiffness of the mastic; instead, a reduction of the bitumen content reduces the stability of the compound. Although there are some studies that demonstrate a reduction of the principal physical-mechanical characteristics of the mixtures containing marginal materials of industrial origin, compared to analogous mixes with conventional materials, recent studies have evidenced the improvements obtainable with the use of filler from MSW slags [5-8]; starting from these, based on the study of the mechanical behaviour of the mastics in the linear viscoelastic field (LVE), it was decided to extend the characterization to the limits of this region, i.e. to the conditions in which the material is subjected to high strain levels, when the normal models start to lose their efficacy. With this aim, a dynamic shear rheometer (DSR) was used to support the dynamic analysis (DMA), in a sinusoidal oscillatory regime, of these materials and to determine the principal rheological properties (complex modulus, phase angle) of the bitumen. The use of a DSR for the characterization of mastics has long been debated, because, in the past, the material drove the instrument to its operative and mechanical limits. In this research an instrument was used that could support the necessary regimes of force and strain [9-13, 15]. Moreover, the quantity of filler added to the bitumen, the dimensions of the filler (maximum nominal diameter) and the states of torsion reached are compatible with the current technical standard and are well within the working limits of the instrument. The results of the analyses conducted during this study have identified the effects of the rate of filler on the mechanical behaviour and consistency of the mastic.

2 Experimental programme and research setup

The aim of this study is to evaluate the improvement in the mechanical properties of bitumen with the addition of filler, seeking the optimal content of the latter, which allows the performances to be optimized at the limit of the LVE field. The performances of the bitumen and mastic were analyzed using a dynamic shear rheometer (DSR), in particularly delicate conditions that are intensified with the increase in the rate of filler. The empirical characteristics of the binders and mastics were analyzed first in order to classify and select them. In the next phase, after having determined the limits of the LVE field, laboratory tests for the rheological characterization of the bitumen and mastics were conducted in controlled conditions, varying the rate of filler.

2.1 Materials

Different types of traditional bitumen were used, with variable origin (all from Italy) and performances. The bitumen specimens were prepared and stored according to the European standard EN 58. The binders were chosen on the basis of their physical-mechanical characteristics: modified bitumens were excluded and those not in line with the prescriptions of the current European standards. The slags from MSW combustion, collected from a North-East Italy plant, were heated in an oven at a temperature of 105 °C to eliminate the residual moisture. Samples were then sieved and the fraction passing through a 0.075 mm diameter sieve was collected. The mastics prepared for the tests contained the following quantities of filler: 25%, 50%, 75% and 100% of the mastic weight. These values are in line with the typical prescriptions for asphalt mixes for road use, and are consistent with the traditional types and with research conducted in this field [14]. In order to guarantee that the standards defined by the project protocol were met, all the preparation and mixing phases were monitored using an infrared NEC Thermo Tracer TS9260 camera (T-IR). The test were entirely performed at the Road Laboratory of the University of Padua.

2.2 Empirical Analysis

The following tests were performed: penetration at 25 °C (EN 1426), softening point (R&B) temperature (EN 1427), Fraass breaking point (EN 12593), dynamic viscosity at 135 °C (EN 13702), mass-loss on heating – LOH (EN 12607/1), penetration at 25 °C after LOH (EN 1426). The initial empirical analysis identified three classes of penetration: 35/50, 50/70 and 70/100 mm/10; a summary of the first two tests for the bitumens and relative mastics is presented.

2.3 Dynamic and Rheological Analysis

The tests, conducted with an AntonPaar MCR 302 DSR, were done in sinusoidal oscillatory regime, varying the frequency (f) and temperature. Two geometries are usually used in this type of test, which have a diameter of 8 mm and 25 mm. However, the preliminary tests demonstrated a discrepancy between the results obtained in the two cases. For this reason it was decided to use the two testing plate geometries (Table 1), again in accordance with the prescriptions in the technical standard. The gap depends on the geometry and amount of material tested (Table 1). The tests were repeated 3 times for each type of material and the results were then averaged. The tests (in frequency sweep) were conducted in accordance with the prescriptions indicated in the standard EN 14770: the bitumen, or mastic, was poured onto the lower plate of the DSR at a temperature of 130 °C (±10°C). The value of normal force was fixed: during the descent of the upper plate (‹ 0.5 N), to avoid the mastic suffering a pre-stress due to excess material; during the trimming phase; during the test (N_f= 0 N), to maintain the contact between plate and bitumen; during the conditioning phases. The limit of the linear viscoelastic field was determined for all the materials with separate tests, in amplitude sweep: strain limit was determined on the average of five specimens and for all temperatures (e.g. 1% to 5%).

Table 1 Test conditions and spindle geometry

Testing geometry	Diameter [mm]	Gap width [mm]	Temperature [°C]	Step Temp. ΔT [°C]	Reduced Frequency [Hz]
PP08	8	2	10 to 35	5	0.016 to 16
PP20	20	1	30 to 80	5	0.015 to 16

For the construction of the isothermal curves of the complex modulus (G*) and the phase angle (δ), different tests were done, in frequency sweep, with gradual 5 °C temperature increases (ΔT), on three identical specimens. The spindle compliance condition was verified directly on the curves in frequency sweep, conducting the tests with both the geometries at temperatures of 30 and 35 °C. The reproduction of the master curves of the complex modulus |G*| and phase angle δ is at the arbitrary reference temperature of 25 °C. The single isothermal curves represent the response of the material to a loading cycle in frequency sweep. These were shifted utilizing a polynomial factor present in the literature [14]. In this connection the principle of time-temperature superposition (TTS) is considered valid and the materials are considered "thermo-rheologically simple": an increase in the temperature reduces the resting times of the structural processes associated to the strain and flow. If this reduction is the same for all the times of the resting spectrum, an increase of the temperature determines in the diagram log(|G*|)-log(f) a horizontal shift of the response G(f) towards shorter times and without substantial vertical movements [15]. All the materials are represented with the respective average model curve, between 1.0E-05 and 1.0E+02 Hz; the lines relative to the extremities of the curve differ according to the maximum (min torque) and minimum temperature (max torque) reachable with the tests in frequency sweep.

3 Results and discussion

The master curves presented in this paper were calculated with reference to a model proposed in previous studies [11, 12, 14, 15] and suitable to represent these materials. The analytical formulation, although not simple, is suited to the programming of iterative cycles that allow a well-defined curve to be reached with five parameters (G_g, R, k, w, δ_∞). For the modelling, as suggested by the authors, it was initially decided to set the maximum value of the complex modulus at 1 GPa. Nevertheless, with the analysis procedure and available data, it was observed that the model adhered better to the experimental data if the first constraint was eliminated. Although the optimization of the model is at an initial stage, for the presentation of the results of this study it was decided to add another degree of freedom, also freeing the constraint of the minimum value of the complex modulus ($Gf_{\rightarrow 0} \neq 0$).

3.1 Unaged unmodified bitumen pen 35/50

Between types available, for this class of penetration, two bitumens were evaluated as being suitable: 35/50Li and 35/50TO. The empirical profile shows that the mastic is more consistent and less sensitive to the temperature of the original bitumen (Table 2). Although the bitumen is the originator of the mechanical behaviour of the mastic, the influence of the filler content is clear on the two bitumens and shows in a different way. The values of the complex modulus are comparable on average and increase with the filler content, the bitumen 35/50TO acquires greater stiffness with the same added filler. The phase angle and, therefore, the resilience of the mastic do not present similarities: in the first case (35/50Li) the resilience tends to diminish with the addition of filler; in the second case (35/50TO) the comparison with the original bitumen depends on the test frequency (figures are omitted due to lack of space).

Table 2 Bit. 35/50. Softening point temperature [°C] and penetration [mm/10]

Property/mastic	0% Filler		25% Filler		50% Filler		75% Filler		100% Filler	
Sign	Li	TO	Li	TO	Li	TO	Li	TO	Li	TO
Soft. Point [°C]	47	48	50	49	52	52	58	57	64	65
Pen. [0.1 mm]	37	39	39	34	30	28	25	23	22	23

3.2 Unaged unmodified bitumen pen 50/70

Three bitumens were examined (50/70Ve, 50/70Li, 50/70TO) that respected the indications in the standard. The expectations about the effect of the filler content were confirmed, for all the empirical tests. Although the bitumens belong to the same class, the filler produces a different evolution of the values of penetration and softening temperature (Table 3), more marked with respect to the class 35/50 pen.

Table 3 Bit. 50/70. Softening point temperature [°C] and penetration [mm/10]

Mastic Property	0% Filler			25% Filler			50% Filler			75% Filler			100% Filler		
Sign	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO
Soft. [°C]	53	45	49	54	47	50	54	49	52	60	54	57	65	60	64
Pen. [0.1 mm]	68	65	54	57	53	48	42	43	36	32	37	34	24	29	27

In all three cases examined an increase of the stiffness is observed, in agreement with the increase in filler content, while the resilience of the mastic reduces. The cause-effect ratio between bitumen and filler is not comparable. In the first case (50/70Ve) the filler determines

a shift of the stiffness towards higher levels of G*; the phase angle remains relatively low (δ<80°) and it is not possible to predict the position of the lower horizontal asymptote: as the temperature rises the mastic loses its resilience, shown by the original bitumen, therefore the value of δ_∞ cannot be assumed constant and equal to 0°.
The results obtained from the tests on the bitumens 50/70Li and 50/70TO, and relative mastics, are congruent with the expectations as regards the stiffness (G*). Nevertheless, the relationship between loss of resilience and filler is strongly influenced by the original bitumen: the 50/70Li appears to be less sensitive than the others; while the 50/70TO appears to be unaffected by the filler at low rates (25% and 50%) to then clearly lose resilience with higher rates (75% and 100%). In this phase tests were not done at temperatures lower than 10 °C, so it is not possible to make precise predictions on the position of the value of the glass modulus. The comparison between the moduli of the three bitumens shows that the 50/70Ve evolves with a lower gradient and towards a lower glass asymptote.

3.3 Unaged unmodified bitumen pen 70/100

The selection of the bitumens produced three samples: 70/100Ve, 70/100Li and 70/100TO. It is not possible to identify a connection between filler content and sensitivity of the bitumen to the variation of the temperature. The ratio is not linear and reflects the dominance of the filler (e.g. 75-100%). Although there are slight differences (see Table 4, penetration at 25% and softening point at 75% filler content), empirically the three bitumens are assimilable. This analogy is completely lost observing the results of the tests with the DSR: both the stiffness and resilience have different evolutions with respect to the loading frequency. The bitumen 70/100Ve is the most sensitive to the filler content: it was originally, under the same conditions, the least rigid and most resilient. The derived mastics show a clear reduction of the resilience, to the point that it is reasonable to assume a lower horizontal asymptote at around 45°.

Table 4 Bit. 70/100. Softening point temperature [°C] and penetration [mm/10]

Mastic Property	0% Filler			25% Filler			50% Filler			75% Filler			100% Filler		
Sign	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO	Ve	Li	TO
Soft. [°C]	46	44	46	48	47	47	50	49	49	56	52	52	61	60	60
Pen. [0.1 mm]	72	75	75	62	67	60	42	49	49	41	40	40	34	31	32

Table 5 Stiffening and melting effects of filler content

Original Binder	1.0E+00 Hz δ [degs]	1.0E+00 Hz |G| [Pa]	1.0E-01 Hz δ [degs]	1.0E-01 Hz |G| [Pa]	1.0E-03 Hz δ [degs]	1.0E-03 Hz |G| [Pa]
30-50 Li	67.46	3.1E+06	75.20	5.8E+05	86.64	1.0E+04
30-50 TO	67.49	6.7E+05	87.36	5.5E+05	72.34	1.1E+04
50-70 Li	69.76	1.7E+06	77.67	2.9E+05	87.84	4.5E+03
50-70 TO	65.78	1.9E+06	73.61	3.4E+05	85.08	5.2E+03
50-70 Ve	57.65	1.7E+06	62.36	4.8E+05	73.52	1.9E+04
70-100 Li	61.78	1.6E+06	80.31	2.4E+05	88.31	3.1E+03
70-100 TO	67.48	1.1E+06	74.98	1.9E+05	85.61	2.8E+03
70-100 Ve	61.93	9.1E+05	68.42	1.9E+05	80.52	4.0E+03

50% Filler	1.0E+00 Hz δ [degs]	1.0E+00 Hz |G| [Pa]	1.0E-01 Hz δ [degs]	1.0E-01 Hz |G| [Pa]	1.0E-03 Hz δ [degs]	1.0E-03 Hz |G| [Pa]
30-50 Li	68.71	4.5E+06	76.12	8.3E+05	87.17	1.3E+04
30-50 TO	67.73	5.4E+06	75.03	1.0E+06	86.61	9.4E+03
50-70 Li	71.75	3.0E+06	78.79	5.0E+05	88.07	7.6E+03
50-70 TO	66.61	2.7E+06	74.02	4.8E+05	85.07	8.4E+03
50-70 Ve	60.62	3.1E+06	64.19	8.9E+05	73.97	3.5E+04
70-100 Li	73.26	2.6E+06	81.01	3.8E+05	88.70	4.5E+03
70-100 TO	68.03	1.7E+06	75.06	3.0E+05	85.82	4.8E+03
70-100 Ve	64.49	1.4E+06	69.73	2.9E+05	80.84	6.3E+03

100% Filler	1.0E+00 Hz δ [degs]	1.0E+00 Hz |G| [Pa]	1.0E-01 Hz δ [degs]	1.0E-01 Hz |G| [Pa]	1.0E-03 Hz δ [degs]	1.0E-03 Hz |G| [Pa]
30-50 Li	71.97	6.0E+06	77.97	1.1E+06	87.56	1.8E+04
30-50 TO	70.89	7.2E+06	76.80	1.3E+06	86.38	2.0E+04
50-70 Li	73.84	4.2E+06	80.00	6.9E+05	88.26	9.3E+03
50-70 TO	68.45	4.3E+06	74.39	7.4E+05	85.36	1.1E+04
50-70 Ve	63.35	4.2E+06	66.40	1.2E+06	74.71	5.2E+04
70-100 Li	73.85	4.1E+06	80.98	6.0E+05	88.46	8.2E+03
70-100 TO	69.69	3.0E+06	75.42	5.3E+05	85.64	8.5E+03
70-100 Ve	66.20	2.1E+06	70.65	4.3E+05	81.16	9.8E+03

An important result obtained from the tests regards the operative limits of the mastic produced with this filler: the greatest effects on the phase angle are located beyond the frequency of 1 Hz and become increasingly more marked. Table 5 shows that all the bitumens only undergo an effective increase of the performances if the rate is above 50% in weight; however, in all the cases, the increased stiffness is limited within same order of magnitude. While the bitumen 35/50TO shows anomalous behaviour at low rates, in general all the other binders establish an effective bond with the filler with stiffness increments that increase with the frequency.

4 Conclusions

A campaign of tests has been formulated for the characterization of unmodified and unaged bitumens that has confirmed the weakness of the empirical tests in the comparison with the results of the DSR, in particular as regards the resilience of the mastic.
As expected, the bitumen tends to show Newtonian type behaviour at high temperatures ($G_0 \rightarrow 0$, T_{max}=80 °C), unlike the mastic. In this particular region of the viscoelastic behaviour, the mastic supplies performances that initially depend on the original bitumen. As the rate of filler increases, this tends to compensate for limits of the initial bitumen, taking stiffness and resilience to comparable levels, unconnected with the penetration class of the original bitumen. At low temperatures, the complex modulus seems to be far removed from the threshold of 1 GPa, for all penetration classes. This classification does not reflect the physical-chemical interaction between the two elements following the mixing, and it is not simple to predict the effects on the loss of resilience. The complex modulus increases significantly with the filler content, therefore this material supplies stiffness to the original bitumen. On the other hand, the resilience is lowered, especially at the extreme limits of the frequencies and temperatures. The greatest benefits are therefore found in the interval of central frequencies, between 0.001 and 1 Hz, where the phase angle results as practically independent of the filler. The domination of the filler, as expected, appears at the extremes of the investigated frequency interval, especially the upper one, i.e. where the instrumental limits of the test configuration are reached. The influence of the stiffness of the material on the results of the test with the DSR should be taken into account: it is reasonable to suppose that the instrument and test configuration play an important role, that there will be errors that increase in intensity with the increase of the filler content, complexity of the mastic and approaching the operative limits of the instrument (max and min torque). The tested mastics are classified as thermo-rheologically complex materials, so it is reasonable to hypothesize that ageing will produce more marked effects on the filler bitumen interaction. The model used to construct the master curves allows a simple and efficient calculation, but tends to fail for mastics with high percentages of filler. It seems that there is a strong physical-chemical interaction between the two materials: the aggregate dominates the bitumen if dosed at above 50% and can no longer be considered as a solid dispersed phase.

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