Shear rheology and microstructure of mining material-bitumen composites as filler replacement in asphalt mastics

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Magnetite is a mineral that occurs in many types of igneous rock and can be found in large quantities in fluvial and marine environments. It can also be extracted by reprocessing of existing mine tailings. In this paper, magnetite supplied from two different mining sites was evaluated as a substitution of natural limestone filler in asphalt mix design. Magnetite and limestone fillers were added to the bitumen according to three filler/bitumen ratios to form composite asphalt mastics.

Rheology of the mixes was analysed to study the effects of magnetite as potential filler in asphalt pave-ment applications and comparisons were made with natural limestone filler-based mastics. In addition, particle size analysis, scanning electron microscope, Cryo-SEM and energy dispersive X-ray spectroscopy, were conducted to comprehensively characterize the composite mixes.

Results showed that the addition of magnetite-based ferromagnetic filler in asphalt mix design repre-sents a suitable way to recycle this material, which is available in large quantities in many countries. In addition, ferromagnetic particles could also be exploited for induction or microwave healing of asphalt cracks as demonstrated by recent studies.

Viscoelastic properties of the bituminous mastic are improved by reducing the mastic's temperature and loading time susceptibility; stiffness and elastic behaviour at high temperature was also improved hence potentially increasing resistance to permanent deformation.

Keywords:
Asphalt
Bitumen
Magnetite
Mastic
Rheology
Viscoelasticity
Ferromagnetic

1. Introduction

Asphalt mastic is the combination of bitumen and filler material when mixed in due proportion; its mechanical and rheological (time-temperature dependency) behaviour is strongly affected by the relative dosage and unique properties of each of the two components. Asphalt mastic greatly contributes to the overall performance of asphalt concrete in road pavements, conveying most of its viscoelastic, chemical and physical properties to the asphalt mixture [1]. In this study, magnetite was added to the asphalt binder as possible replacement for filler material (particle size smaller than 75 μm), commonly sourced from nearby quarries. Magnetite (Fe_3O_4) is a mineral that can be easily found in Australia, Europe and USA; large deposits are also found in India and China, where tailing ponds of magnetite occupy vast spaces and represent serious risk of pollution, health hazard and environmental danger [2]. Magnetite is a quite inexpensive iron ore commonly

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used in coal processing and, together with other iron oxides, in the production of steel and iron products.

Nowadays, since non-renewable resources used in road pavements (i.e. natural aggregates and bitumen) are limited [3], researchers are trying to include alternative materials such as recycled and waste materials into asphalt pavement mixes [4]. At the same time, research effort is being carried out to reuse and recycle products that also provide additional properties to the asphalt mixes by means of improved strength, durability, lower environmental impacts, or various engineered properties; e.g. waste oil to promote rejuvenation of asphalt or ferrous material to generate induction healing capabilities [5,6].

Among the most used recycled materials in road engineering it can be found plastic [7,8], shredded tire rubber [9,10], foundry sand [11], glass [12], reclaimed asphalt pavements (RAP) [13,46], and metal-related tailings from mining industry. The latter counts many studies for the use of asbestos [14], coal [15], copper mine [16], iron and magnetic ore waste [17] as potential aggregates to enhance asphalt's mechanical behaviour and several other properties (e.g. healing, snow-melting, self-sensing). Wang et al. [18] used magnetite as aggregates in asphalt mixes to enhance the healing property of bitumen by microwave heating due to the ferromagnetic properties of magnetite. Several research studies also focused on the effects of different types of filler in asphalt mastics [19]. Glass waste was used as filler in asphalt material [12]; the study proved that stiffer and denser asphalt could be achieved and more stone-to-stone contact points were found in the aggregate matrix, which suggested improved strength. Li et al. [20] used steel slags as filler; their results showed improved high temperature rheological properties in comparison to limestone natural fillers. Asphalt mixes involving ferromagnetic materials such as magnetite or residuals from steel wool fibres processing [21] can be very useful to enhance healing properties of bitumen by induction heating, electric conductivity or microwave heating [17].

In this study, rheology of standard bitumen and asphalt composite mastics was evaluated to analyse and compare the viscoelastic behaviour at different temperature and loading condition; specifically, three magnetite-based filler/bitumen ratios (0.5, 1.0 and 1.5) were studied in accordance with Superpave spec-ifications [22] and common road construction practice and mix-design.

As shown by several studies [23–25], the addition of filler to bitumen makes the mastic stiffer; the stiffening effect commonly improves the resistance to high temperature deformation (e.g. rutting) although resistance to cracking and low-temperature behaviour could be jeopardized. The magnitude of these effects depends on the type of mineral filler, its size and shape [26], alka-linity and surface characteristics [27], and physical and chemical interactions that happen between the filler and the bitumen [28].

2. Materials and experimental plan

The present research study aimed to evaluate the viscoelastic properties of twelve different magnetite-bitumen composite mixes obtained by combining two types of bitumen, magnetite from two mining sites and three filler/bitumen ratios. Comparisons were also made with six mixes containing standard bitumen and natural limestone filler supplied from a nearby quarry.

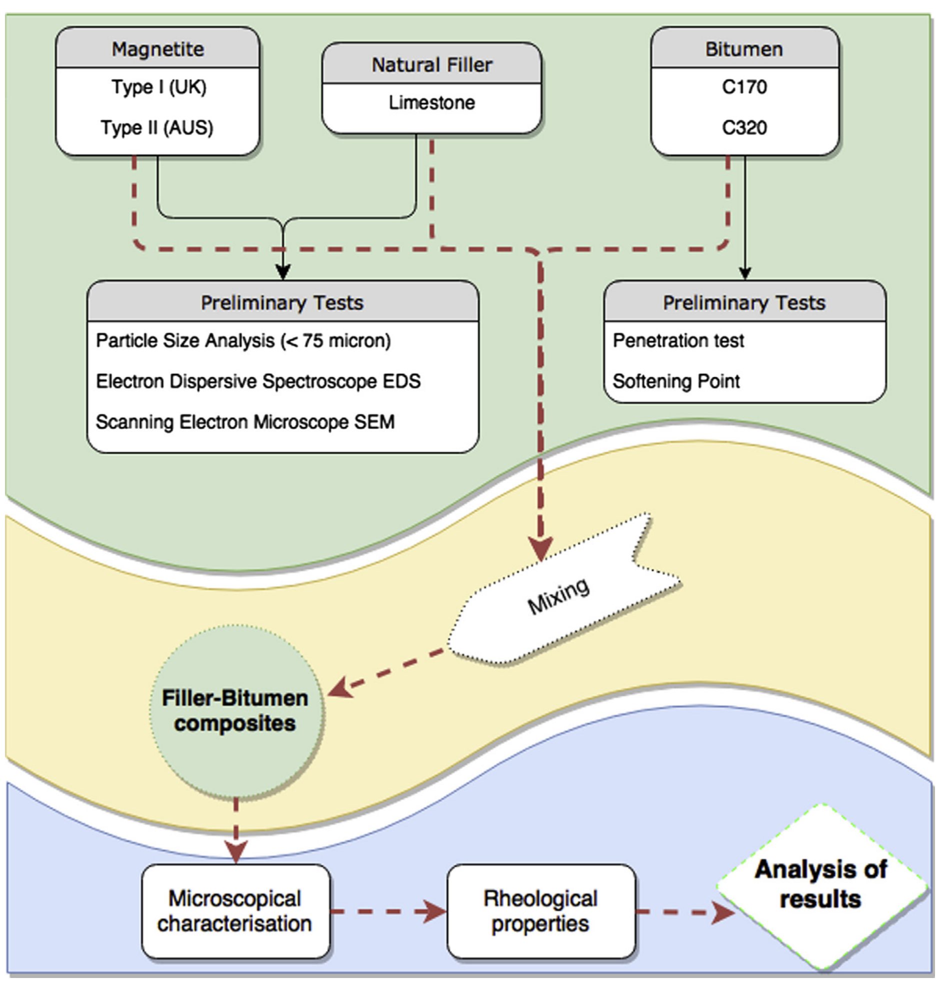

Fig. 1. Experimental plan.

Magnetite samples were analysed with SEM analysis (scanning electron microscope, FEI Quanta 200) to preliminarily evaluate optical differences in size, shape and elongation of magnetite particles supplied in UK and Australia; a particle size analysis (Mastersizer Hydro LV, wet mode) was also conducted to find the effective grading curve of the material below 75 μm. Filler material in road pavement application is in fact commonly recognised as the final part of the aggregate size distribution curve and includes all particles passing at 0.075 mm sieve; however, the relative size and shape of particles below 75 μm is of utmost importance and contributes to the final performance of the entire mix.

EDS (energy dispersive spectroscopy) showed what types of elementary elements form the magnetite samples. Preliminary, bitumen was subject to standard physical tests such as the penetration test [29] and softening point test [30].

Magnetite mastics were created by mixing filler particles and bitumen with an overhead stirrer at mixing temperature of 150 ± 5 °C for six minutes at 2000 rpm. The same procedure was used for preparing bitumen-natural limestone filler samples.

Frequency sweep tests were run according to [31] using the HR3 TA Instruments dynamic shear rheometer (DSR) to study the viscoelastic behaviour of the composite mixes at various temperature and loading frequency; Cryo-SEM microscopic analysis was also conducted to evaluate the dispersion of the magnetite in the composite mixes.

The experimental plan is summarised in Fig. 1.

2.1. Material characterization

According to the Australian bitumen classification system [32], bitumen used in this research is classified as C170 and C320 in accordance with the viscosity value at 60 °C. Table 1 shows the results of penetration and softening point tests on bitumen.

Three replicates were analysed during the entire experimental campaign at each test condition to guarantee repeatability of results. Frequency sweep tests were conducted at 0.1% controlled strain (within the region of linear viscoelastic response) and frequency range of 0.1–30 Hz. Tests were run from 5 °C to 80 °C at 15 °C-intervals (e.g. 5–20–35–50–65–80 °C). The 8-mm parallel plate geometry was used at 5 °C and 20 °C while 25-mm plate was used between 35 °C and 80 °C.

Complex shear modulus $|G^*|$ and phase angle δ for standard C170 and C320 bitumen were analysed; in particular, Fig. 2 shows isothermal curves (same temperature, variable frequency of loading) for bitumen C170.

The complex shear modulus can be expressed as the ratio of maximum stress to maximum strain and it is a common measure of the resistance to deformation of the bitumen when subject to loading (shear loading in case of DSR testing). The elastic component of $|G^*|$ ($G' = G^*{\cdot}\cos\delta$) is named storage modulus and the viscous components ($G'' = G^*{\cdot}\sin\delta$) is called loss modulus. The phase angle is instead the time lag between the applied stress and deformation response during a loading cycle of the oscillatory test. δ measures the viscous and elastic behaviour of the material being δ comprised within 0° (perfectly elastic material and no time lag between stress and strain) and 90° (purely viscous material).

Raw data from the DSR were first used to develop Black diagrams ($|G^*|$ on the y-axis and δ on the x-axis) as a practical indication of the thermo-rheologically simple behaviour of the bitumen [33]. Thermo-rheologically simple materials can deploy the time-temperature superposition principle to plot all isothermal curves as a single master curve at a reference temperature.

Table 1
Physical properties of bitumen.

Bitumen	Penetration test [0.1 mm]	Softening point [°C]	Viscosity at 60 °C [Pa·s]
C170	76	47.4	170
C320	47	52.1	320

The two curves depicted in Fig. 3 clearly show time-temperature equivalency whereas a fragmented type of curve would have shown the inapplicability of the superposition principle [34,35]. The latter is quite common in case of polymer modification of the bitumen.

Isothermal curves were combined using time-temperature superposition principle to develop bitumen master curves at a reference temperature of 20 °C.

Shift factors were calculated to horizontally shift isothermal curves at each test temperature using Arrhenius equation [36]. The activation energy (ΔH) in Arrhenius equation was calculated by plotting the logarithm of complex viscosity against the reciprocal of temperature and determining the slope of a linear fit to the data. The activation energy was then simply calculated as the slope multiplied by the universal gas constant (R = 8.314 J/mol·K); bitumen C170 and C320 showed activation energies of 126 and 122 kJ/mol, respectively. Fig. 4 depicts the master curves of neat binders C170 and C320.

Bitumen C320 is slightly stiffer than C170 in the frequency-temperature domain, especially at low loading frequencies (less than 0.01 Hz). This becomes more evident for even lower loading frequencies (high temperatures). The master curves of the neat bitumen provide a useful control parameter to further compare rheological modifications incurred due to mining (or limestone) filler addition. Frequency sweep tests were conducted on magnetite-bitumen composite mixes as well as on natural limestone filler-bitumen mixes, as shown in the following sec-tion. Eighteen mixes were prepared in order to study how 1) magnetite content affected rheology of bituminous mastics and 2) results compared to natural rock-based filler, commonly used in road industry (e.g. crushed limestone). Mixes ID (Table 2) were named after the type of bitumen (C170 or C320), followed by the type of filler (''I'' for magnetite I from the UK, ''II'' for magnetite II from Australia and ''N'' for natural limestone filler) and filler/bitumen ratio (f/b = 0.5, 1.0 and 1.5).

2.2. Scanning electron microscopy (SEM)

SEM tests were conducted on FEI Quanta 200 ESEM; previous evaluations showed that the best image quality was obtained by setting a high voltage (20 kV), spot size of 5.0 and working distance of 10 mm. ETD (Everhart-Thornley Detector) was used in SED mode (second electron detector mode). (See Table 3).

For the preparation of the sample, magnetite was dispersed in polyethylene using an ultrasonic bath and consequently attached on the sample holder through a silicon tape. Due to the high electrical conductivity of magnetite, there was no need to coat the sample for image analysis. Similar methodology was also used for the Energy Dispersive Spectroscopy (EDS) sample preparation.

At this stage, the two types of magnetite were compared optically; SEM images of magnetite were taken at different magnifications (1000× and 3000×), as shown in Fig. 5 for Magnetite I and Fig. 6 for Magnetite II.

The optical analysis revealed a variable particle size distribution for both types of magnetite, with irregular shaped grains. Magnetite I seemed more elongated (two dimensions being predominant on the third) and sharp-edged compared to particles of Magnetite II, more spherical and rounded on the edges. Furthermore, Magnetite I showed two prevalent size of particles; one being coarser (25–35 μm) than the other (0–2 μm). Magnetite II seemed instead well-graded within the filler common gradation limits (less than 75 μm). As detailed in the following sections, these features could provide variable rheological properties to the com-posite bituminous mastics and enhance (or reduce) the workability of the final pro-duct in real plant scenarios.

2.3. Particle size

A particle size analysis was conducted to analytically confirm the deductions from SEM images and plot the grading curve of both magnetite and natural limestone fillers. The test was run using the wet mode where almost 50 g of material were added to water in a Mastersizer Hydro LV. The following gradation curves were obtained (Fig. 7).

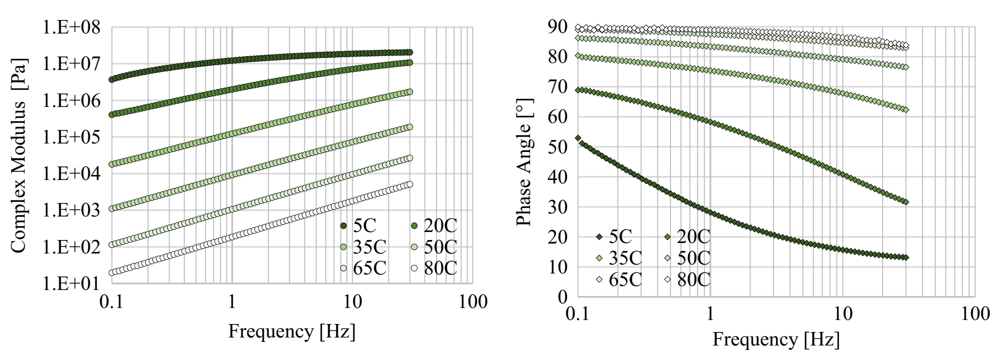

Fig. 2. Complex shear modulus and phase angle – C170 standard bitumen.

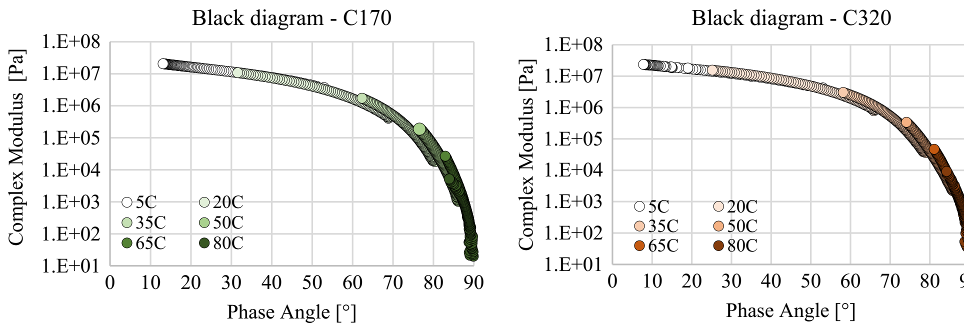

Fig. 3. Black diagrams for standard bitumen.

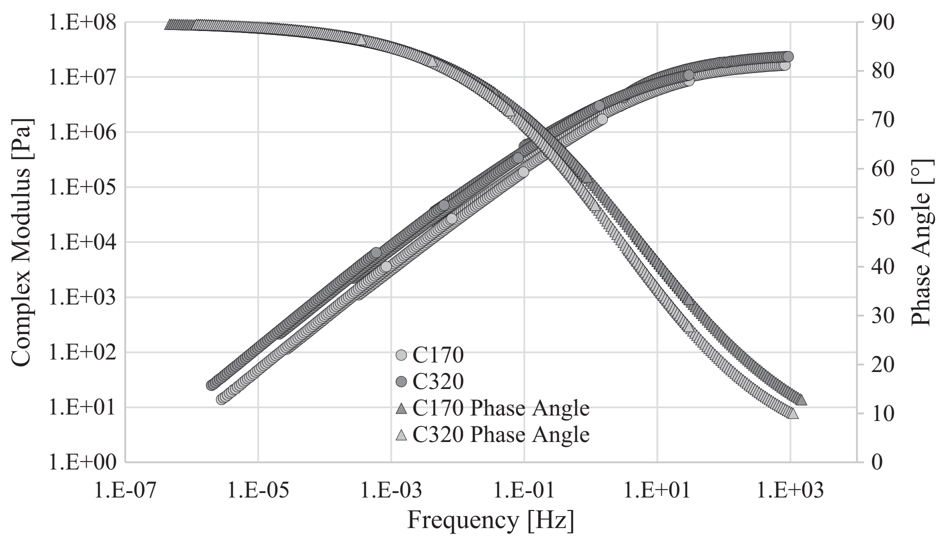

Fig. 4. Rheology of neat bitumen C170 and C320.

Table 2
Nomenclature of bituminous mastics.

	C170			C320		
Magnetite I	C170_I_0.5	C170_I_1.0	C170_I_1.5	C320_I_0.5	C320_I_1.0	C320_I_1.5
Magnetite II	C170_II_0.5	C170_II_1.0	C170_II_1.5	C320_II_0.5	C320_II_1.0	C320_II_1.5
Natural Filler	C170_N_0.5	C170_N_1.0	C170_N_1.5	C320_N_0.5	C320_N_1.0	C320_N_1.5

The different filler-bitumen ratios are shown in Table 3.

Table 3
Filler-bitumen ‘f/b’ ratios.

f/b [% mass]	Filler [% total mass]	Bitumen [% total mass]
0.5	33%	67%
1.0	50%	50%
1.5	60%	40%

The test confirmed the overall maximum size of the particles (less than 75 μm) and showed that the two ferrous fillers are generally similar in size below 5 μm but vary significantly in the range of 5–75 μm. In fact, Magnetite I showed a greater vertical ‘jump’ between 10 and 30 μm with almost 58% of the particles being retained within this range; Magnetite II is more well-graded with a gentler slope of the curve and no significant fraction being preponderant. The smallest detected particle was 0.52 μm on Magnetite I samples. It should be noted that smaller particles are ascribed to provide greater surface area and, potentially, generate more widespread heat during the induction heating processes [6,21], for instance.

Natural limestone filler proved to be coarser than magnetite fillers above 30 μm but much finer particles were found below 10 μm hence showing a lack of particles in the range of 10–30 μm compared to ferrous fillers. D(10) – the diameter at which 10% of material is passing – was close to 4.6 μm for both magnetite samples and 2.7 μm for the natural filler hence confirming the greater number of finer particles (e.g. below 5 μm) in the limestone sample. The Specific Surface Area also varied between approximately 475 and 665 m^2/kg for magnetite and natural filler samples, respectively.

2.4. Energy dispersive spectroscopy – EDS

EDS microanalysis technique was used to determine the chemical elements inside magnetite fillers using a Philips XL30 SEM. The test involves the generation of an X-ray spectrum from the entire scan area of the SEM due to inelastic interaction of the electron beam with the specimen atoms. Machine settings were as follows: electron beam of 20.0 kV, spot size of 6.0, magnification of 1000x and working distance of 10 mm. EDS samples were prepared according to the same procedure already explained for SEM samples.

A silicon tape was preferred to carbon in order to avoid false detection of elements during the test. Fig. 8 shows the EDS graphical results for Magnetite I and Magnetite II; peaks in the graph show the elements contained in the samples. Analytical results of EDS (e.g. percentage of elements in the sample) are shown in Table 4. As expected, magnetite (Fe_3O_4) mainly contains iron (Fe) – average 75% -and oxygen (O) – average 22%; although small amounts of Silicon (Si) and Carbon (C) were detected. It is believed that the small presence of Silicon (less than 1%) was related to the specific tape used during sample preparation; no trace of Silicon was in fact detected when other tapes were used.

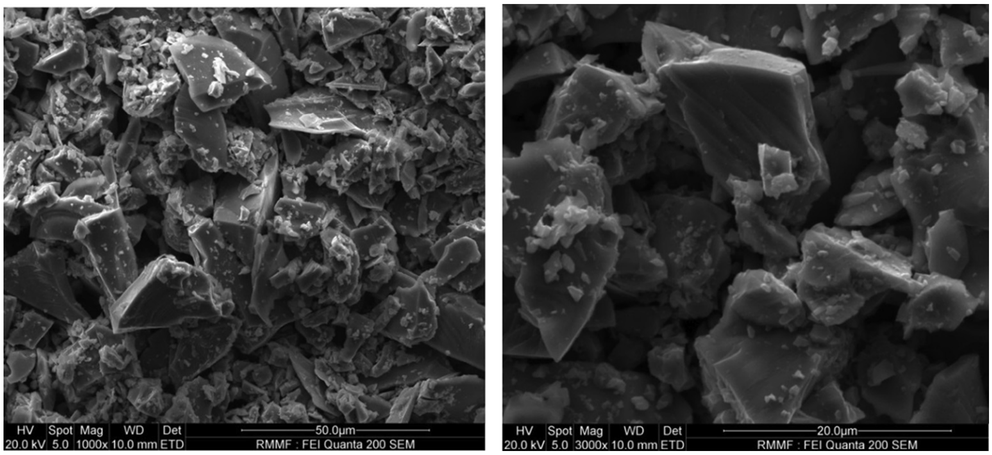

Fig. 5. Magnetite I, 1000× (left) and 3000× (right).

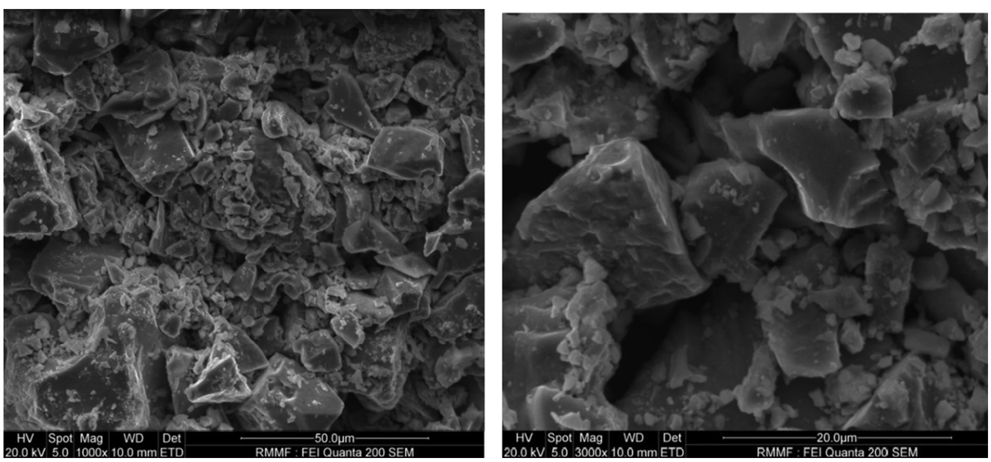

Fig. 6. Magnetite II, 1000× (left) and 3000× (right).

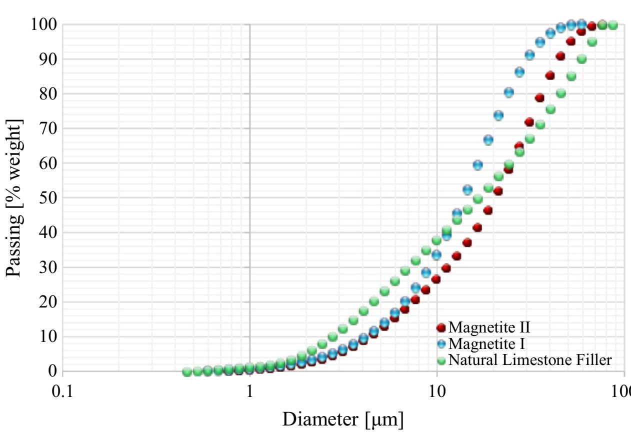

Fig. 7. Filler gradation curves.

The main difference between the two samples was the presence of Carbon in Magnetite II, probably attributed to the contamination or after-extraction washing treatments undergone at the mining site. Magnetite II therefore exhibited lower degree of purity than Magnetite I.

3. Results and discussion

Cryo-SEM was used to evaluate the dispersion of magnetite particles into the bitumen samples. Almost 0.4 g bitumen samples were submerged in a liquid nitrogen bath (−196 °C) for 10–15 s, then taken to a vacuum chamber at 10^{-5} Pa and −180 °C where the sample's upper tip was cut thus forming a pyramid-shaped sample. The samples were gold coated – as bitumen is non-conductive material – and finally inserted in the SEM chamber, where vacuum and temperature were maintained constant.

Images in Fig. 9 were taken on the top part of the fractured tip of the sample to optically quantify the dispersion of the magnetite in the mix. By comparing the three images, it was noticed that a sample with low filler/bitumen ratio showed larger areas of flat and smooth surfaces (e.g. bitumen-only phase) compared to high filler/bitumen ratio, where greater number of crests and fragmented peaks (e.g. magnetite phase) were spotted. However, a precise analytical evaluation of the magnetite dispersion into the samples was hard to establish through optical analysis due to the broad particle size distribution. It was observed though that no major clusters of magnetite particles were formed during the mixing and sample preparation processes.

The rheological characterization of the mixes was conducted with a Dynamic Shear Rheometer (HR3 TA Instruments). Tests were run on three different types of mix; neat bitumen samples (C170 and C320), bituminous mastics containing natural limestone filler in three proportions (f/b = 0.5, 1.0, 1.5) and bituminous mastics containing two types of magnetite at identical dosages. All the combinations of bituminous mastics included both the softer (C170) and the harder bitumen (C320) to evaluate the effect of

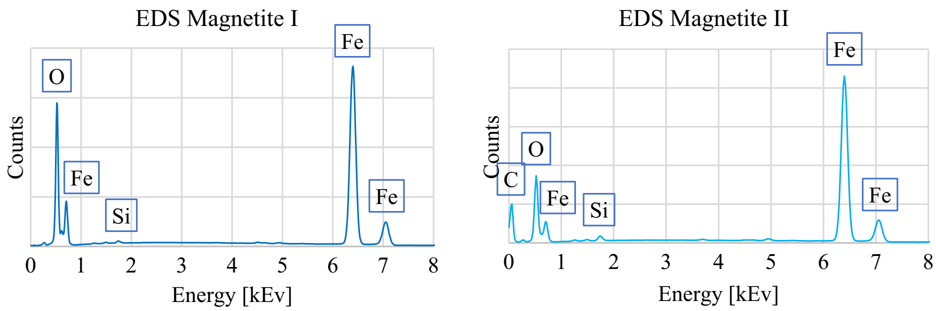

Fig. 8. EDS on magnetite fillers.

Table 4
Elements in the ferrous fillers by weight.

Element	Magnetite I	Magnetite II
Fe	79.1%	71.4%
O	20.7%	23.1%
Si	0.2%	1.0%
C	0.0%	4.5%

the base binder on the rheological behaviour of the mastic. Although the Superpave Mix Design Manual SHRP A-407 [22] indicates that filler proportion has to be defined as ‘the ratio of the percent by weight of aggregate passing the 75 μm sieve to the effective asphalt binder content expressed as percent by weight of the total mix’, it is commonly acknowledged that volumetric proportioning of filler in mastics could affect the physical and rheological behaviour [37,38]. This is also recognised by [22]. Usually, increasing the volumetric concentration of filler makes the mastic stiffer and reduced its strain at failure; this behaviour is emphasized at low temperature. The present research used f/b ratio according to mass proportions; the same approach has been adopted by many authors [39–44].

Frequency sweep tests were conducted in the linear viscoelastic region applying oscillating shear stresses at 0.1% controlled strain;

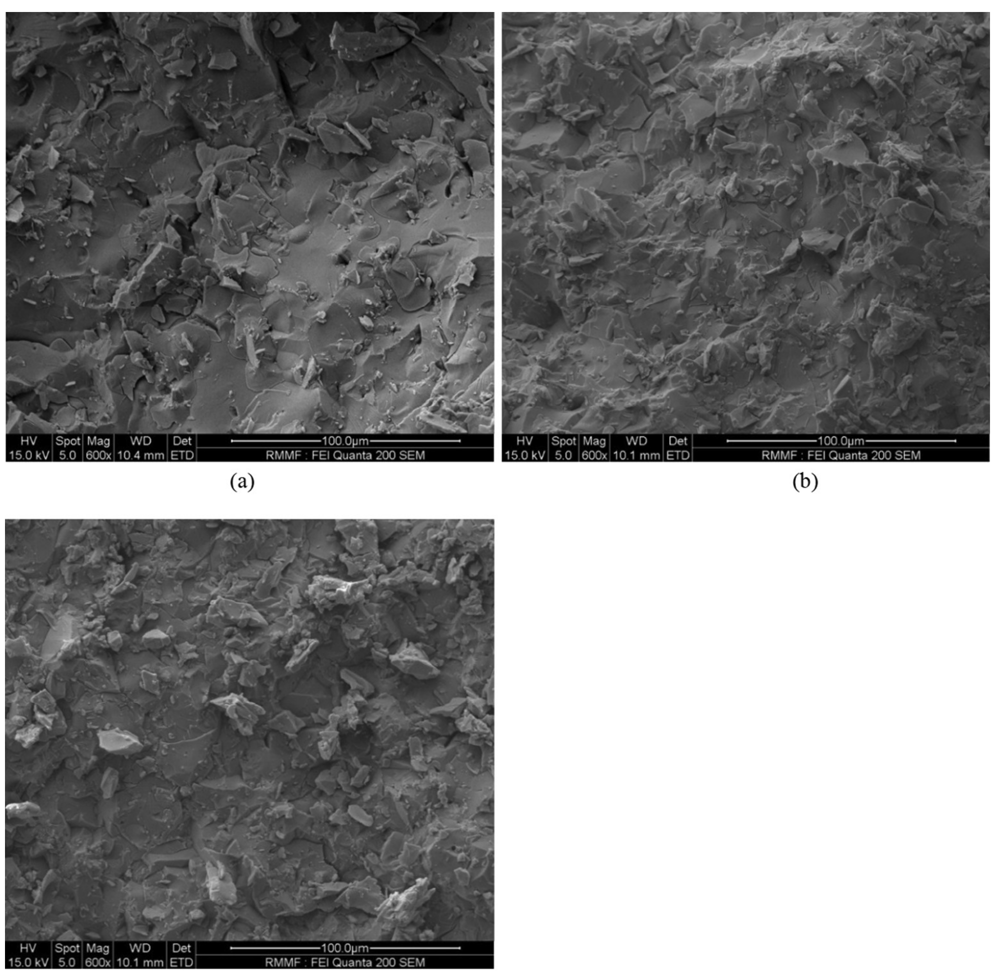

Fig. 9. Cryo-SEM images: Bitumen C170 with Magnetite I at three filler-bitumen ratios (0.5, 1.0, 1.5).

frequency varied from 0.1 Hz to 30 Hz. Temperature ramps were conducted from 5 °C to 80 °C at 15 °C intervals. Master curves of the complex modulus and phase angle were finally plotted shifting the isothermal curves as shown in the previous sections. Time dependency was represented using a reference temperature of 20 °C.

All composite mastics' master curves followed sigmoidal trends approaching a horizontal asymptote at high frequencies; this commonly corresponds to the stiffness value of the bituminous material at the glassy temperature. The so called glassy modulus has been indicated to vary between 1 and 1.5 GPa [45].

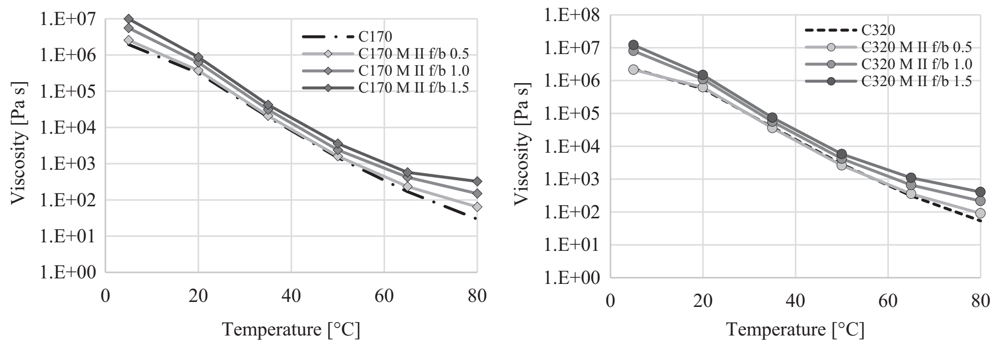

Fig. 10. Viscosity of bituminous mastics.

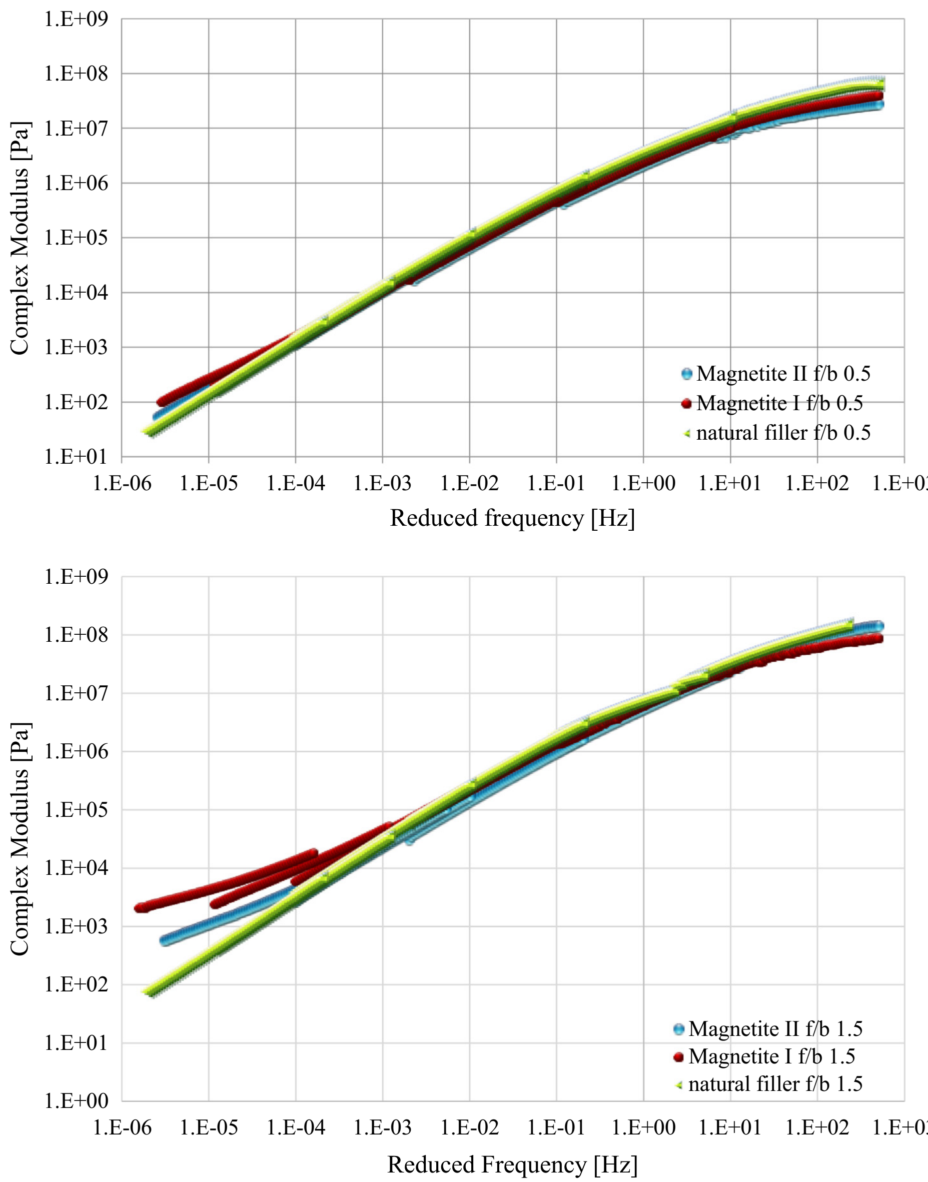

Fig. 11. Time dependency through master curves representation.

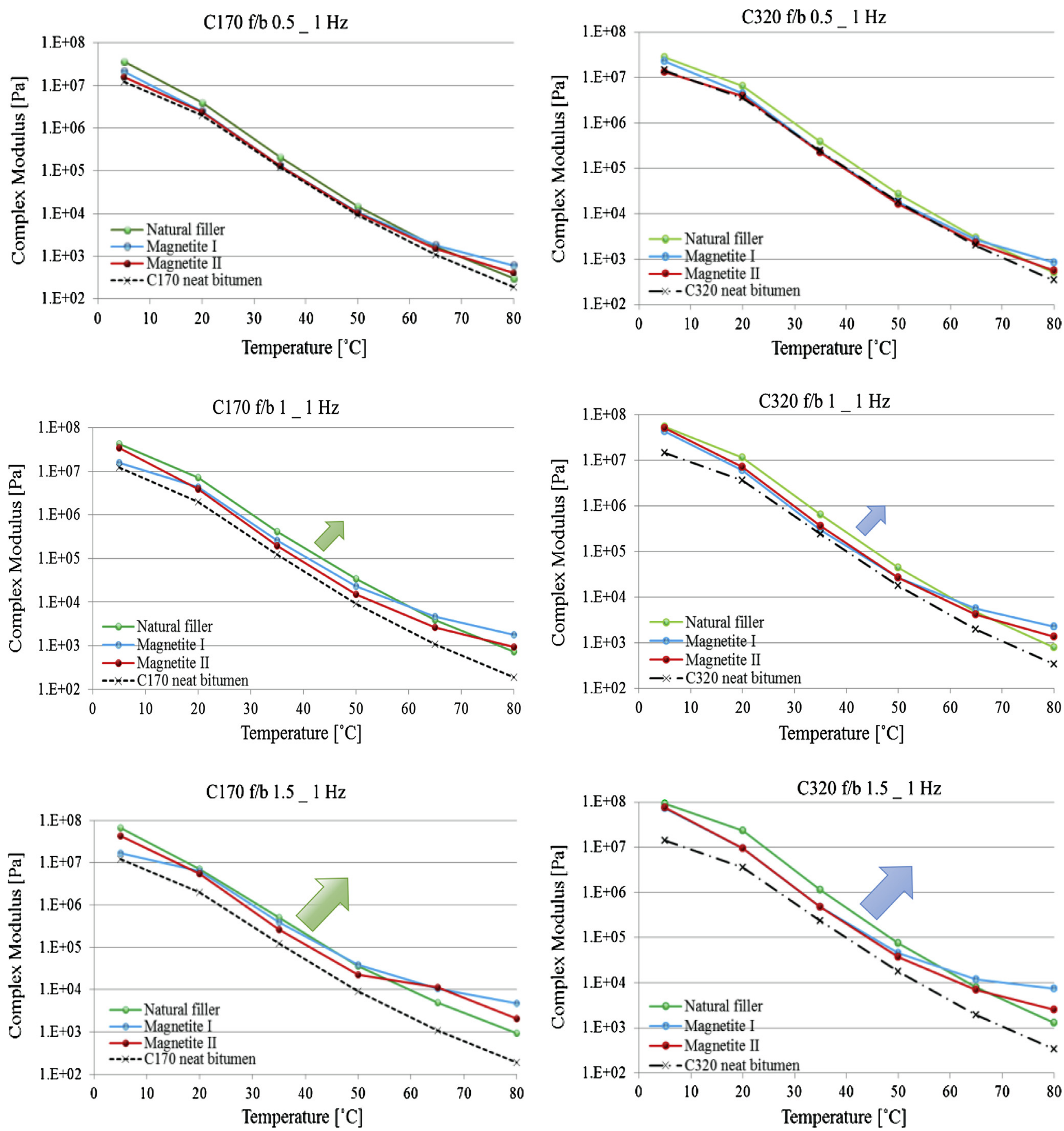

Fig. 12. Temperature dependency through isochronal curves representation.

Filler content proved to significantly affect the rheological behaviour of mastics. An increase in filler – from f/b of 0.5 to 1.5 – generated a rapid upward shift of the master curves. The rheological behaviour was also affected by the type of filler in the composite mastic. Specifically, the complex shear modulus of natural limestone filler mastics (Fig. 11) was predominant at high frequencies for all combinations of bitumen and filler/bitumen ratio although this change is modest for low filler contents (e.g. f/b 0.5). Ferrous fillers instead showed reduced stiffness at high frequency and increased stiffness at very low frequencies (from 0.01 Hz downward) for all combinations of bitumen and filler/bitumen ratio. In addition to that, ferrous composite mastics also exhibited lower phase angle (greater elastic response) compared to natural limestone filler. This trend proved lower temperature (and time) susceptibility of ferrous bituminous mastics compared to natural limestone ones. At high temperature (low frequencies), ferrous composite mastics can thus provide the road with greater elastic response and stiffness to better withstand permanent deformation. At low temperature (high frequencies), lower complex modulus values and increased viscous response could instead help to avoid thermal cracking. Although a slight reduction in stiffness was noticed for ferrous composite mastics, no appreciable improvements in viscous response were noticed compared to natural limestone filler.

Small differences were noted between the rheological behaviour of mixes containing Magnetite I and Magnetite II; in particular, Magnetite I showed greater complex shear modulus (stiffness) than Magnetite II at low frequency (high temperature). When the temperature increases the resistance to flow of bituminous mastics is determined by the solid particles of the filler since the viscosity of the bitumen is significantly low (Fig. 10). As observed through the SEM analysis, Magnetite I particles had in fact sharper edges hence capable of greater interlocking potential and resistance to flow compared to rounded particles in Magnetite II.

In regard to the base bitumen used to prepare the mastic samples, all trends and rheological modification were confirmed for both bitumen C170 and C320. As already observed in Fig. 4, the stiffer behaviour of bitumen C320 shifted all C320 master curves upward compared to C170.

Isochronal curves are a straightforward way to easily capture the temperature dependency of the mixes. Experimental data points were plotted (Fig. 12) at each of the test temperatures and at a frequency of 1 Hz. Compared to the neat bitumen (both C170 and C320), bituminous mastics all show an increase in com-plex modulus at all testing temperatures. This stiffness increase is strictly related to the quantity of filler in the mix; modest to significant changes in the complex modulus were noticed for f/b ratios ranging from 0.5 to 1.5, respectively. In particular, differences in stiffness were observed at high temperature (e.g. more than 50 °C), with f/b 1.5 mixes showing an increase of up to two orders of magnitude compared to the neat bitumen.

Different fillers produced different rheological responses; in particular, it was interesting to notice that natural limestone filler showed greater stiffening potential at low and intermediate temperature (e.g. between 5 and 35 °C) compared to ferrous fillers while an inversion occurred at high temperature (magnetite-based filler being stiffer than natural filler). Commonly, greater stiffness is appreciated at high temperature in road applications due to reduced susceptibility to permanent deformation (rutting) on the pavement whereas lower stiffness and flexibility of the mixes are generally valued at low (thermal cracking) and interme-diate (fatigue) temperatures. The analysis of the phase angles (e.g. viscoelastic response) produced similar results; lower phase angle hence increased elastic response was noticed at high temperature for magnetite fillers compared to natural filler, which was instead more viscous.

4. Conclusions

This paper evaluated the potential effect of including mining material, abundant in many countries, in road pavements. In particular, magnetite powder was added to bitumen in substitution of natural limestone filler, commonly used in road pavement applications. Being a ferromagnetic material, magnetite has also been deployed to provide the road with engineered capabilities and promote induction healing of cracks, for instance. A comprehensive evaluation of the physical, rheological and chemical properties was thus needed. This study comprised a multi-attribute evaluation of ferrous fillers particularly focusing on their rheological behaviour compared to standard filler material.

Based on the experimental results, the following conclusions can be drawn.

- Magnetite filler particles sourced at different sites differ by means of particle size distribution, particle shape and chemical composition. Two types of magnetite were supplied for this study from two mining sites. Slight difference chemical composition showed that magnetite can be contaminated during excavation and processing operations.
- Differences in size, shape and chemical composition, can modify the rheological behaviour of magnetite composite bituminous mastics. Overall, the rheological properties of mastics were improved when a small addition of magnetite powder was added compared to the same content of natural limestone filler. In particular, smaller temperature (and time) susceptibility was found in magnetite-based mastics, which can provide superior performance of the road pavement at low and high temperature. This was emphasised by filler/bitumen ratios greater than 1.
- Increasing the content of filler (high f/b ratio), notwithstanding of the type, produces increased complex shear modulus (stiffness) and reduces the phase angle (enhanced elastic behaviour).
- Magnetite fillers, potentially useful to generate particular engineered properties in road pavements, also showed improved rheological behaviour compared to natural limestone filler hence demonstrating their suitability as a valuable multipurpose road construction material.

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Declarations of interest

None.

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