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ARTICLE IN PRESS

Construction and Building Materials xxx (2014) xxx–xxx

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Toward more realistic viscosity measurements of tyre rubber–bitumen blends

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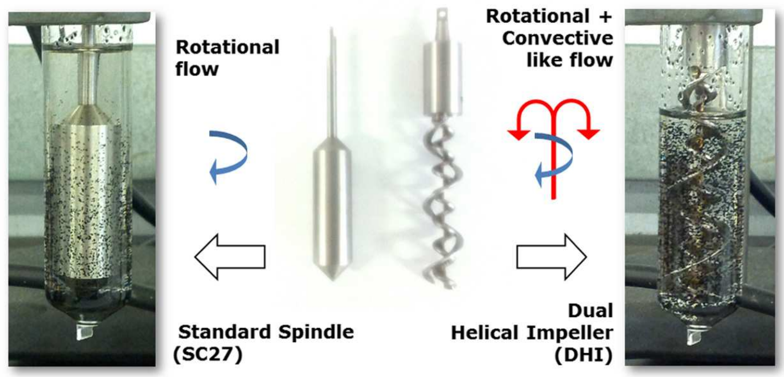
HIGHLIGHTS

- Introducing the practical issue with viscosity measurements of recycled tyre rubber modified bitumen.
- Rapid prototyping and visual assessment and dual helical impeller manufacturing.
- Dual helical impeller calibration.
- Validation of results through comparison with viscosity measurements undertaken with a standard impeller.
- Adapting the rotational viscometer as a low-shear mixer by using the DHI.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 14 August 2013
Received in revised form 24 March 2014
Accepted 25 March 2014
Available online xxxx

Keywords:
Dual helical impeller
Rapid prototyping
Electrical discharge machining
Viscometry
Recycled tyre rubber
Rubberised bitumen

ABSTRACT

The measurement of rheological properties of the tyre rubber bitumen blends is often challenging due to presence of suspended tyre rubber's crumbs. Furthermore, the phase separation during the course of measurements makes the viscosity of these non-homogeneous blends difficult to ascertain. In this study, a new dual helical impeller was designed and manufactured to be used with a rotational viscometer in order to have a real-time control of the viscosity while performing a laboratory mixing of the blends. Layer based manufacturing techniques showed to be a convenient method to produce complex shaped impeller prototypes before manufacturing the more expensive stainless steel assembly. Impeller geometry was optimised to create a convective like flow within the sample and so minimise phase separation. Shear rate constant is geometry dependent and a calibration exercise was carried out to ascertain this. Results of both calibration and validation phases showed that the new impeller provides reliable viscosity measurements of homogenous fluids such as neat bitumen. With regards to complex fluids the new impeller showed a more stable and realistic trend than that obtained by using a standard spindle. In fact, it was demonstrated that the new impeller significantly decreases phase separation within the blend and in turns provides a more realistic measurement of the viscosity. This system represents a feasible and improved solution for optimising the laboratory modification process of tyre rubber bitumen blends by adapting the rotational viscometer as a low-shear mixer.

Abbreviations: EDM, electrical discharge machining; ELTs, end-of-life tyres; FDM, fused deposition modelling; HA, high torque viscometer; LV, low torque viscometer; RTR-MB, recycled tyre rubber modified bitumen; RTR, recycled tyre rubber; SRC, shear rate constant.

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http://dx.doi.org/10.1016/j.conbuildmat.2014.03.038
0950-0618/© 2014 Published by Elsevier Ltd.

Please cite this article in press as: Lo Presti D et al. Toward more realistic viscosity measurements of tyre rubber–bitumen blends. Constr Build Mater (2014), http://dx.doi.org/10.1016/j.conbuildmat.2014.03.038

1. Introduction

1.1. Background

The accumulation of end-of-life tyres (ELTs) and premature pavement failures are both interconnected and dependant of each other due to enormous increase in traffic density and axle loading respectively. The use of RTR in asphalt pavements started 170 years ago, with an experiment involving natural rubber with bitumen in the 1840s [1], attempting to capture the flexible nature of rubber in a longer lasting paving surface. In 1960s scrap tyres were processed and used as a secondary material in the pavement industry. One application was introduced by Charles McDonald, a materials engineer of the city of Phoenix in Arizona (USA), who was the first to find that after thoroughly mixing crumbs of RTR with bitumen and allowing it to react for a period of forty-five minutes to an hour, this material captured beneficial engineering characteristics of both base ingredients. He called it Asphalt Rubber and the technology is well known as the "Wet process". By 1975, crumb rubber was successfully incorporated into asphalt mixtures and in 1988 a definition for rubberised bitumen was included in the American Society for Testing and Materials (ASTM) D8 and later specified in ASTM D6114-97. In 1992 the patent of the McDonald's process expired and the material is now considered a part of the public domain. Furthermore, in 1991, the United States federal law named "Intermodal Surface Transportation Efficiency Act" (then rescinded), mandated its widespread use, the Asphalt–Rubber technology concept started to make a "quiet come back" [2].

Nowadays, these rubberised bitumen materials, obtained through the wet process, have spread worldwide as solutions for different quality problems (asphalt binders, pavements, stress absorbing lays and inlayers, roofing materials, etc.) with much different evidence of success demonstrated by roads built in the last 30 years. Since the invention of McDonald, the wet process technology has been used and modified more widely in four states in the US: Arizona, California, Texas, and Florida. More recently wet process has been used also in South Carolina, Nevada and New Mexico. South Africa and Australia started introducing bitumen–rubber as a binder for asphalt and for seals from the early 1980s and mid 1970s respectively [3,4]. In Europe wet rubberised asphalt has been successfully used in road pavements application since 1981 in Belgium, as well as in France, Austria, Netherlands, Poland and Germany [5], more recently also in Greece [6] and UK [7], but the countries with a higher numbers of applications are Portugal [8], Spain [9], Italy [10], Czech Republic [11] and Sweden [12]. Nowadays the rubberised asphalt technology is being adopted in many other parts of the world: Taiwan was reported to have adopted wet process rubberised asphalt mixtures for flexible pavement rehabilitation [4]; furthermore, wet process rubberised asphalt has been trialled in Beijing and for use in new and maintenance work as part of the preparation for the 2008 Olympics in China and it has also been used in EcoPark Project in Hong Kong. On the basis of first positive experiences also Brazil [13] and Sudan [14] are strongly investing in the application of this technology for road pavements.

The term "wet process" refers to a whole family of technologies which varies a lot with regards with the chosen processing conditions. The nature of the mechanism by which the interaction between bitumen and RTR crumbs takes place has not been fully characterised. Traditionally it is reported that bitumen–rubber interaction is not chemical in nature [1], but other studies claim that the increase in binder viscosity cannot be accounted for only by existence of the rubber swelling particles [16]. The reaction itself is made up of two simultaneous processes (Fig. 1): partial digestion of the rubber into the bitumen on one hand and, on the other, adsorption of the aromatic oils available in this latter within the polymeric chains that are the main components of the rubber, both natural and synthetic, contained in the RTR. The absorption of aromatic oils from the bitumen into the rubber's polymer chains causes the rubber to swell and soften [17]. RTR particles are swollen by the absorption of the bitumen oily phase at high temperatures (160–220 °C) into the polymer chains, which are the key components of the RTR-MB to form a gel-like material. Therefore, during the reaction there is a contemporaneous reduction in the oily fraction and an increase of rubber particle sizes with a consequent reduction of the inter-particle distance. This implies the formation of gel structures that produce a viscosity increase up to a factor of 10 [1].

Rubber reacts in a time–temperature dependent manner (Fig. 2). If the temperature is too high or the time is too long, the swelling will continue to the point where, due to long exposure to the high temperatures, swelling is replaced by depolymerisation/devulcanisation which causes dispersion of the rubber into the bitumen. Depolymerisation starts releasing rubber components back to the liquid phase causing a decrease in the stiffness (G^*) while the elastic properties (δ) continues to modify (Fig. 1a and b). If temperature is high or time is long enough, depolymerisation will continue causing more destruction of the binder networking and so δ modification is lost [18]. The interaction between bitumen and rubber materials is material-specific and RTR-MBs are extremely dependent on the variability of these processing conditions, particularly to what concerns the temperature and time of reaction [19–21]. RTR-MBs must be properly designed and, where necessary, produced to comply with specifications and provide a quality product suitable for the expected climate and traffic conditions. Research is still on-going worldwide to validate and improve technologies related to rubberised bitumens and particularly to what concern the monitoring of the quality of the rubber–bitumen blends in the field-production as well as in the laboratory design stage. A key property of RTR-MBs that needs particular attention and definitely further improvement in its evaluation is viscosity. In fact as a consequence of the non-homogeneity of this material, ASTM D-6114 specification uses the concept of "apparent viscosity" to categorise the different type of rubberised binders. This means that as opposed to more homogeneous materials (i.e. neat bitumen) to evaluate the viscosity of RTR-MBs properly, information must be given about the shear conditions [22]. In this sense ASTM D6114 specifies to use rotational viscometer in low shear by performing measurements at 12 or 20 rpm depending on the type of viscometer: low torque (LV model), or high torque (HA model).

Since viscosity is the key properties monitored during the field-production and laboratory design of bitumen–rubber blend [24], in a previous study [25] a rotational viscometer has been adapted to act as a low shear mixer able to provide real-time viscosity measurements. This solution helps optimising a RTR–bitumen blend with an accurate control of temperature and by consuming only a small amount of the materials. Nevertheless, this procedure presents some downside due to phase separation occurring during the mixing process. In fact RTR particles tend to float on the surface, thus a manual movement up and down of a standard impeller is necessary to keep a good distribution of the particles within the blend. Of course, this practice affects the viscosity measurements and could damage the equipment. The rotational viscometer used in this previous study (commonly used worldwide) can be equipped with special accessories and impellers that can help with the measure of viscosity of the blend with suspended particles [26]. Nevertheless, none of them is apparently able to provide a better distribution of the particles through the sample volume. Therefore, when dealing with complex materials, such as RTR-MBs, there is the need of some innovation to improve standard rotational viscometers to adapt them as a low-shear mixer for

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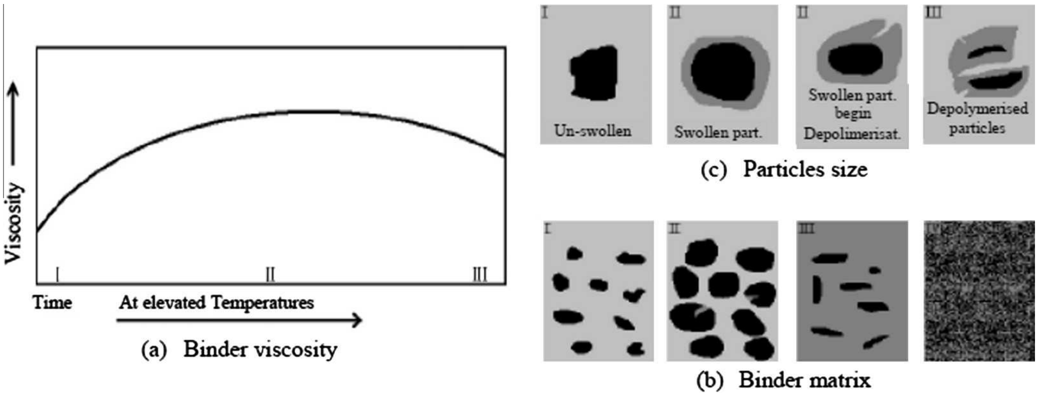

Fig. 1. Bitumen–RTR interaction phenomenon at elevated temperatures: change of properties over time. Adapted from [15].

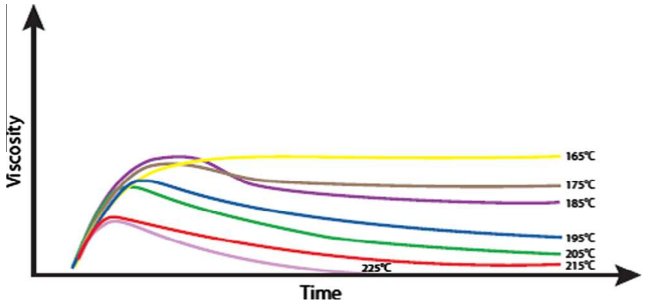

Fig. 2. Typical changes in viscosity values from RTR-MBs at different temperatures over time. Adapted from [23].

laboratory design of the blends and to improve the meaningfulness of the viscosity measurements.

1.2. Aim of the study

With this study the authors aim to design, calibrate and validate a tool which could help limiting phase separations issues of RTR–bitumen blends, so leading to a better mixing process and more realistic viscosity measurements. The new impeller helps to get a better distribution of the particles within the fluid by creating a convective like flow during the laboratory measurements (Fig. 3). As a consequence the rotational viscometer could be adapted to act as a low shear mixer of rubberised bituminous blends and will provide more realistic real-time viscosity measurements.

For these reasons, the authors have carried out the following investigation which includes the development, calibration and validation of an original dual helical impeller (DHI) for a rotational viscometer. The idea of this type of impeller has been investigated within research conducted in other fields [27,28] but the adaptation to the tyre rubber–bitumen blending process and the actual manufacturing and calibration of a stainless steel impeller for the specific equipment is an original work of this research. The study has been developed in two phases: a preliminary study, involving the rapid prototyping of the helical impeller and a visual control of its validity; secondly the manufacture of a stainless steel impeller followed by its calibration and validation to obtain more realistic viscosity measurements.

2. Dual helical impeller manufacture

2.1. Rapid prototyping

In order to understand the effect of the dual helical impeller (DHI) a preliminary study was performed by designing and manufacturing a plastic impeller to be used with a mixture of a transparent viscosity standard fluid and a high percentage of a ASTM 40# RTR. The plastic impeller has been designed with a dual helix in order to obtain a convective like flow within the sample. The manufacture was undertaken using the fused deposition modelling (FDM) technique, which is a rapid prototyping technique capable of realising nested features and texture in recessed surfaces which may not be realised by subtractive methodologies. Perhaps the most salient feature of this technique for prototyping work is the nominal cost of complexity. That is to say introducing new features or modifying designs has little or no cost implications apart from the time invested at the design stage. In this way designs can be rapidly modified allowing more iteration for experimental work. This allowed the mixer to be produced as one component without the need for assembly.

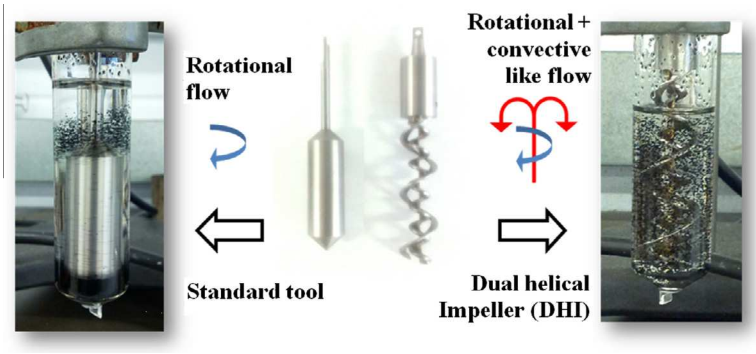

Fig. 3. The idea through comparison between the impellers and the generated flows during rotation.

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2.2. Preliminary visual assessments

The preliminary study consisted in a comparison of the distributions of ASTM 40# tyre rubber particles, within a transparent fluid of known viscosity, by using the plastic dual helical impeller and the Brookfield SC 27 (Fig. 4). A viscosity standard fluid was chosen with a viscosity of about 100 Pa s at 25 °C (1% accuracy), which is approximately the viscosity of a bitumen pen 40–60 at 160–180 °C. This represents the usual range of temperature at which the modification of bitumen with RTR is usually performed. The transparency of the fluid allowed the distribution of the particles within the blend to be monitored while the impeller was operating at different speeds: 10 RPM and 100 RPM. The study also involved the use of a standard steel impeller, Brookfield SC series no 27, with the same blend and rotation speeds. This allowed a visual comparison of the distribution of the particles within the blend by using the two impellers. The analysis was conducted at 25 °C by monitoring the blends for 15–20 min. This time represents the time used to achieve a sufficient thermal equilibrium all over the sample before viscosity measurements.

Fig. 4 shows the begin of the visual assessment at the point where rubber particles have been added to the fluid and the blend has been shake to favour a good distribution of the particles. In a second stage both the impellers have been immersed in the blend and viscometer was turned on to carry out a visual assessment at two different rotation speeds: 10 RPM and 100 RPM. Fig. 5 shows the distribution of the particles within the blend after 15–20 min of rotation. Results show that at 10 RPM both standard and DHI do not maintain particles in suspension. At 100 RPM, it is interesting to note how, due its shape, the standard impeller creates two layers of rubber on top and bottom. Thus, there is a clear evidence of phase separation in this instance. On the contrary, the DHI creates a convective like flow and it was visible how the inner thread raises up the rubber while the outer helps the particles to go downwards. As a result of this, particles are suspended and move vertically, this permits a homogeneous distribution of RTR crumbs all over the blend even after 20 min.

This preliminary study shows that rotational speed is an important factor in determining the efficacy of the impeller. Furthermore, the viscosity readings (not reported at this stage) are shown to be stable despite the shape of the impeller and the heterogeneity of the sample. Thanks to these encouraging results, the authors decided to proceed with the manufacture of a stainless steel DHI and its calibration to obtain reliable viscosity measurements.

2.3. DHI manufacturing

Fig. 6 shows the DHI manufacturing process which passed through an initial 3D design of the tool and then through, FDM rapid prototyping and the final manufacture in stainless steel. In fact, while FDM is a suitable technique for prototypes the current availability of robust materials compatible with this technique are limited. Due to the inherent requirement of the deposition methodology, the polymers used must exhibit a propensity to flow at relatively low temperatures; this in turn limits applicability of the manufactured product to high temperature tests. In the light of this issue and considering that the final application of the DHI should allow to use it to mix bituminous blend at over 150 °C, the second impeller was manufactured in stainless steel, similar to materials used in commercially available instruments. This second DHI was fabricated using a conventional machining approach. Due to the necessity of allowing a tool to engage with the work, the impeller could not be fabricated as one component and the design was modified to form an assembly of two helices. These were fabricated separately using a 4-axis machine tool in aluminium initially as a test cut and finally in 316L stainless steel for the final mixer. Electrical discharge machining (EDM) was also used to remove the core of the outer helix. This technique was selected to prevent distortion of the slender blade as a result of machining forces. The cost incurred in producing this impeller is significant with respect to producing the FDM mixer discussed earlier and hence producing low cost, proof of principal prototypes was a vital stage in this study.

3. Calibration procedure

The principle of operation of a rotational viscometer is to drive a impeller, immersed in the test fluid. Torque is transmitted through a calibrated spring. The viscous drag of the fluid against the impeller is measured by the spring deflection. Spring deflection is measured with a rotary transducer. Two Brookfield DV-II PRO Digital Viscometers, a low torque (LV model) and high torque (HA model) were used within this study. The two rotational viscometers differ mainly for the viscosity measurements range. The LV model is designed for low viscosity fluids (100% torque = 673.7 dyne cm), while the HA model is more appropriate for medium–high viscous fluid (100% torque = 14,374 dyne cm) [29]. Torque measurement accuracy specified by manufacturer is 1% of the full-scale range.

Two silicon standard viscosity Newtonian fluids were used for calibration in this study. The two calibration fluids are indicated

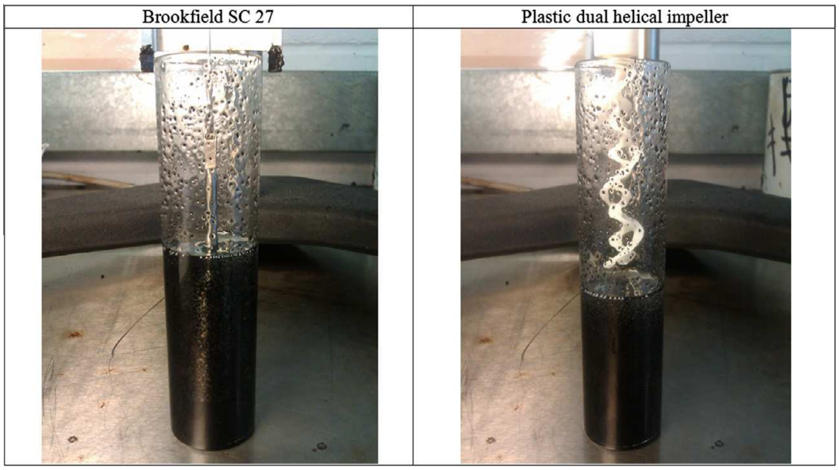

Fig. 4. Preliminary study: begin.

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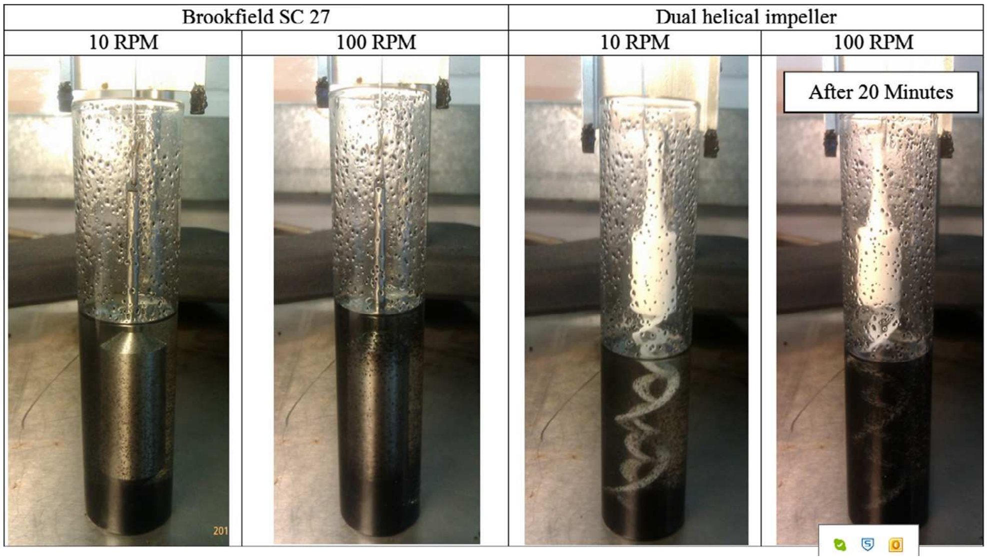

Fig. 5. Preliminary study: after 15 min (SC27), 20 min (DHI prototype).

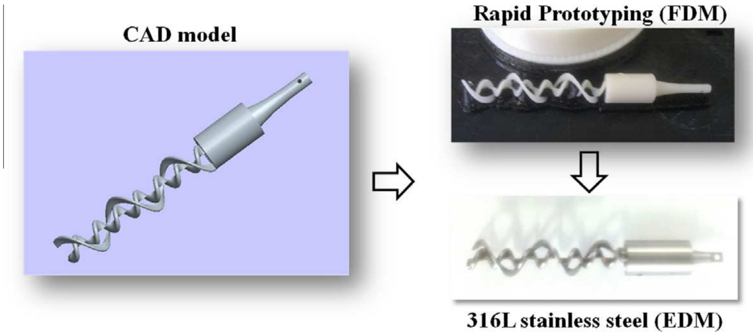

Fig. 6. DHI manufacturing process: design, rapid prototyping through FDM and final tool produced with EDM.

from now on as fluid A and B, with viscosities at 25 °C of 99.6 cP and 960 cP respectively. Viscosity measurements were carried out on both the standards fluids by using a Thermosel Impeller SC-27 and the new dual helical impeller in order to perform the calibration procedure. Once the new impeller was calibrated, viscosity measurements were carried out on 40/60 pen bitumen and on a RTR-MB.

3.1. Theoretical considerations

Bitumens are found to be non-Newtonian with viscosity values varying with the shear rate. This implies that the effective viscosity of the non-Newtonian fluid varies from point to point in the mixing vessel. Then the knowledge of the shear rate distribution is central to evaluate the viscosity of the fluid. But the complex flow field created by the helical impeller in the mixing vessel is not known a priori and does not allow to easily estimating the shear rate distribution. This issue may be ameliorated by defining an average shear rate value corresponding to an apparent viscosity value that can be determined as suggested by the Metzner and Otto method [30]. Within this simplified approach the average shear rate $\dot{\gamma}$ in the measuring vessel is assumed to be proportional to the impeller speed N and independent on the rheology of the fluid as shown in Eq. (1).

$$\dot{\gamma} = \mathrm{SRC} \cdot N \tag{1}$$

In Eq. (1) SRC is the shear rate constant to be determined for each impeller geometry.

The Brookfield viscometer automatically calculates the applied shear rate through Eq. (1). In particular an SRC value is associated with each impeller in order to calculate the shear rate, the shear stress and then the viscosity of the test fluids. If a non-standard impeller is used, a calibration procedure has to be performed to determine the corresponding SRC value. The mathematical expressions of the operating parameters found on various Brookfield viscometers are stated in Eqs. (2)–(4):

$$\dot{\gamma} = \frac{2\omega R_s^2 R_c^2}{x^2(R_c^2 - R_s^2)} \tag{2}$$

$$\tau = \frac{T}{2\pi R_s^2 L} \tag{3}$$

$$\eta = \frac{T}{\dot{\gamma}} \tag{4}$$

where w is the angular velocity of impeller (rad/s)[=(2π/60) N], N is the rotational speed (rpm), R_c is the radius of container (cm), R_s is the radius of impeller (cm), x is the radius at which shear rate is being calculated (cm), M is the torque input by instrument (N cm), L is the effective length of impeller (cm).

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The above mentioned equations apply to cylindrical geometries only. In particular Eq. (2) is currently used in the calibration procedure of non-standard impellers which conforms to cylinder or coaxial cylinder geometry. In this procedure the shear rate is first calculated by means of Eq. (2) for a certain impeller speed and then the SRC is calculated by applying Eq. (1). However, the same procedure cannot be applied for the dual helical impeller since it is not conformed to a cylindrical geometry. Therefore, an alternative procedure based on a robustness analysis to evaluate the appropriate SRC value for the new helical impeller, is proposed in the next section.

3.2. Robustness analysis

Viscosity measurements were carried out with both the standard fluids by using the standard impeller SC-27 provided by the Brookfield and the DHI. The tests were performed at a temperature of 25 °C while viscosity and torque values were measured with varying rotational speeds.

Measurements with the DHI were carried out with three different sets of measurements, each of them by setting a different SRC value. In particular the SRC value corresponding to the standard impeller SC-27, SC-28 and SC-29 have been chosen. The diameters of the three before mentioned impellers are respectively equal or smaller to the diameter of the external helix of the DHI. Figs. 6 and 8 show the measured viscosity values taken at different rotational speeds, respectively for fluid A and B. The average value and the mean square error (MSE) of the measured viscosity have been calculated for each set of measurement and for each viscometer. Results are detailed in Table 1. As previously discussed, the calibration procedure has been performed on both LV and HA viscometers. Results are shown to be comparable, therefore only those related to the LV viscometer are here reported.

Figs. 6 and 8 show also that the SRC value of 0.28 provides the best approximation to the true viscosity value for both the tested fluids. Furthermore, the corresponding average value of the measured viscosity is close to the true one and the mean square error is rather small as shown in Table 1. Therefore the SRC value of 0.28 has been chosen as the shear rate constant of the DHI.

4. Validation of results

4.1. Measurements on a 40/60 bitumen

Once the DHI was calibrated and the most reliable SRC had been determined, viscosity measurements were carried out on a 40/60 pen bitumen at two test temperatures of 135 °C and 177.5 °C. The first temperature is identified by the ASTM D4402 standard as the level at which characterising the viscosity of a bitumen at elevated temperatures. The latter is the temperature suggested by the standard specification for Asphalt Rubber (ASTM D 6114-97), to assess the apparent viscosity of the binder, and it is also the temperature that has been used within this study to adapt the viscometer as a low shear mixer (§4.3). Fig. 9 shows the results of the viscosity measurements at different rotational speeds by using both the SC-27 and the DHI impellers.

4.2. Viscosity measurements on recycled tyre rubber modified bitumen

After the positive tests performed on the standard viscosity fluids and on neat bitumen, the DHI has been used to measure the apparent viscosity of a RTR-MB containing 15% of 30# RTR and produced in high shear. The tests have been performed in both LV and HA models of rotational viscometers and compared with measurements undertaken with the SC-27 impeller. According to the Standard specification for Asphalt Rubber (ASTM D 6114-97), the apparent viscosity measurement of the modified binder has to be performed at 177.5 °C with a rotational speed of 12 RPM for LV models and 20 RPM for the HA model. Measurements have produced similar results with both viscometers, so for the sake of simplicity Fig. 10 reports only the results obtained with LV viscometer. From Fig. 7, it can be observed that SC-27 impeller provides measurements which pass through a wider transition period before achieving a stable measurement, while DHI allows measurements with a more stable trend. This could be explained by the capability of the DHI to create a convective flow within the sample which allows reducing the initial effort needed to accelerate the bitumen–rubber blend from a stationary position to a uniform shear speed.

It is well known that for non-homogenous fluids subjected to turbulent flow, the shear rate can affect viscosity measurements. Furthermore, considering the eventual use of the DHI as impeller to produce RTR-MB, by adapting the rotational viscometer as a low shear mixer, also measurements at higher rotational speed (50 and 100 RPM) have been performed.

Due to the expected higher torque, only the HA model was used in this phase. Fig. 11 shows that the transition period toward a stable measurement is similar for all the combination of speed and impellers. However, the initial effort needed to accelerate the bitumen–rubber blend is reduced when the DHI is used. Furthermore, it has to be noticed that DHI shows almost uniform results also changing shear speed, while SC-27 impeller shows higher susceptibility to the variation of the rotational speed. At last it has to be highlighted, that at both high speeds DHI provides a lower apparent viscosity value which is 40% less of that measured by using the standard SC-27 impeller.

4.3. Adapting the rotational viscometer as a low-shear mixer by using the DHI

A previous study [25], has demonstrated the possibility of adapting a Brookfield rotational viscometer as a low shear mixer. This practical protocol allows constant monitoring of the viscosity of the binder, with accurate control of the temperature, and it offers the opportunity of understanding what is physically occurring during the process by monitoring the key parameter, rotational viscosity. Furthermore, the protocol offers the chance

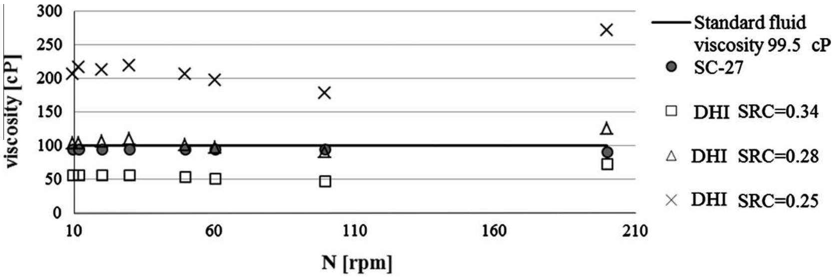

Fig. 7. Viscosity measurements by using LV viscometer on standard fluid 99.5.

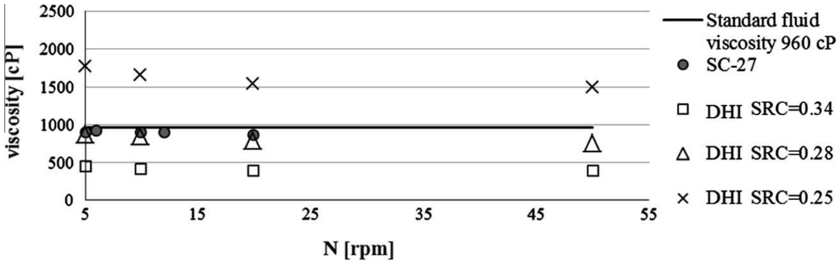

Fig. 8. Viscosity measurements by using LV on standard fluid 960.

Table 1
MSE and average value of the measured viscosity for fluid A and fluid B.

Fluid A	SC-27 SRC = 0.34	DHI SRC = 0.34	DHI SRC = 0.28	DHI SRC = 0.25	Fluid B	SC-27 SRC = 0.34	DHI SRC = 0.34	DHI SRC = 0.28	DHI SRC = 0.25
MSE	0.004	0.66	0.005	0.25	MSE	0.004	1.502	0.025	0.216
Average	93.7	54.42	98.93	200.86	Average	910.427	437.175	855.8129	1803.204

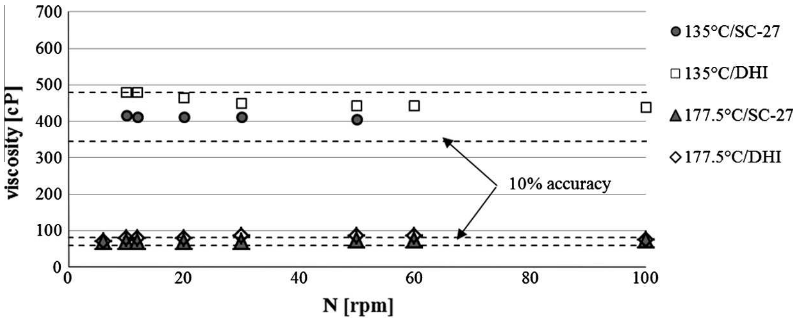

Fig. 9. Viscosity measurements by using LV on a 40/60 bitumen at T = 135 °C and 177.5 °C.

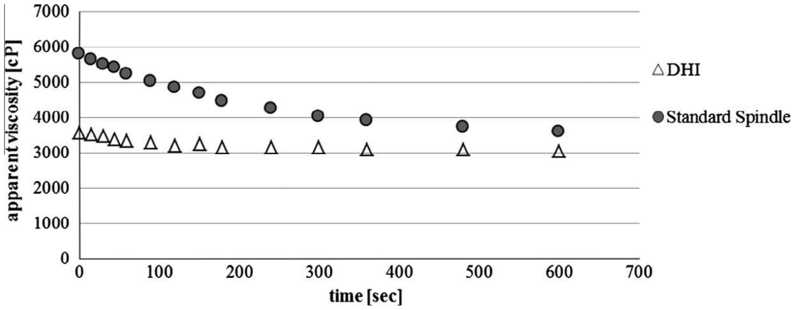

Fig. 10. Apparent viscosity of a TR-MB at 177.5 °C by using LV viscometer at 12 rpm.

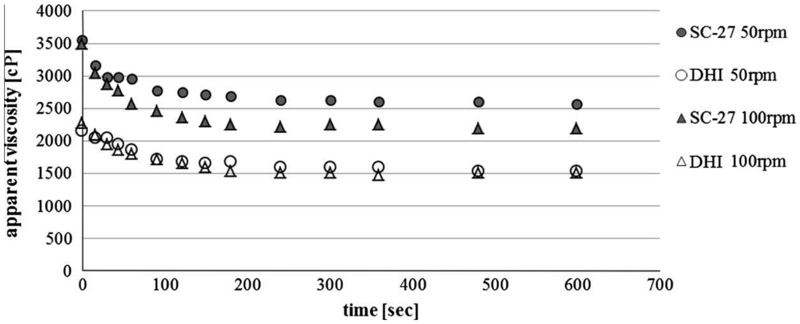

Fig. 11. Apparent viscosity of a TR-MB at 177.5 °C by using HA viscometer at 50–100 rpm.

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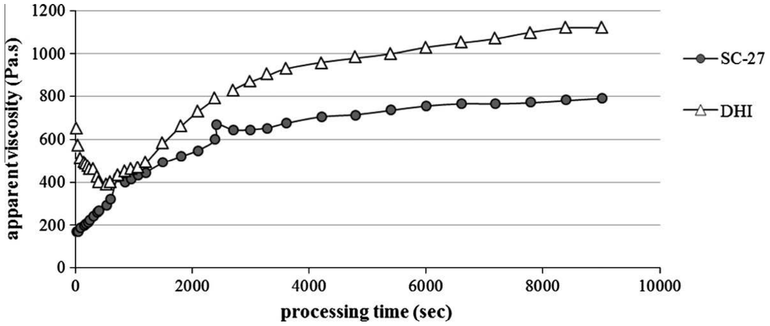

Fig. 12. Results of constant monitoring using the Brookfield viscometer as a low shear mixer at 177.5 °C.

to study the change in rheology of RTR-MBs blended with different base binders, or different TR content, by drastically reducing the material and time consumption. It is to be noticed that the procedure allows producing a maximum of 15 g of modified bitumen for further tests. This quantity is not enough to perform conventional tests like penetration, but it results to be sufficient in order to perform Dynamic Mechanical Analyses (DMA) with a DSR. However, the adaptation of a Brookfield viscometer with standard impellers is not trivial. In fact, as shown before in Fig. 5, the particular shape of the SC series impeller causes a phase separation with some of the rubber particles settling down and most of them accumulating on the top of the sample. This phenomenon does not allow having a homogenous distribution of rubber particles within the binder and so moving the impeller up and down during the mixing, is necessary to help distribution of the rubber particles.

Fig. 12 shows the curve of the real-time apparent viscosity measurements produced by mixing a 40/60 pen base bitumen with 15% of 30# RTR crumbs (max diameter = 0.5 mm). The mixing process has been performed at 177.5 °C, and 100 rpm for both SC-27 and DHI impellers. A total time of 2 h and 30 min has been necessary to obtain the peak viscosity with both impellers. Analysing the curve obtained by using the standard impeller, peaks are present at about 1000 and 2500 s. These are due to the manual lifting of the impeller to prevent the accumulation of rubber particles on top of the sample. This is clear evidence of phase separation which occurs when the impeller SC-27 is used to produce TR-MBs. It has to be noticed that this manual moving of the impeller could lead to damage of the equipment.

Mixing bitumen with RTR by using the DHI shows that the helical impeller is a feasible solution for optimising the laboratory production of RTR-MB in low shear. In fact, there was no need of moving the impeller during the mixing because no accumulation of rubber particles on the top of the sample took place. However, DHI was raised as well at 1000 s and 2500 s, but no evident changes in viscosity have been noticed because the particles were well distributed throughout the sample. Furthermore, Fig. 9 shows that the viscosity value obtained with the DHI is always higher that those obtained with the SC-27. This is in contrast with what has been shown before and it possibly due to the better distribution of rubber particles within the binder which leads to a higher rate of RTR involved in the viscosity measurements and/or wider extent of modification. These results represent an even stronger evidence of the phase separation occurring by mixing bitumen and rubber with the SC-27 impeller and show how a different solution is needed to adapt the rotational viscometer as a low shear-mixer but also to obtain more-realistic viscosity values for RTR-MBs.

5. Conclusions

In this study, a dual helical impeller (DHI) was designed and manufactured in order to adapt the Brookfield (rotational) viscometer as a low shear mixer and to guarantee more realistic viscosity measurements of samples which contains suspended particles. The DHI geometry was designed to create a convective like flow within the sample which allows the uniform distribution of suspended solids within a low viscosity fluid. The results have highlighted that:

- Layer based manufacturing techniques are shown to be a convenient method to for rapid prototyping of complex geometries for surface–fluid interaction. This method also brings with it the benefits of reduced cost of testing and accelerated lead times.
- The robustness analysis, conducted as calibration procedure for low viscosity and high viscosity Brookfield viscometers, and the validation of results showed that using the DHI with a shear rate constant of 0.28 (impeller 28), provides reliable viscosity measurements of homogenous fluids.
- DHI provides similar results than the cylindrical standard impeller (SC-27) only with the neat bitumen. In fact, by testing the non-homogenous binder, RTR-MB, the DHI showed always lower values of apparent viscosity but with a more stable trend. This could be explained by the capability of the DHI to create a convective like flow within the sample which allows reducing the initial effort needed to accelerate the bitumen–rubber blend from a stationary position to a uniform shear speed.
- DHI is a feasible and improved solution for optimising the laboratory production of RTR-MB by adapting the rotational viscometer as a low-shear mixer.
- Further studies are necessary to better understand the nature of the convective like flow created by the DHI and to optimise its shape with regards to different viscosities range. However, results obtained in this study show that an impeller with a dual helical shape could leads to more realistic viscosity measurements of any fluids with suspended particles.

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Please cite this article in press as: Lo Presti D et al. Toward more realistic viscosity measurements of tyre rubber–bitumen blends. Constr Build Mater (2014), http://dx.doi.org/10.1016/j.conbuildmat.2014.03.038

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