NEW HELICOIDAL SPINDLE FOR MIXING AND VISCOSITY MEASUREMENTS OF TYRE RUBBER MODIFIED BINDERS

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ABSTRACT

The use of modified bituminous binders, in place of pure bitumen, is a common practice to enhance performance of road pavements. The measurement of rheological properties of the modified binders is often challenging due to the presence of suspended solids. Phase separation during the course of measurements makes viscosity of non-homogeneous blends, such as tyre rubber modified bitumens, difficult to ascertain. In this study, a new spindle was designed and manufactured to be used with a rotational viscometer in order to adapt the viscometer as a low shear mixer and to guarantee reliable viscosity measurements of samples which contain suspended particles. This helps to optimize the modification process of tyre rubber modified bitumen by having a reliable real-time viscosity measurement. Spindle geometry was optimised to create a convective like flow within the sample and so minimise phase separation. A calibration exercise was carried out to determine the value of a geometry dependent shear rate constant for the new spindle. Development of prototype spindles was undertaken using a layer based manufacturing technique which allows the rapid realisation of a design which could not be achieved in another fashion. In this case nested features are created in a prototype to verify the design before a fully functional prototype is produced using conventional 4-axis machining. The new spindle allows the Brookfield viscometer to be used as a low shear mixer of small volumes of bitumen-rubber blends while allowing real-time reliable measurements of the viscosity.

Keywords: Rotational viscometer, Dual Helicoidal spindle, Bitumen rubber, rapid prototyping

1. INTRODUCTION

The modification of pure bitumen with various kinds of modifiers is a common practice which has engaged researchers in the field of pavement materials for the last 30 years. The control of processing conditions is a key aspect to the optimisation of the final product. The temperature of the fluid during mixing is a critical factor. Furthermore, viscosity variation during the modification process is an easily determined parameter that is often used to monitor the modification process and to assess its progress, especially for tyre rubber modified binders obtained through the standard MacDonald process [1]. For this reason the authors used a Brookfield viscometer as a low shear mixer for tyre rubber bitumen blends. The Brookfield viscometer offers the opportunity of mixing a small amount of tyre rubber and bitumen, with an accurate control of temperature, and with a real-time measurement of the apparent viscosity. This helps to optimise a tyre rubber bitumen blend by using many different processing conditions but consuming only a small amount of the material. During experimentation with this fluid, phase separation often occurs during mixing. Tyre rubber particles tend to float onto the surface, thus requiring a manual movement up and down of the spindle to keep a good distribution of the particles within the blend. Of course, this practice also affects the viscosity measurements. Brookfield viscometers can be equipped with special accessories and spindles that can help with the measure of viscosity of the blend with suspended particles, such as vane spindles or a “Helipath stand” equipped with a T-bar spindle [2]. However, none of these spindles are able to evenly distribute the particles through the sample volume and therefore enable the viscometer to be used as a rheo-mixer. Based on research conducted in other fields [3,4], this study investigates the manufacture and calibration of an original spindle that is able to create a convective like flow within the sample allowing a good distribution of suspended particles while also providing reliable viscosity measurements. The success of this research could lead to the adaption of the viscometer as a mixer of bitumen with tyre rubber, but also as a reliable viscosity measurement technique for any fluid with suspended solids.
The study has been divided in two phases: a preliminary study, involving the rapid prototyping of a helicoidal spindle and a visual control of its validity; and, secondly, the manufacture of a stainless steel dual helicoidal spindle followed by its calibration and validation to obtain reliable viscosity measurements.

2. DUAL HELICOIDAL SPINDLE MANUFACTURE

In order to understand the effect of the dual helicoidal spindle (DHS), a preliminary study was performed by designing and manufacturing a plastic spindle to be used with a mixture of a transparent viscosity standard fluid and a high percentage of ASTM 40# tyre rubber. The plastic spindle 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 prototype 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 iterations for experimental work. This allowed the mixer to be produced as one component without the need for assembly.

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 40/60 pen bitumen at 160-180°C. This represents the usual range of temperature at which the modification of bitumen with tyre rubber is usually performed. The transparency of the fluid allowed the distribution of the particles within the mixture to be monitored while the spindle was operating at different speeds: 10 RPM and 100 RPM. The study also involved the use of a standard steel spindle, Brookfield SC series n°27, with the same blend and rotation speeds. This allowed a visual comparison between the distribution of the particles within the blend by using the two spindles.

2.1 Preliminary assessments

As discussed above, the preliminary study consisted of a comparison of the distributions of ASTM 40# tyre rubber particles, within a transparent fluid of known viscosity, by using the manufactured dual helicoidal spindle (DHS) and the Brookfield SC-27. The analysis was conducted at 25°C by monitoring the blends for 15-20 minutes. This time represents the time used to achieve a sufficient thermal equilibrium all over the sample before viscosity measurements. Figures 1 and 2 show the results of the analysis that has been conducted at two different rotation speeds of 10 RPM and 100 RPM.

Results in Figure 2 show that at 10 RPM both standard and dual helicoidal spindles do not maintain particles in suspension. At 100 RPM, it is interesting to note how, due its shape, the standard spindle creates two layers of rubber at the top and bottom providing clear evidence of phase separation in this instance. On the contrary, the dual helicoidal spindle creates a convective like flow with the inner thread raising up the rubber while the outer helps the particles go downwards. As a result of this, particles are suspended and move vertically, thus permitting a homogeneous distribution of tyre rubber all over the blend even after 20 minutes.

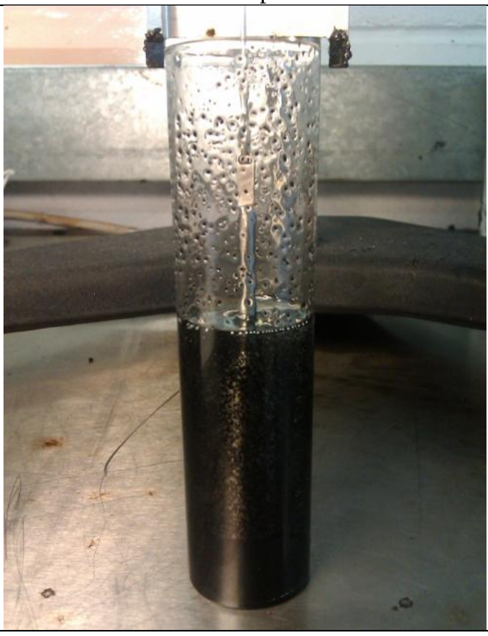
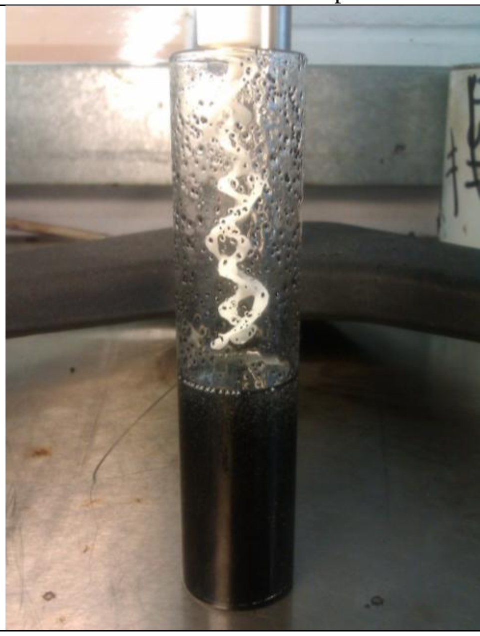
Brookfield spindle 27	Plastic dual helicoidal spindle
	

Fig 1. Preliminary study: prior to mixing

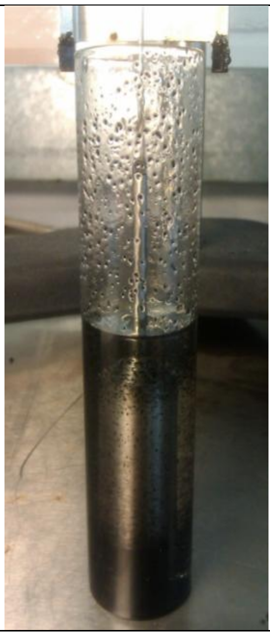
Brookfield spindle 27		Dual helicodial spindle	
10 RPM	100 RPM	10 RPM	100 RPM
			

Fig 2. Preliminary study: after 15-20 minutes mixing

This preliminary study shows that rotational speed is an important factor in determining the efficiency of the spindle. Optimisation of spindle shape may be undertaken to improve performance at a given speed. The viscosity readings are shown to be stable despite the original shape of the spindle and the heterogeneity of the sample. Thanks to these encouraging results, the authors decided to proceed with the manufacture of a stainless steel spindle and its calibration to obtain reliable viscosity measurements.

2.2 DHS manufacturing

While FDM is a suitable technique for prototypes, the current availability of robust materials compatible with this technique is limited. In addition, due to the inherent requirement of the deposition methodology that the polymers used must exhibit a propensity to flow at relatively low temperatures, the applicability of the spindle at high temperatures is limited. For this reason a second spindle was manufactured in stainless steel, similar to materials used in commercially available instruments. The second mixer was fabricated using a conventional machining approach. Due to the necessity of allowing a tool to engage with the work the mixer could not be fabricated as one component and the design was

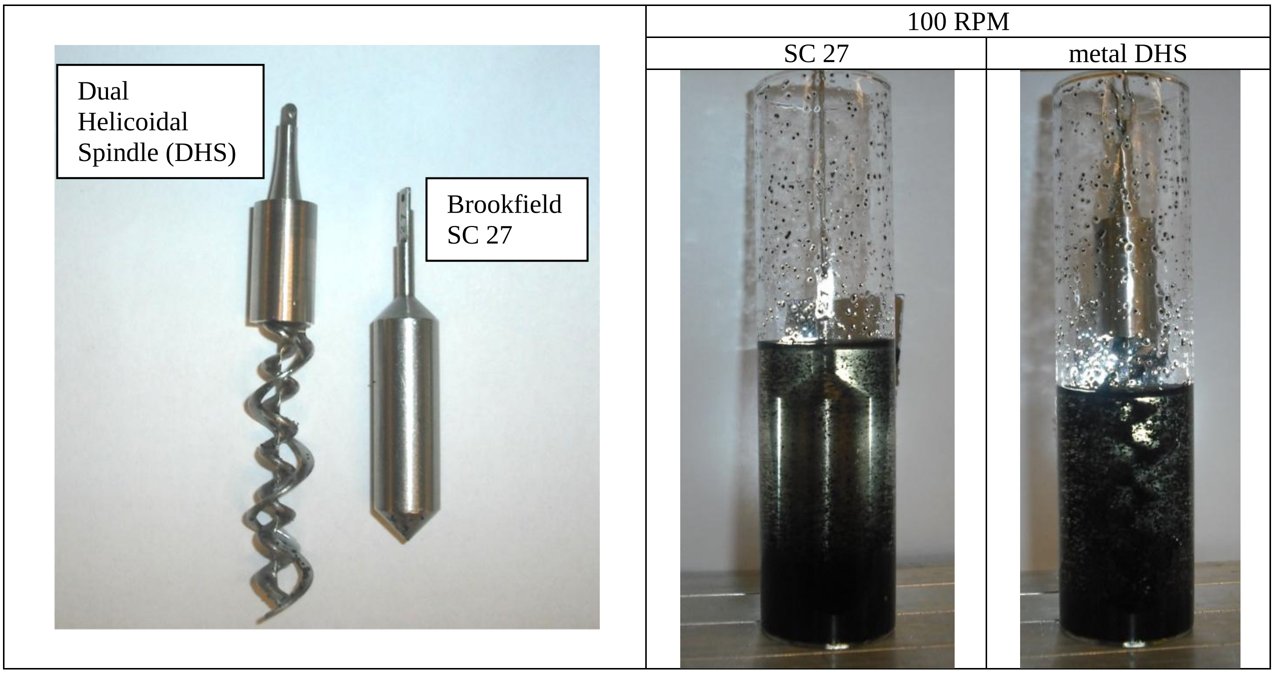


Fig. 3. Steel DHS and its effect of keeping particles suspended after 15-20 minutes of mixing

modified to form an assembly of two helices as shown in Figure 3. 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 mixer 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 spindle, immersed in the test fluid. Torque is transmitted through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. Two Brookfield DV-II PRO Digital Viscometers, a low viscosity (LV) and high viscosity (HA) were used within this study. The two rotational viscometers differ mainly for the viscosity measurement range. The LV is designed for low viscosity fluids (100% torque = 673.7 dyne·cm), while the HA is more appropriate for medium-high viscous fluid (100% torque = 14374 dyne·cm) [5]. 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 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 Spindle SC-27 and the new Dual Helical Spindle in order to perform the calibration procedure. Once the new spindle was calibrated, viscosity measurements were carried out on 40/60 pen bitumen and a tyre rubber modified bitumen.

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. Therefore knowledge of the shear rate distribution is central to evaluate the viscosity of the fluid. However, the complex flow field created by the helical impeller in the mixing vessel is not known a priori and does not allow easy estimation of the shear rate distribution. This issue may be ameliorated by defining an average shear rate value corresponding with an "apparent" viscosity value that can be determined as suggested by using the Metzner and Otto method [6]. 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 of the rheology of the fluid as shown in Eq.1.

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

Where *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 spindle in order to calculate the shear rate, the shear stress and then the viscosity of the test fluids. If a non-standard spindle 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. 2,3 and 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 spindle (rad/sec)[=(2π/60) N]
N is the rotational speed in RPM
R_c is the radius of container (cm)
R_s is the radius of spindle (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 spindle (cm)

The above mentioned equations apply to cylindrical geometries only. In particular Eq.2 is currently used in the calibration procedure of non-standard spindles which conform to cylinder or coaxial cylinder geometries. 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 helicoidal spindle since it does not conform to a cylindrical geometry. Therefore, an alternative procedure based on a robustness analysis was used to evaluate the appropriate SRC value for the new helical spindle as detailed in the next section.

3.2 Robustness analysis

Viscosity measurements were carried out with both the standard fluids by using the standard Brookfield spindle SC-27 and the DHS. The tests were performed at the temperature of 25°C and viscosity and torque values were monitored at different rotational speeds.
Measurements with the DHS 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 spindle SC-27, SC-28 and SC-29 have been chosen. The diameters of the three spindles are respectively equal or smaller than the diameter of the external helix of the DHS. Figures 4 and 5 show the measured viscosity values taken at different rotational speeds in RPM, respectively for fluids A and B. The average value and the mean square error (MSE) of the measured viscosity have been calculated for each set of measurements and for each viscometer. Results are detailed in Table 1. As mentioned before, the calibration procedure has been performed on both LV and HA viscometers. Results have been shown to be comparable, therefore only those related to the LV viscometer are reported here for brevity.

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

FLUID A	SC-27 SRC=0.34	DHS SRC=0.34	DHS SRC=0.28	DHS SRC=0.25
MSE	0.004	0.66o	0.005	0.250
AVERAGE	93.7	54.4	98.9	200.9

FLUID B	SC-27 SRC=0.34	DHS SRC=0.34	DHS SRC=0.28	DHS SRC=0.25
MSE	0.004	1.502	0.025	0.216
AVERAGE	910.4	437.2	855.8	1803.2

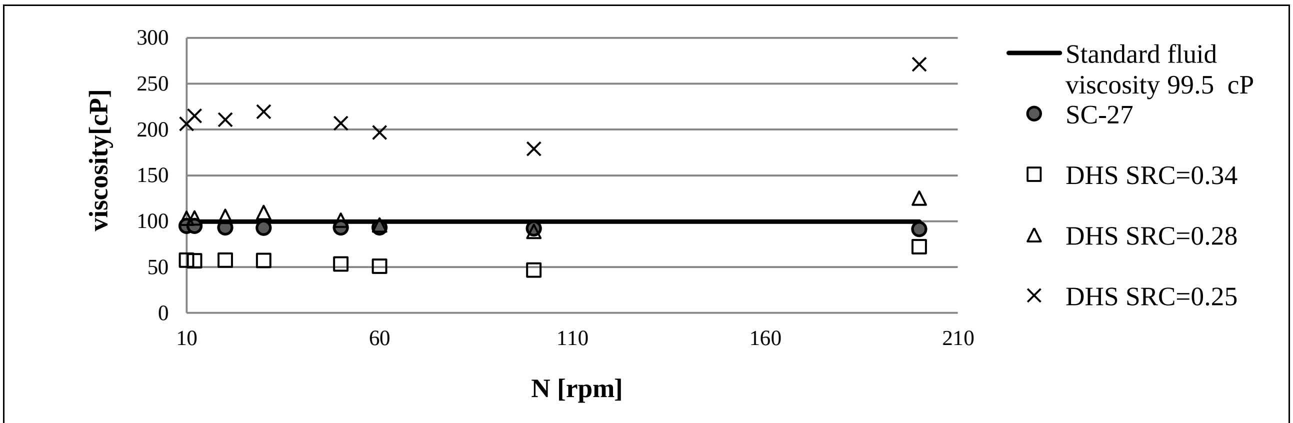


Fig. 4 Viscosity measurements by using LV viscometer on standard fluid 99.5 cP

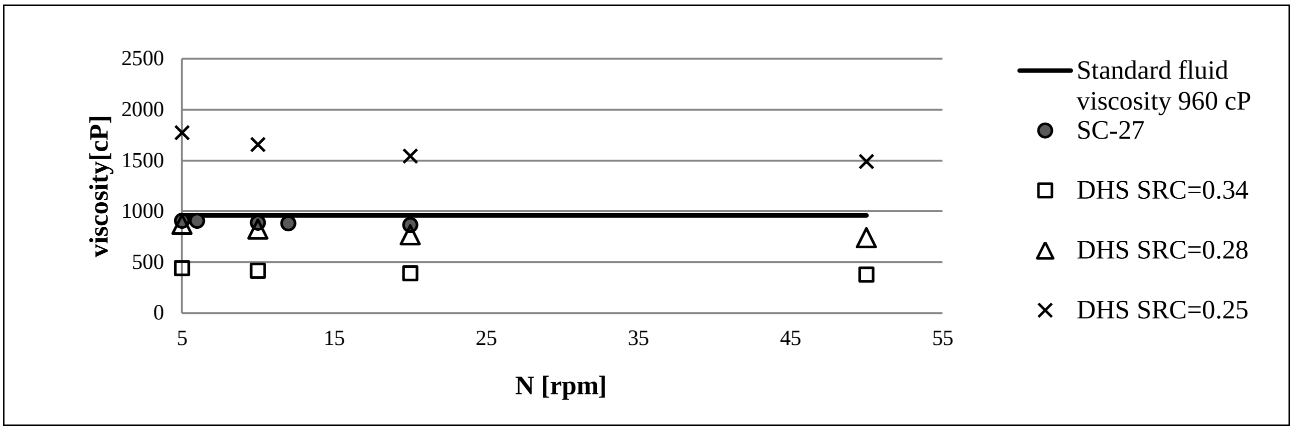


Fig. 5 Viscosity measurements by using LV on standard fluid 960 cP

Figures 4 and 5 show 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 DHS.

4. VALIDATION OF RESULTS

4.1 Measurements on a 40/60 pen bitumen

Once the DHS has been calibrated and the most reliable SRC determined, viscosity measurements have been 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 to characterise 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 (see Section 4.3). Results shown in Figure 6 show viscosity measurements reported at different rotational speeds for both the SC-27 and the DHS spindles. Measured values do not differ more than 10%.

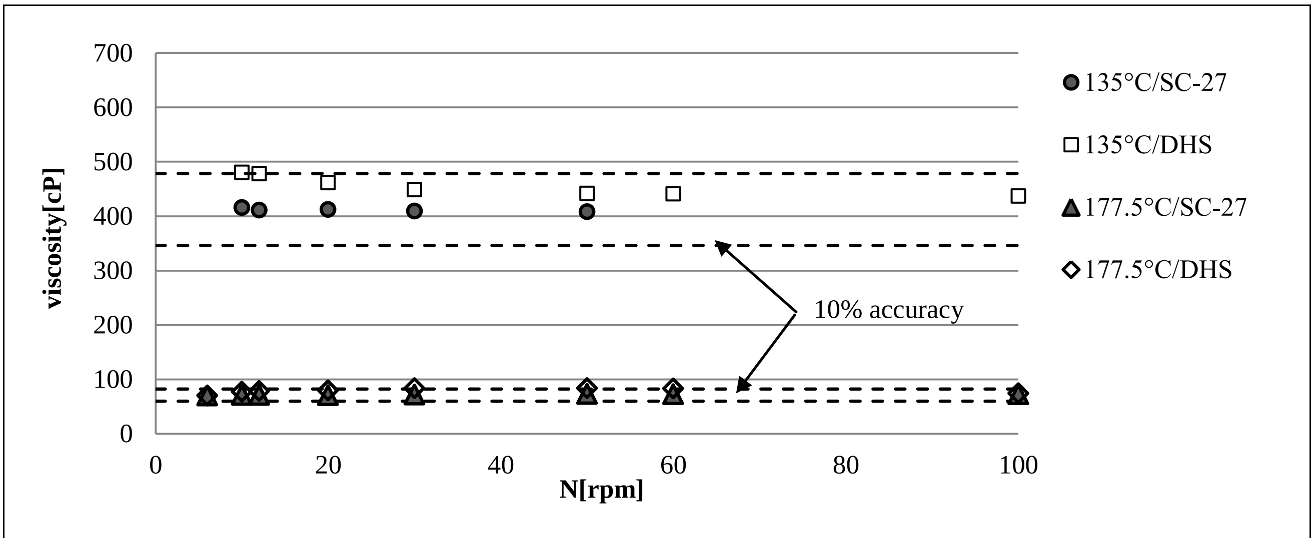


Fig. 6 Viscosity measurements by using LV on a 40/60 pen bitumen at T=135°C and 177.5°C

4.2 Viscosity measurements on Tyre Rubber Modified Bitumen

Apparent viscosity of a TR-MB containing 15% of 30# TR and produced in high shear, has been measured. Both LV and HA models of rotational viscometers and both SC-27 spindle and the new DHS, have been used. 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. For the sake of simplicity, Figure 7 reports only the results obtained with the LV viscometer. From Figure 7, it can be observed that the SC-27 spindle gives higher values of viscosities while the DHS allows measurements with a more stable trend. This could be explained by the capability of the DHS to create a convective flow within the sample which reduces the initial effort needed to accelerate the bitumen-rubber blend from a stationary position to a uniform shear speed.

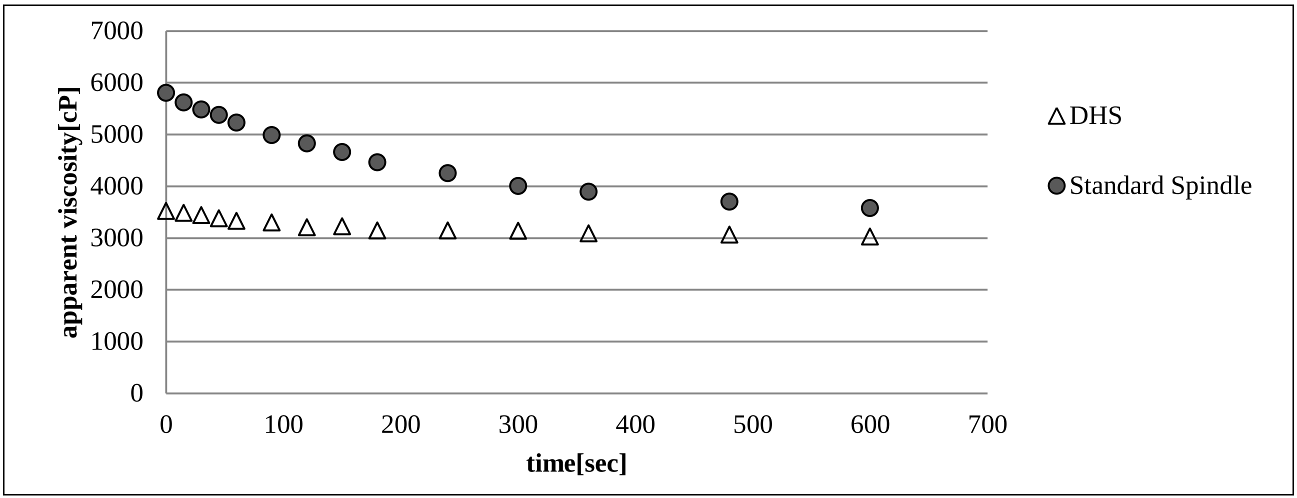


Fig. 7 Apparent viscosity of a TR-MB at 177.5°C by using LV viscometer at 12 RPM

It is well know that especially for non-homogenous fluids and in presence of turbulent flow, the shear speed can affect the viscosity measurement. Furthermore, considering the eventual use of the DHS as an impeller to produce TR-MB, by adapting the rotational viscometer as a low shear mixer, measurements at higher shear speed (50 and 100 RPM) have also been performed.

Due to the expected higher torque, the HA model was used in this phase. Figure 8 shows that an initial effort is needed to accelerate the bitumen-rubber blend for all the combinations of speed and spindles. However, using the DHS reduces the gap between the initial and the stable apparent viscosities. Furthermore, it has to be highlighted that DHS shows almost uniform results against changing shear speed, while SC-27 spindle shows higher shear speed susceptibility. Finally, it should be noticed that with higher shear speeds, apparent viscosity values of a TR-MB tested with the DHS are always lower than those measured by using the standard SC-27 spindle.

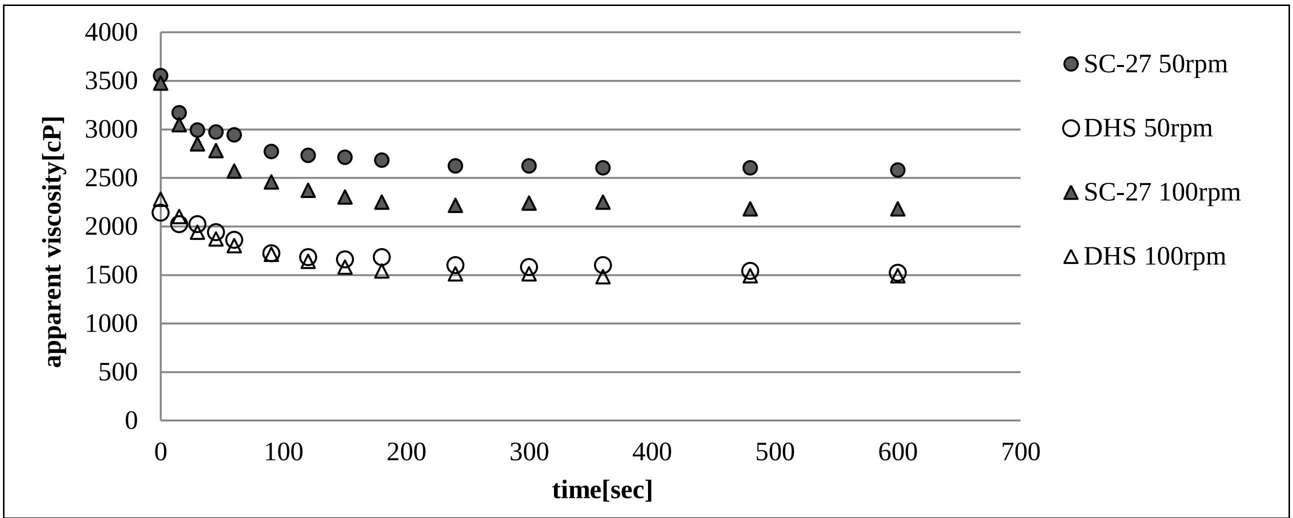


Fig. 8 Apparent viscosity of a TR-MB at 177.5°C by using HA viscometer at 50 – 100 RPM

4.3 Mixing TR-MB with a rotational viscometer

A previous study [7], has demonstrated the possibility of adapting a Brookfield rotational viscometer as a low shear mixer. This practical protocol allows the 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. Many studies have demonstrated that the modification process is mainly time-temperature dependent, but also that applied shear has almost negligible effects on the rheology of the modified binders [8,9]. Therefore, the protocol offers the chance to study the change in rheology of TR-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 only allows the production of a maximum of 15 grams of modified bitumen for further tests. This quantity is not enough to perform conventional tests like penetration, but it is sufficient to perform Dynamic Mechanical Analyses (DMA) with a DSR.
However, the adaptation of a Brookfield viscometer with standard spindles is not without its problems. Figure 2 shows that the particular shape of the SC series spindle causes a phase separation with some of the rubber particles settling down and most of them accumulating on the top of the sample.

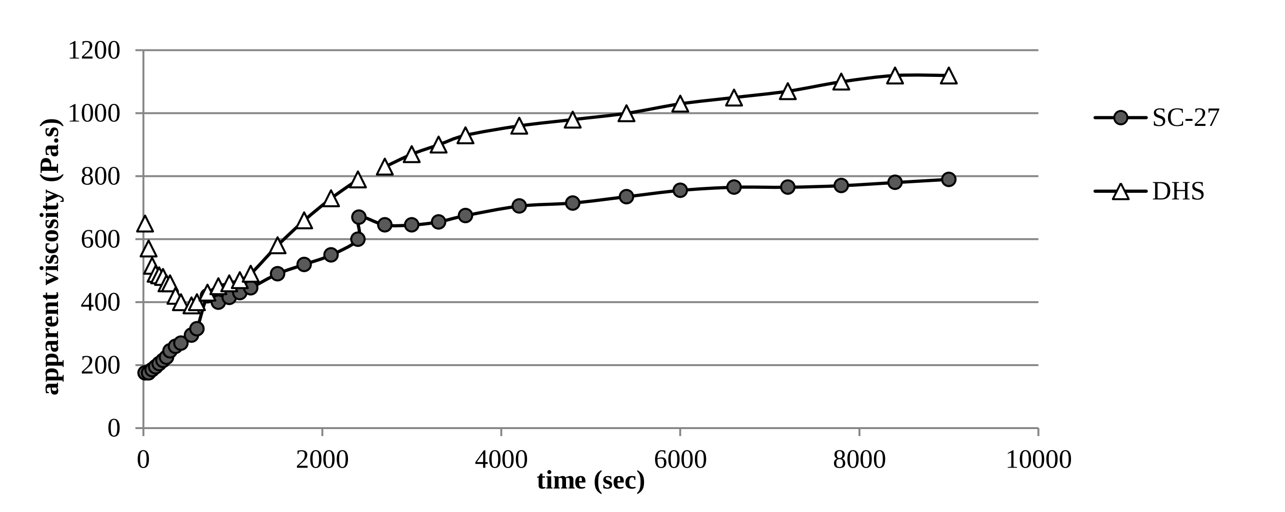


Fig. 9 Results of constant monitoring using the Brookfield Viscometer as a low shear mixer at 177.5°C

This phenomenon does not allow an even distribution of rubber particles within the binder and so moving the spindle up and down during the mixing, is necessary to help distribution of the rubber particles. Figure 9, shows the curve of the real-time apparent viscosity measurements produced by mixing a 40/60 pen bitumen with 15% of 30# TR crumbs (max diameter = 0.5 mm). The mixing process has been performed at 177.5ºC, and 100 rpm for both SC-27 and DH spindles. A total time of 2 hours and 30 minutes has been necessary to obtain the peak viscosity with both spindles. Analysing the curve obtained by using the standard spindle shows strange peaks present at about 1000 and 2500 seconds. These are due to the manual raising of the spindle and due to the accumulation of rubber particles on top of the sample. This is clear evidence of phase separation which occurs when the spindle SC-27 is used to produce TR-MBs. It has to be noticed that this manual moving of the spindle could also lead to damage of the equipment.

Mixing bitumen with tyre rubber by using the DHS shows that the helicoidal impeller is a feasible solution for optimising the laboratory production of TR-MB in low shear. In fact, there was no need to move the spindle during the mixing because there is no accumulation of rubber particles on the top of the sample. However, DHS was raised as well at 1000 sec and 2500 sec, but no evident changes in viscosity have been noticed. Furthermore, Figure 9 shows that the viscosity values obtained with the DHS are 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 proportion of tyre rubber involved in the viscosity measurements. This is therefore further evidence of the phase separation occurring by mixing bitumen and rubber with the SC-27 spindle.

5. CONCLUSIONS

In this study, a dual helicoidal spindle (DHS) was designed and manufactured in order to adapt the Brookfield (rotational) viscometer as a low shear mixer and to guarantee reliable viscosity measurements of samples which contain suspended particles. DHS geometry was designed to create a convective like flow within the sample. This allowed a better distribution of suspended solids within low viscous fluids. A layer based manufacturing technique was shown to be a very convenient method to produce complex shaped spindle samples before manufacturing the more expensive stainless steel spindle. The robustness analysis, conducted as a calibration procedure for Low viscosity and High viscosity Brookfield viscometers, has shown that using the DHS with a shear rate constant of 0.28 (spindle SC-28), provides reliable viscosity measurements. Validation of these results has been performed by testing a 40/60 pen bitumen and a tyre rubber modified bitumen at different torque levels (shear rates). Results show that DHS provides similar results to that of the cylindrical standard spindle (SC-27) for the neat bitumen. When the non-homogenous binder, tyre rubber modified bitumen (TR-MB), was tested, the DHS showed lower values of apparent viscosity but with a more stable trend. This could be explained by the capability of the DHS to create a convective like flow within the sample which allows a reduction of the initial effort needed to accelerate the bitumen-rubber blend from a stationary position to a uniform shear speed. Finally, DHS has been used as an impeller to produce small amounts of laboratory TR-MB by adapting the viscometer as a low shear mixer. From the evaluation of the obtained real-time apparent viscosity curve, and comparing it with the one obtained by using the SC-27, it is possible to affirm that the DHS is a feasible and improved solution for optimising the laboratory production of TR-MB in low shear. Further studies are necessary to better understand the nature of the convective like flow created by the DHS and to optimise its shape with regards to different viscosity ranges. However, results obtained in this study show that a spindle with a dual helicodial shape could lead to reliable measurements of fluids with suspended particles.

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