INTRODUCTION

Premature failure of fuel oil distribution pumps supplying fuel oil to support coal burners on coal-fired power plants in South Africa raised concerns due to the high cost of replacement of these pumps in addition to unacceptable downtime on power generating capacity [1].

Depending on the power station, fuel oil supplied per tender has to comply with one of three grades, i.e. Grade 1, 2 or 3, differing with regard to origin and characteristics, i.e. crude oil based, synthetic (coal-based), viscosity, sulphur content and nature of the fuel oil burners [2].

Grade 1: A light to medium distillate fuel.
Grade 2: A blend of ± 80% medium to heavy distillate fuel (typically crude oil derived diesel) and ± 20% residual fuel (typically crude oil derived bitumens).
Grade 3: A blend of ± 40% medium to heavy distillate fuel (typically crude oil derived diesel) and ± 60% residual fuel (typically crude oil derived bitumens).

In an earlier investigation [3], it was shown that, due to the large variation in characteristics of fuel oils used, the standard method of separating particles from the fluid via filtration through a 0,8 micron filter according to ASTM D 6217 [4], could not be used. Similarly, performing the standard lubricity test as applicable for standard diesel samples [5], [6], was also not sufficient.

It was proposed to find a method based on a standard laboratory-based lubricity test that can be used to positively identify the cause of wear-based failures involving fuel oils due to either:

(i) Presence of abrasive particles in the fuel oil. (These particles could be of a submicron size.)
(ii) Lack of lubricity performance of the fuel oil

The challenge was to find a way to separate any particles from the as-received fuel oil and then to perform a lubricity test on the samples with and without particles.

Use of the Complementary Rating (CR) method [7], taking the indicators of abrasion inside the wear scar into account in addition to the Wear Scar Diameter (WSD), was proposed as the key to identifying the source of wear.

The objective of the current investigation was to expand the range of fuel oil samples and to perform the tests in triplicate in order to confirm that the proposed method can indeed be used.
Experimental and method

A sample of ten (10) fuel oils, representative of the three grades of fuel oils supplied by current suppliers, was used. Each determination was performed in triplicate.

The sequence of actions that had to be executed was:
(i) Run a standard lubricity test [5],[6] on the sample as received, using the HFRR-apparatus.
(ii) Record coefficient of friction (COF), percentage film formation (%Film), Complementary Rating (CR) and obtain an image of the wear scar using optical microscopy.
(iii) Separate all particulate matter from the fuel oil.
(iv) Repeat steps (i) & (ii), but this time on the particle-free sample.
(v) For the particulate matter, obtain the particle size distribution (PSD) and analyse for species present.

When performing the HFRR lubricity test, the recorded and measured performance characteristics as described in (ii) above, provide information on the friction and wear performance of the sample under test. The difference in HFRR results obtained before separation and after separation of any particulate matter, determines the success of the test procedure.

High-speed centrifugation using a Hermle Z323 centrifuge with a maximum rotational speed of 17,000 rpm. was used to separate the particles from the fuel oil samples. High clarity 100ml conical shaped polypropylene tubes were used in the centrifuge. A mixture of 50/50 (v/v) of fuel oil (FO) and hexane (BP=68 °C) was made by starting with a 40 ml sample of the as-received fuel oil, which was then diluted with 40 ml of hexane. This sample was thoroughly mixed using an ultrasound mixer. Small adjustments were made by adding hexane and weighing the centrifuge tubes to ensure a balanced pair in the centrifuge. Centrifugation of each sample was done at room temperature for 45 minutes.

After centrifugation, the supernatant liquid was decanted from the particulate matter in the bottom of the centrifuge tubes.

Before HFRR-testing of the supernatant, the hexane was evaporated by slowly heating the mixture in a heating bath, while continuously stirring with a magnetic stirrer, until the sample volume was reduced to 50% of the original mixture, ie 40 ml. This was done to ensure that no physical or chemical changes are caused by thinning down the sample with hexane.

The residue, containing the particles, was used to perform the particle size analysis and the elemental analysis.

The particle size analysis was done using a Malvern Zetasizer Nano ZS machine with a measuring range of 0.3 nm – 10,000 nm. All measurements were done in triplicate. The sample preparations and measurements were performed at room temperature. For the determination, 1 ml of each of the Grade 1 samples was diluted with 20 ml of hexane in a glass beaker. Because of the very dark brown colour of the Grade 2 and Grade 3 samples, 1 ml of each sample was diluted with 40 ml of hexane. This was done because of the light-scattering measurement principle of the Zetasizer and to ensure that light can pass through the samples for the measurements. The lighter the sample, the more it allows light to pass through the glass cuvette which was filled with the sample and placed into the measuring cell of the Zetasizer.

The elemental analysis of the particles of each oil sample was done using inductively coupled plasma (ICP) - optimal emission spectroscopy (OES) on a Spectro Arcos apparatus supplied by Ametek Materials Analysis Division. The samples were prepared by following the wet ashing method with sulfuric acid (H2SO4). 2 g of each sample was accurately weighed and mixed with 2 ml of concentrated sulfuric acid in a platinum crucible.

The samples were then placed in a furnace where the temperature was gradually increased to 550 °C. This temperature was maintained for 2 hours, which was followed by gradual cooling of the ashed samples. The resulting ashes were dissolved in 1.5 ml of concentrated nitric acid (HNO3), which was further diluted with 40 ml of distilled water. The final samples were mixed and the ICP analysis performed on each sample.
Results

**HFRR friction and wear plots**

In Table 1 below a summary of results obtained during the HFRR lubricity tests is presented.

ViscSpec = The required kinematic viscosity range of the relevant fuel oil grade at 40°C, just for information.

Film% = Averaged extent of film formation over the test duration. Low value can be indicative of a weak film & possible high wear, while a high value can be indicative of protective film formation.

WSD = Wear Scar Diameter, where WSD = (WSD_X + WSD_Y)/2, where WSD_X and WSD_Y are the measured wear scar diameters in the X-direction and the Y-direction respectively, on the outside of the wear scar.

WSD_{1.4} = This is the WSD corrected to a standard vapour pressure of 1.4 kPa

CR = Complementary Rating [7], a visual rating of the inside of the wear scar, based on a scale of 1 – 6.

PreCentr = Results for samples as-received, i.e. pre-centrifugation.

PostCentr = Results for samples after centrifugation, without particulates, i.e. post--centrifugation.

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**Table 1: Summary of HFRR results (significant differences are marked in red)**

The numbers shown in Table 1 should be seen as broad indicators only, highlighting major differences, since recorded values over the test duration of 75 minutes are averaged for each of three repeat runs per sample.

After each HFRR run, the wear scar diameters of the HFRR-balls were measured by means of an optical microscope set at 10X magnification, equipped with a Zeiss Axiocam ERc 5s digital camera. The photographs of the ball scars are shown in Figure 1 below. Note the following observations:

(i) Wear scar images for the triplicate runs of the three different fuel oil grades were consistent and showed a general decrease in size moving from Group 1 to Group 3.

(ii) The WSD’s of the PreCentr results and those of the PostCentr results, were the same within the constraints of the test method, with one exception, namely that of Sample 6, where the WSD’s for the PostCentr runs were larger than those for the PreCentr runs.

(iii) Differences like scratches and abrasion marks inside the wear scar, as indicated by the Complementary Rating (CR) values between the PreCentr and PostCentr results were all similar with one exception, namely that of sample Sample 10, where the PreCentr results show severe abrasion marks inside the wear scar, while the PostCentr results show essentially no sign of abrasion inside the wear scar, confirming that the source of wear can be attributed to abrasive particulate matter.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Pre-Centr Run 1</th>
<th>Pre-Centr Run 2</th>
<th>Pre-Centr Run 3</th>
<th>Post-Centr Run 1</th>
<th>Post-Centr Run 2</th>
<th>Post-Centr Run 3</th>
</tr>
</thead>
<tbody>
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<td>Sample 1 GR1</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
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<td><img src="image6.png" alt="Image" /></td>
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<td><img src="image8.png" alt="Image" /></td>
<td><img src="image9.png" alt="Image" /></td>
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<td><img src="image19.png" alt="Image" /></td>
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<td><img src="image35.png" alt="Image" /></td>
<td><img src="image36.png" alt="Image" /></td>
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<td><img src="image41.png" alt="Image" /></td>
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<tr>
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<td><img src="image44.png" alt="Image" /></td>
<td><img src="image45.png" alt="Image" /></td>
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<td>Sample 9 GR3</td>
<td><img src="image49.png" alt="Image" /></td>
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<td><img src="image59.png" alt="Image" /></td>
<td><img src="image60.png" alt="Image" /></td>
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</tbody>
</table>

*Figure 1: HFRR wear results (ball scars)*
**Particle size analysis**

A key element to the success of the investigation depended on the assumption that abrasive particles could be the cause of damage – especially if they were of submicron size. In Figure 2 below, the Particle Size Distribution (PSD) of the particles obtained after separation via centrifugation is presented.

![Fuel oil particle size distributions](image)

**Figure 2 Particle Size Distribution of samples**

It is noteworthy that Sample 8 contained a significant amount of particulate matter between 1 – 2 nm, while the other samples had particle size distributions varying between 30 and 6 500 nm. This confirms the fact that, should abrasive particulate matter be the source of wear, these could be of a size that normal filtration systems are not catering for and that this should be a consideration when preventative measures are considered.

**ICP analysis of particles**

Particles were analysed using ICP-analysis. The samples were combusted in a furnace to eliminate all carbon-containing components and then dissolved in a nitric acid solution. The results obtained are shown in Table 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>V (mg/l)</th>
<th>Ni (mg/l)</th>
<th>Na (mg/l)</th>
<th>Fe (mg/l)</th>
<th>Mn (mg/l)</th>
<th>Mg (mg/l)</th>
<th>K (mg/l)</th>
<th>S (mg/l)</th>
<th>Cr (mg/l)</th>
<th>Ca (mg/l)</th>
<th>Al (mg/l)</th>
<th>P (mg/l)</th>
<th>Zn (mg/l)</th>
<th>Si (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1 GR1</td>
<td>&lt; -0.185</td>
<td>&lt; -0.060</td>
<td>2.156</td>
<td>0.166</td>
<td>0.008</td>
<td>0.572</td>
<td>0.446</td>
<td>4.992</td>
<td>&lt; -0.075</td>
<td>6.026</td>
<td>0.572</td>
<td>&lt; -0.093</td>
<td>0.211</td>
<td>0.202</td>
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<tr>
<td>Sample 2 GR1</td>
<td>&lt; -0.178</td>
<td>&lt; -0.045</td>
<td>2.377</td>
<td>&lt; -0.025</td>
<td>0.009</td>
<td>0.516</td>
<td>0.26</td>
<td>1.64</td>
<td>&lt; -0.095</td>
<td>1.47</td>
<td>&lt; -0.015</td>
<td>&lt; -0.073</td>
<td>0.163</td>
<td>0.157</td>
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<tr>
<td>Sample 3 GR1</td>
<td>&lt; -0.188</td>
<td>&lt; -0.061</td>
<td>1.802</td>
<td>0.103</td>
<td>&lt; -0.002</td>
<td>0.495</td>
<td>0.295</td>
<td>2.724</td>
<td>&lt; -0.074</td>
<td>3.284</td>
<td>0.26</td>
<td>&lt; -0.091</td>
<td>0.161</td>
<td>0.175</td>
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<tr>
<td>Sample 4 GR1</td>
<td>&lt; -0.189</td>
<td>&lt; -0.060</td>
<td>1.932</td>
<td>0.157</td>
<td>0.001</td>
<td>0.515</td>
<td>0.28</td>
<td>2.289</td>
<td>&lt; -0.066</td>
<td>3.358</td>
<td>0.057</td>
<td>&lt; -0.006</td>
<td>0.201</td>
<td>0.164</td>
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<tr>
<td>Sample 5 GR1</td>
<td>&lt; -0.191</td>
<td>&lt; -0.055</td>
<td>1.976</td>
<td>0.193</td>
<td>0.002</td>
<td>0.501</td>
<td>0.333</td>
<td>3.011</td>
<td>&lt; -0.071</td>
<td>3.4</td>
<td>0.397</td>
<td>&lt; -0.094</td>
<td>0.16</td>
<td>0.171</td>
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<tr>
<td>Sample 6 GR2</td>
<td>0.169</td>
<td>0.156</td>
<td>1.712</td>
<td>0.343</td>
<td>0.001</td>
<td>0.475</td>
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<td>&gt;33.580</td>
<td>0.039</td>
<td>&gt;26.556</td>
<td>&gt;108.877</td>
<td>3.071</td>
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<td>Sample 8 GR3</td>
<td>0.548</td>
<td>0.155</td>
<td>1.943</td>
<td>0.187</td>
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<td>0.49</td>
<td>0.289</td>
<td>&gt;12.300</td>
<td>&lt; -0.090</td>
<td>1.465</td>
<td>0.217</td>
<td>0.476</td>
<td>0.343</td>
<td>0.211</td>
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<tr>
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<td>0.073</td>
<td>4.074</td>
<td>0.075</td>
<td>0.001</td>
<td>0.532</td>
<td>0.376</td>
<td>3.747</td>
<td>&lt; -0.093</td>
<td>1.547</td>
<td>0.301</td>
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<td>Sample 10 GR3</td>
<td>0.038</td>
<td>0.148</td>
<td>3.339</td>
<td>0.115</td>
<td>&lt; -0.002</td>
<td>0.663</td>
<td>0.292</td>
<td>3.415</td>
<td>&lt; -0.097</td>
<td>2.287</td>
<td>0.712</td>
<td>0.61</td>
<td>0.789</td>
<td>0.229</td>
</tr>
</tbody>
</table>

**Table 2: Elemental analysis per sample**

Table 2 shows the measured concentrations of 14 elements in the separated particulate matter of each fuel oil. What can be noted from these results is that the heavier fuel oils (Grade 3 and Grade 2) show presence of Ni and V, while the light oils (Grade 1) show no presence of Ni and V. Sample 7 and Sample 8 show the highest S-content. Sample 7 interestingly, shows the highest content of all the elements analysed for, yet its tribological behaviour does not seem to be affected by this. This needs further investigation.
Discussion

In this study it was shown that a simple laboratory-based lubricity test could be used as a means to identify the origin of wear. Wear can be due to abrasive particles present in the fuel oil, or it could be due to lack of lubricity of the fuel oil. Particles can be separated via centrifugation after dilution with a suitable solvent (hexane) and then the lubricity test can be repeated on the particle-free sample after evaporation of the solvent. The inside of the wear scar, as quantified by the Complementary Rating (CR) and supplemented by a microscope image, are key components when wear is studied and complements the WSD. Particle Size Analysis (PSA) confirmed the presence of submicron particles, while chemical analysis of the elements (ICP) in the particulate matter ensured quantitative information.

Conclusion

The initial objective, namely to adapt a simple laboratory friction and wear test to identify the origin of wear performance was achieved. Separating abrasive particles from the fuel oil was a key element of this.

The role of asphaltenes, which are present in some of the fuel oil samples, and which are known to have unique characteristics affecting the behaviour of fuel oils, were not investigated. This should be considered.

ACKNOWLEDGMENTS

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REFERENCES


KEYWORDS
Applied Tribology; Power Generation; Boundary Lubrication, Test Methods, Maintenance; Cleanliness