WEAR CHECK
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We are ready for more soot

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'Get Ready for More Soot' is the title of an article written by Drew Troyer of the Noria Corporation for publication in *Practising Oil Analysis*. It deals in part with the effect that current and proposed environmental legislation would have on the performance of engine lubricants. This Technical Bulletin takes a close look at soot, why it is a problem, why is it becoming more of a problem and how to measure its presence in oil.

Both the Montreal and (more recently) Kyoto protocols dictate that exhaust emissions from internal combustion engines must be reduced and target levels and dates for meeting these levels are stipulated. Various countries around the world also have their own legislation regulating exhaust emission and pollution levels. Although South Africa has recently become a signatory of the Kyoto protocol, it is not subject to the environmental legislation of other countries.

Emissions are dealt with through engine design.

This, however, does not mean we do not have a problem on our hands that needs to be dealt with. The major and most effective way of dealing with exhaust emissions is through engine redesign. In effect, we are going to put the pollutants in the oil instead of the atmosphere. South Africa buys nearly all its diesel engines and equipment that is powered by them from countries that have very strict environmental legislation so we are subject to the engine technology of America, England, Germany and Japan.

One of the two major pollutants that need to be reduced is soot, a particulate consisting of almost pure carbon resulting from the incomplete combustion of any hydrocarbon fuel. In an ideal world the burning of petrol or diesel would produce nothing but water and carbon dioxide. In the real world, petrol and diesel are not pure compounds and no combustion process is 100% efficient. The end result is soot, which is what turns diesel engine oil black within a very short period of time.

Pollutants now end up in the oil.

The other major pollutant is a family of gases known as nitrogen oxides, usually referred to as NOx, which results from chemical reactions in the combustion chamber. NOx is linked to the formation of acid rain and the depletion of the ozone layer, whilst soot and other particulate emissions can cause a wide range of pulmonary and respiratory health problems.

Unfortunately, with recent engine technology, the steps taken to reduce one would increase the other and vice versa. An increase in engine combustion temperature would ensure more efficient combustion and reduce the amount of soot generated but would increase the amount of NOx produced. Likewise a reduction in combustion temperatures coupled with the timing being retarded would reduce the NOx levels but increase soot levels and exhaust smoke.

The American EPA (Environmental Protection Association) has imposed standards that will reduce NOx emissions in 2004 by 50% compared with 1998 and to decrease particulate matter by 43 000 tons per annum. The engine technology is available to achieve this but where will all that soot go? Correct, in the oil.

Soot particles are vanishingly small when they are generated in the combustion process. Typically they are nearly spherical in shape and are between 1/100th and 1/20th of a micron in diameter (10 - 50 nanometers). Through various physical processes these particles agglomerate or stick together and the average soot ‘group’ is about 0.078 micron in size which is well below the filtration level of most standard engine oil filters. Engine oil contains chemicals called dispersants that help keep a certain amount of this soot in suspension and the rest of it ‘disappears’ out the exhaust pipe and into the atmosphere.

In order to reduce NOx levels, combustion temperatures have been reduced and injection timing retarded with a subsequent increase in the production of soot. This has led to more soot being generated in the combustion process, more soot that now has to end up in the oil instead of the environment.

One recent development in engine design that reduces particulate emissions is Exhaust Gas Recirculation, better known as EGR. Engines designed to meet the most restrictive of environmental regulations employ EGR whereby a certain percentage of the exhaust gases are sent back to the combustion chamber creating a multi-pass opportunity for the soot to end up in the oil. An EGR valve regulates how much of the exhaust is recirculated, typically 10 - 20% under load and about 70% at idle.

So what effect does this extra soot loading have on the oil and, in turn, the engine? The most obvious effect that soot has on the oil is to increase its viscosity, that is, high soot concentrations reduce the ability of the oil to flow easily. This results in increased wear on start up (where the majority of engine wear takes place), increased friction and operating temperature, and a reduction in fuel economy. Unrelated to an increase in viscosity, the likelihood of dispersancy failure, fouling and deposits is also increased. So, high soot concentrations cause plenty of problems for both the oil and the engine.

EGR technology is relatively new to South Africa but it is not the only aspect of engine design that needs to be considered in terms of soot levels in the oil. Fuel injection timing, combustion chamber design, ring position, engine type, scavenge efficiency, air cleaner restriction, air/fuel ratio, low compression, excessive idling, lugging, etc., can and will affect the amount of soot ending up in the oil.

All this legislation and new engine technology has made it necessary for the oil companies to formulate new engine lubricants and to do this far more quickly than has been done in the past. Oils can be formulated with more dispersants, which are chemicals that keep soot in suspension to retard viscosity increase and stop deposits forming on engine parts. However, there is a limit to how much of an additive can be put in an oil before other properties become affected. It must also be remembered that additives are sacrificial in that once they have done their job, that's it, they cannot be regenerated to do that job a second time. Synthetic and severely hydrotreated oils (API base oil groups II, III and IV) all show a resistance to thickening (viscosity increase) under conditions of high soot loading so this may also be used to control the problem.

Soot can be more abrasive than dirt.

It is interesting to note that most people tend to think of soot, carbon black, exhaust smoke, that 'stuff' around a candlewick or whatever you want to call it, as being 'soft and crumbly'. It's not! Soot is harder and more abrasive than dirt. In fact, carbon in its other form, although not strictly related, is what diamonds consist of.

A recent study showed that as long as the dispersancy of the oil was okay and the soot was kept in suspension, then there did not appear to be any systematic link between soot concentration and engine wear rate. What did appear significant was that when the soot loading of the oil exceeded the oil's ability to carry that amount of soot, then wear rates did increase.

All this means that being able to measure the amount of soot in a used engine oil, is now more vitally important than ever before. There are a number of ways that soot concentration can be measured in used oils - the blotter method, the insolubles test, light extinction measurement, thermogravimetric analysis and infrared analysis. Wearcheck has always measured soot levels, either by blotter method or, since 1990, by Fourier Transform Infrared analysis (FTIR). This method has been chosen over others because of its speed, cost, accuracy and ease of operation in a condition monitoring application.

There has, however, been one problem with the way in which Wearcheck expresses soot contamination levels. The number reported is merely an index, it is not a measurement in grams per millilitre or % by mass which many people are a lot more comfortable with. The reason for the soot index is historical and comes from certain laboratory equipment manufacturers, a particular engine manufacturer and the United States military.

Because increased soot levels in engine oils dramatically affect all the players, engine manufacturers and oil formulators alike, Wearcheck has developed a soot reading that everyone is intuitively comfortable with and that is soot by percentage mass. This does not mean that the soot index is in any way inaccurate; in fact the method currently employed by Wearcheck has been put to the ASTM (American Society of Testing and Materials) for ratification as an internationally accepted method for the FTIR analysis of used lubricating oil.

INSTRUMENT CALIBRATION

As mentioned earlier Wearcheck has historically provided the soot loading of a sample as a soot index. This number is derived from the FTIR spectrum. This instrument relies on the absorbance of infrared light by the species of interest. i.e. sulphate, NOx, etc. However, soot doesn't absorb infrared light, it disperses it. This has the effect of raising the baseline of the FTIR trace. Because of this, soot is measured by the decrease in the transmittance of infrared light at 2000cm^{-1}. This wavelength was chosen, as there are no other absorbing species in this area (Figure 1).

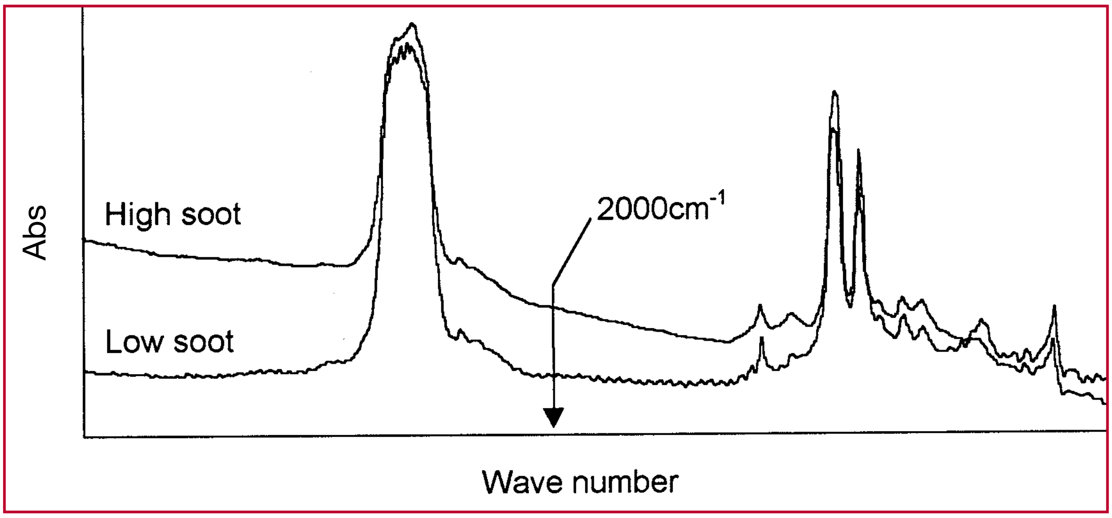

Figure 1: Soot wave length

Soot is measured in percentage mass.

Although the soot index is a useful indicator of the soot loading of oil it is effectively a dimensionless number. It was therefore decided to investigate the relationship between the Wearcheck soot index and the actual percentage by mass of the soot in oil.

The generally accepted method to determine this is ASTM E1131-98, a very costly and time-consuming process. Essentially a very small sample of oil, typically 20 milligrams, is placed on a very sensitive balance, in an enclosed furnace. The sample is then heated at a controlled rate in stages and the change in the weight recorded. The first stage involves heating the sample under an inert atmosphere such as nitrogen up to 600°C. At this point it is assumed any oil and additives will be vaporised. The gas is then switched to air and the temperature raised to 750°C. It is at this stage that everything combustible, i.e. soot, will burn off. All that remains at the end of the process is ash and non-combustibles, bearing in mind that soot is essentially pure carbon and will burn to produce only carbon dioxide gas.

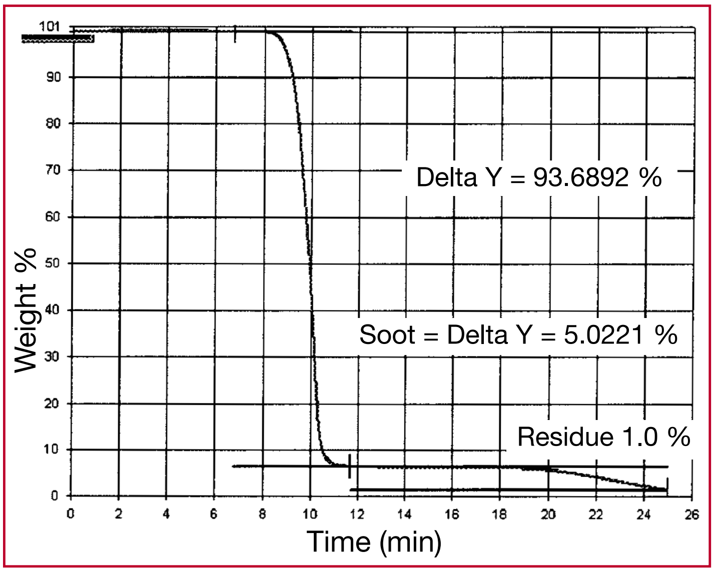

Figure 2: Soot percentage

The weight loss versus temperature/ time profile (Figure 2) is then examined and the percentage soot calculated.

In order to get the correlation data that was needed, two centres of excellence were approached, the Department of Polymer Chemistry at the University of Stellenbosch and The Cummins Test Centre (CTC) in the United States, and a set of samples was sent to each. These samples were analysed in duplicate by thermogravimetric analysis (TGA). Additionally CTC ran the samples through their FTIR instrument and these results were also compared.

The results are shown overleaf in figures 3 and 4, and on page 6 in figure 5.

As can be seen from the results there

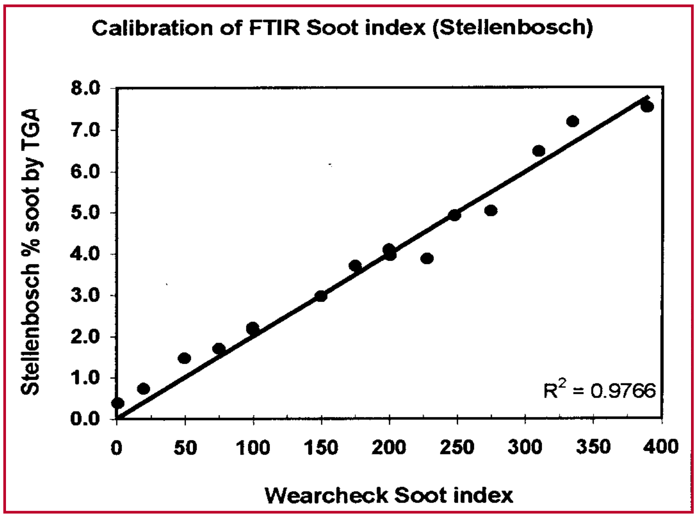

Figure 3: Comparison of soot index: Stellenbosch TGA vs Wearcheck FTIR

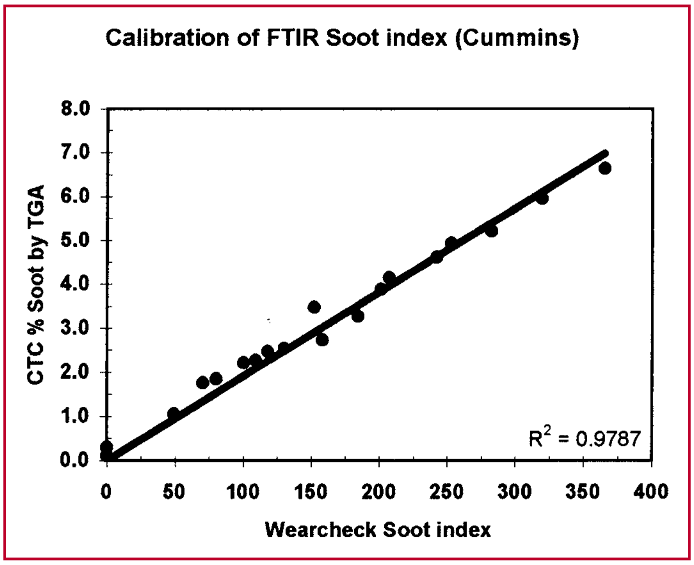

Figure 4: Comparison of soot index: Cummins TGA vs Wearcheck FTIR

Wearcheck's results were matched by two external laboratories.

was an excellent and linear relationship between the Wearcheck soot index and the mass % soot in the oil. It was especially pleasing that both external test laboratories returned nearly identical relationships.

Additionally it can be seen in Figure 5 that there is excellent agreement between the FTIR instrument at the Cummins test centre and that of the Wearcheck instrument.

Other than cost and ease of application, the FTIR method has also been shown to be more reproducible than the TGA method, which requires samples to be run in duplicate in order to produce reliable results.

This confirms that using FTIR for the routine analysis and determination of soot loading of used engine oils is a sound practice and that Wearcheck is also able to offer a fast and low cost % soot by mass that is traceable to ASTM E1131-98. However, due to the nature of the method, which relies on infrared radiation passing through the sample, once the soot content reaches a level where this is compromised, the results will become unreliable. This is generally when the soot loading reaches about 8%.

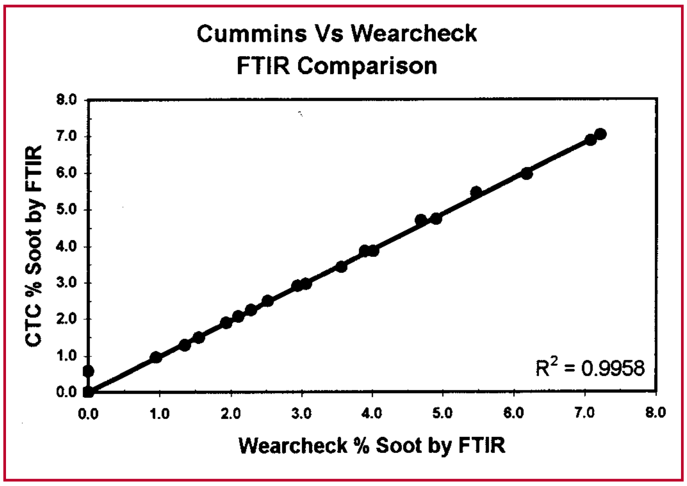

Figure 5: FTIR comparison: Cummins vs Wearcheck

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John Evans is the diagnostic manager: mobile equipment and Neil Robinson is technical manager for the Wearcheck Division of Set Point Technology.

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