High Performance Lubricants for Conventional Medium Density Compaction

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Abstract

The development of more challenging PM parts goes hand in hand with improved lubricants formulations. As parts become more complex and thus more difficult to eject, lubricants need to perform at unachieved levels. Furthermore, environmental concerns tend to limit the usage of metallic stearates, which offered great lubrication properties. Meanwhile, the desired properties of a performing lubricant remain. Lubricants should be able to maintain the compressibility of powder mixes, offer good flow ability and die-filling performance, facilitate the ejection of long and tall parts and provide a good surface finish while burning without leaving soot marks.

In this article, the ejection behaviour of several innovative lubricant formulations is described. The lubricants are compared to conventional PM lubricants on an industrial press at high shear stresses and under different compaction conditions. It is demonstrated that some composite wax lubricants can achieve excellent ejection behaviour under a wide array of compacting conditions. It is also shown that the flow rate, an important characteristic to ensure consistent part to part weight, can be improved through different manufacturing conditions even without the use of metallic stearates. Furthermore, such excellent flows can be maintained even under warm and humid environmental conditions.

Keywords: Lubricants, high-performance, ejection

Introduction

Increasing parts complexity as well as higher green density requirements in the powder metallurgy industry is stimulating the development of high-performance lubricants. The definition of a high-performance lubricant is not clearly established, but it can be tentatively described. High-performance lubricants should generally exhibit better ejection behaviour (reduced ejection forces), rapid and even flow properties, high apparent density and resistance to humidity. Furthermore, a lower amount of high-performance lubricant can be added to metal powder mixes while maintaining equivalent ejection behaviour. Since lubricants greatly limit the green density which can be reached with the single compaction process, higher densities can be obtained [1]. For several decades – and to some extent still today – Kenolube was considered as one of the best performing lubricants on the market. It is, however, plagued by some characteristics that are nowadays preventing it from being the lubricant of choice for new PM applications. For instance, Kenolube is sensitive to high temperatures and mostly high ambient humidity, which promotes the formation of lumps and negatively impact the flow behaviour. These are major drawbacks for many PM parts manufacturers. From an environmental standpoint, the zinc content of Kenolube is being pinpointed as the culprit for the emission of zinc-bearing contaminants from sintering furnaces exhaust stacks and for potential decarburization issues [2].

These fundamental changes in the PM industry are the driving force behind the development of new fatty acid based composite waxes or polymeric lubricants. Many properties are desirable for such lubricants. Their lubricating properties need to be excellent in order to provide the lowest ejection forces possible, even at lower concentrations [1] or when used to obtain higher densities. Since these new lubricants are mostly free of metallic stearates, it is imperative that they offer good flow properties. It is also desirable that they favour high apparent densities, have a low hygroscopicity, are effective to bind finer additives and can be mostly eliminated during sintering without leaving residuals behind. The nature of the lubricant itself clearly has an impact on the lubrication performance. Yet, the best ejection behaviour is obtained through a careful optimisation of the particle size distribution of the lubricant. Indeed, coarse particles were shown to lead to better ejection performance and higher green strength. Adversely, the compressibility of the powder mix is often negatively impacted while lower sintered properties are obtained. The latter is due to larger voids within the iron matrix following sintering. Consequently, a compromise to achieve both the better ejection performance and maintain the sintered strength could be the use of a bimodal distribution, composed of finer and coarser particle sizes [3,4,5]. In previous work, Thomas et al. (2011)[6] demonstrated the superior ejection behaviour of a new composite wax having larger particle sizes over lubricants with a finer particle size distribution. Fig. 1 shows that green densities similar to Kenolube and above 7.30 g cm^{-3} can be obtained with a lubricant containing large particles while considerably lowering the stripping pressure required to initiate the ejection process. Likewise, it is possible that the lubricant particle morphology also affects the ejection properties, although this was never clearly demonstrated.

Green Density
WC–Co Die at 60°C – 12.7 mm parts – 5 parts/minute

Stripping Shear Stresses
WC–Co Die at 60°C – 12.7 mm parts – 5 parts/minute

× Kenolube ◆ Composite Wax – Large Particle Size ■ Composite Wax – Small Particle Size

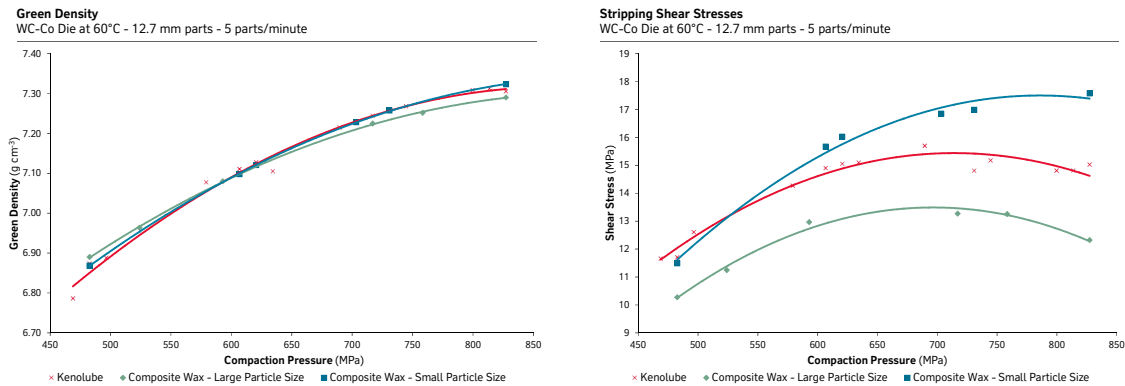× Kenolube ◆ Composite Wax – Large Particle Size ■ Composite Wax – Small Particle Size

Fig. 1 Effect of different particle size distributions on the maximum green density and stripping ejection pressure of a FD-2000 mix containing 0.3%wt. graphite and 0.7%wt. lubricant. (Adapted from Thomas *et al.* (2011)[6])

Adding to the complexity of using the proper lubricant for a given application are the requirements of the said application. For instance, tall parts would benefit from a lubricant which can remain at the die wall and part interface for a long period of time. The exhaustion of the lubricant film at the interface would cause an increase of the shear stresses which could potentially lead to a difficult ejection and even scoring on the part or the tooling. Consequently, a lubricant which performs well on short parts will not necessarily be as efficient for taller parts.

Following the same reasoning is the effect of the compaction rate on the ejection behaviour of different lubricants. At higher shear rates, which are induced trough faster motion of the upper punch, some lubricants may perform better than others. This might be caused by the different responses of the lubricant to varying shear rates and the higher part temperature which is attained at higher compaction speeds. The elevated temperature may favour the melting or the softening of the lubricant, which in turn would facilitate the migration of the lubricant to the die wall, where it can contribute to lower the ejection forces. The flipside of this phenomenon would be that lubricants having a lower viscosity at the die wall/part interface would not be as efficient for taller parts, as the lubricant film would tend to exhaust faster.

Consequently, finding the proper lubricant for a given application is a challenge of its own. This paper describes the ejection behaviour of novel types of composite waxes which were developed at Rio Tinto Metal Powders. These lubricants are compared to a Kenolube and an Acrawax C benchmarks and their ejection behaviour under different compaction conditions is detailed. Particular attention will be given to the impact of the increased compaction rate as well as the behaviour of the lubricant when pressing taller parts. The resistance to warm and humid environment is also evaluated.

Experimental Procedure

All powder mixes were prepared using ATOMET 1001HP, a water-atomised steel powder, manufactured by Rio Tinto Metal Powders. Each was admixed with 1.8%wt. copper, 0.7%wt. natural graphite and 0.7%wt. of the lubrication system. Two powder mixes were used as benchmarks; one containing Kenolube P11 and the other Acrawax C atomized. These are two well-known lubricants which are widely used in the PM industry. Table 1 describes the powder mixes that were evaluated for their ejection performance.

Table 1. Powder mixes used to determine the ejection behaviour of the lubricants.

Mix ID	Base Powder	Copper	Graphite	Lubricant	Flow Enhancer Additive
1	AT-1001HP	1.8%wt.	0.7% wt.	PR-1 0.7% wt.	0.016% wt.
2				PR-1E 0.7% wt.	0.016% wt.
3				PR-2 0.7% wt.	-
4				PR-3 0.7% wt.	-
5				Kenolube P11 0.7% wt.	
6				Acrawax C 0.7% wt.	0.016% wt.

The proprietary composite lubricant PR-1 was first described in Paris *et al.* (2012)[7] and is a formulation aimed at providing lower ejection forces. Other lubricants were derived from this experimental formulation. PR-1E is an enhanced version of PR-1 designed to improve the flow rate and apparent density of the mix, while maintaining the ejection performance. PR-2 and PR-3 are proprietary enhancements of PR-1 and PR-1E in which the manufacturing method was changed and the particle size distributions slightly modified.

The apparent density and flow rate were measured using a Hall flow meter apparatus, according to MPIF Standard 4 and 3, respectively [8]. The compaction and ejection behaviour were evaluated at the National Research Council Canada (Boucherville, Canada) on a 150 t mechanical press. The press is equipped with strain gauges which can record the pressure applied on the top and bottom punch throughout the entire compaction and ejection process. A tungsten carbide die was used to press rings of 25.4 mm across with a core pin diameter of 14.2 mm. Two different part heights were tested, 12.7 mm and 25.4 mm. Furthermore, the parts were compacted at two different speeds, namely 5 parts per minute and 10 parts per minute, which drastically increases the shear stresses during compaction and can impact the lubrication performance. The parts had an M/Q ratio of 4.54 (12.7 mm high parts) and 9.07 (25.4 mm high parts). As a reference, a standard TRS bar made according to MPIF standard 60 has an M/Q ratio of about 1.4. In order to obtain complete compressibility curves, parts were pressed at four compaction pressures of 485, 620, 715 and 825 MPa.

The press software outputs single ejection curves for each stroke. Consequently, a second software was used to recover the stripping pressure, sliding pressure and ejection pressure. More details about how this procedure is being performed can be found in Paris *et al.* (2012). In order to account for possible variations in the parts height, the forces obtained were converted to shear stresses using Equation 1.

$$\tau = \frac{F}{\pi h (d_o + d_i)}$$ (1)

In Equation 1, d_o is the outer diameter (in this case, 25.4 mm), d_i the inner diameter, equal to the core pin diameter of 14.2 mm and h is the part height. This procedure ensures that the reported shear stresses are independent of possible differences in the part heights.

The sample parts were measured after compaction with at CMM apparatus; model SmartScope Flash 300, with a precision of 1.5 μm. The diameter is measured at 40 points around the circumference of the part, at mid-height. These measurements are used to determine the radial springback according to Equation 2.

$$s = \frac{d_p - d_d}{d_d} \times 100$$ (2)

In Equation 2, d_d is the diameter of the die and d_p is the diameter of the part. The springback is expressed as a percentage and consequently, the higher the percentage of springback, the greater the parts have swelled after being ejected out of the die.

The resistance of the powder mixes to warm and humid environments was measured according to a procedure established in Thomas *et al.* (2009) [6]. Samples based on an AT-1001HP base powder and containing 0.6%wt. of natural graphite, 0.3%wt. MnS and 0.8%wt. of lubricant were prepared. The mixes are described in Table 2.

Table 2. Description of the powder mixes used to evaluate the resistance to humidity.

Mix ID	Base Powder	Graphite	MnS	Lubricant	Flow Enhancer Additive
1	AT-1001HP	0.6%wt. F25	0.3%wt. Arcmetal	PR-1 0.8%wt.	0.016%wt.
2				PR-3 0.8%wt.	-
3				KEN 0.8%wt.	-

It is believed that the MnS additive contained in the powder mix can interact with hygroscopic lubricants and deteriorate the flow rate when the mix is being exposed to warm and humid conditions [6,9]. As such, highly hygroscopic lubricants would not flow after the conditioning period whereas non-hygroscopic lubricants are expected to maintain their flow behaviour. To perform this test, samples of 1 kg of the powder mixes are placed in a Blue M climate-controlled chamber which is equipped with a small V-type blender. Each powder blend was placed in the blender which was left open for an approximate period of one hour. This time span is necessary for the powder to reach equilibrium with its surrounding environment. For this test, the chamber was set

at a temperature of 60°C and 60% RH. After this period, the blender was closed and the powder blended for 30 minutes, after which a sample was collected. After the sampling was completed, the blender was turned on for a period of 24 hours. Once this period was over, another sample was taken. The flow rate and apparent density were measured on the first sample (taken out after 30 minutes of blending time). The last sample was also measured after a 24 h rest period.

Results and Discussion

Green Densities Achievable with High-Performance Lubricants
Fig. 2 and Fig. 3 show the maximum green densities which can be achieved at the selected compaction pressures. With conventional compaction (room temperature, WC-Co die not heated) at 5 parts per minute and 12.7 mm high parts, the maximum green density is about 7.21 g cm^{-3} and is reached with the Kenolube mix, the PR-1 mix and the PR-1E mix. The lubricants PR-2 and PR-3 offer lower green densities with respectively 7.17 g cm^{-3} and 7.18 g cm^{-3}. With the warm die compaction technique (WC-Co die heated at 60°C) at 5 parts per minute and 12.7 mm high parts, the highest green density attained was about 7.22 g cm^{-3}. This value was obtained with the lubricants PR-3, PR-1E and Kenolube. The lubricants PR-1 and PR-2 allowed for densities near 7.21 g cm^{-3} to be reached. The lower densities obtained with the PR-2 and PR-3 lubricants at room temperature can potentially be explained by a slightly larger particle size distribution, which may hinder the compressibility. The difference between all the lubricants is significantly narrower when using warm die compaction. It is likely that the higher compaction temperature caused some partial melting or some softening of the coarser lubricants. In effect, a partially molten lubricant would not limit the compressibility as much as a solid lubricant because no energy is lost deforming the lubricant particles.
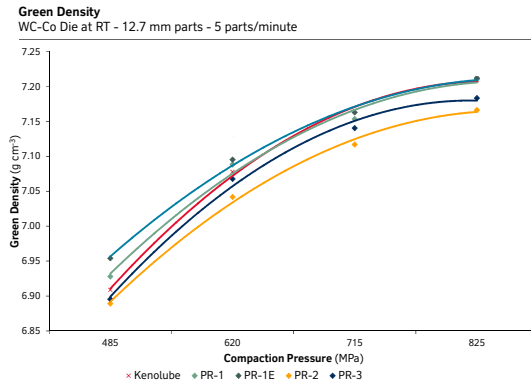

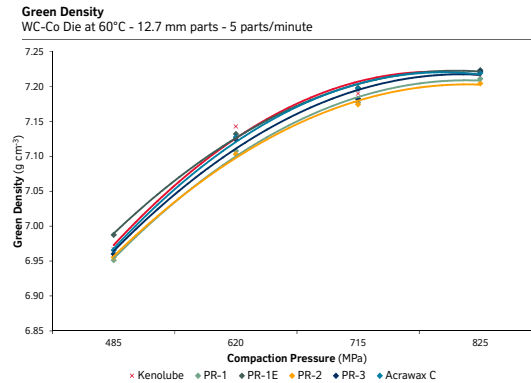Fig. 2. Green densities achievable at room temperature.

Fig. 3. Green densities achievable using warm-die compaction at 60°C.

Influence of Part Height on Ejection Behaviour
Fig. 4 through Fig. 6 show the ejection behaviour of the different mixes when compacted on a heated die at a temperature of 60°C, at both part heights of 12.7 mm and 25.4 mm. In all cases, the ejection shear stresses for the Kenolube and the proprietary lubricant mixes are lower than the shear stresses for the Acrawax C mix. The stripping, sliding and ejection shear stresses of the Acrawax mix are either maintained constant or increase with increasing compaction pressure, whereas a different phenomenon is observed for the other lubricants. In effect, the shear stresses tend to diminish with increasing compaction pressures. As expected, for taller parts (25.4 mm), the shear stresses increase significantly for the mix containing Acrawax. For the Kenolube mix, the stripping, sliding and ejection shear stresses are, as expected, significantly lower than the Acrawax Mix and tend to diminish for taller parts. In effect, the stripping shear stress for the Kenolube mix at a compaction pressure of 825 MPa is about 11.7 MPa for a 12.7 mm high part. For taller parts (25.4 mm), the stripping shear stress at 825 MPa drops to about 11.2 MPa. This behaviour is also observed for the other high-performance lubricants, PR-1E and PR-2. For the lubricant PR-2, the difference in shear stresses measured between shorter and taller parts is less than the difference for the Kenolube mix. Nevertheless, they typically show lower shear stresses across the entire compaction pressure range. This is particularly noticeable for the PR-1E lubricant, which has an ejection shear stress as low as 6.31 MPa at a compaction pressure of 825 MPa.

The significant increase in shear stresses observed for the Acrawax C mix demonstrates that the lubricant performance of this EBS wax is adversely affected by the part height. It appears intuitive that taller parts would require more force to be ejected from the die, as the surface area in contact with the die is more important. This is actually reflected in the higher M/Q ratio of the 25.4 mm part. However, shear stresses, as they account for the part height, should remain unaffected providing that similar lubrication is supplied at the die wall to part interface. On the other hand, the reduced stripping shear stresses that were

4

observed on the taller parts for the lubricants Kenolube, PR-1E and PR-2 were slightly surprising. Although the difference is moderate – mostly contained within a 10% range – this tends to demonstrate that these three lubricants have a totally different behaviour than Acrawax C when the part height is increased. It is however possible to tentatively explain why such differences between the lubricants were observed. The EBS wax generally has a higher melting point than Kenolube. In a previous study [10], it was demonstrated that Kenolube has several endotherms corresponding to the melting point of its constituents. The DTA analysis confirmed the different melting points to be at 71°C, 103°C and 134°C compared to 145°C for the Acrawax C. Since more heat is generated during the compaction of taller parts, it is possible that the Kenolube and the other high-performance lubricants achieved a higher degree of melting, thereby facilitating the migration of the lubricant to the die walls and improving the lubrication [11]. It is also possible that an increased density gradient would affect the flow of the partially molten lubricant within the part. It was recently demonstrated that volumes of lower density within a part would delubricate first, due to a different pore structure in such areas [12]. This different pore structure could provide more lubricant at a key location for the ejection process, namely at half the height of the part.
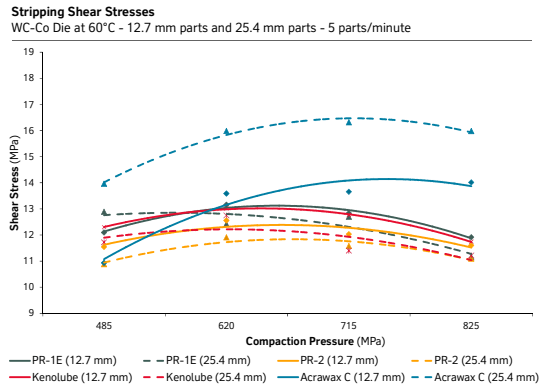

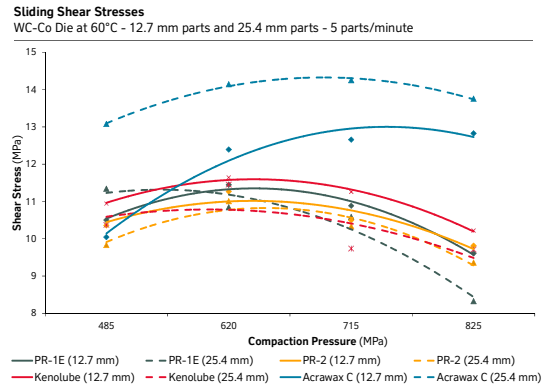Fig. 4. Stripping shear stresses for lubricants PR-1E, PR-2, Acrawax C and Kenolube at 60°C.

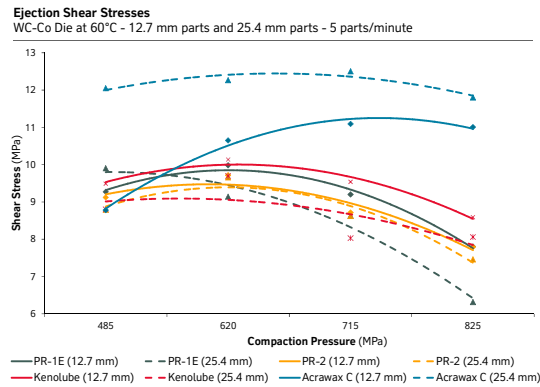Fig. 5.Sliding shear stresses for lubricants PR-1E, PR-2, Acrawax C and Kenolube at 60°C.

Fig. 6. Ejection shear stresses for lubricants PR-1E, PR-2, Acrawax C and Kenolube at 60°C.

Influence of Compaction Rate on Ejection Behaviour

Fig. 7 through Fig. 9 are demonstrating the effect of an increased shear rate on the ejection behaviour of three lubricants. The parts were pressed on a WC-Co die heated at 60°C. The proprietary lubricant PR-1 has a similar behaviour to Kenolube. Nevertheless, its ejection properties are far superior to standard Acrawax. Increasing the stroke rate from 5 to 10 parts per minute is causing the stripping and sliding shear stresses to drop for the EBS wax mix, while the ejection shear stresses remained more constant. The stripping shear stress for the Kenolube mix is little changed between compaction at 5 and 10 parts per minute. However, at low compaction pressures, there is a tendency for the sliding and ejection shear stresses to increase. At higher compaction pressures, this difference eases up to the point where no difference can be seen. For the PR-1 lubricant, the stripping shear stress diminishes at higher compaction pressures when the stroke rate increases to reach about 9.78 MPa, equal to the Kenolube mix. The diminution provided by PR-1 in the sliding and ejection shear stresses is more evident at higher compaction pressures. For instance, the PR-1 mix has an ejection shear stress of 5.83 MPa at 5 parts per minute and 6.40 MPa at 10 parts per minute. This compares to about 7.31 MPa for the Kenolube mix at 5 parts per minute and

7.46 MPa at 10 parts per minute. The difference between the Acrawax mix and the PR-1 and Kenolube mixes can be attributed to the lower melting point and higher particle size distribution of the latter two lubricants. At higher stroke rates, higher shear rates are applied on the lubricants and more thermal energy is released into the parts and consequently, shear rate dependent and lower melting point lubricants will tend to migrate to the die wall with more ease.
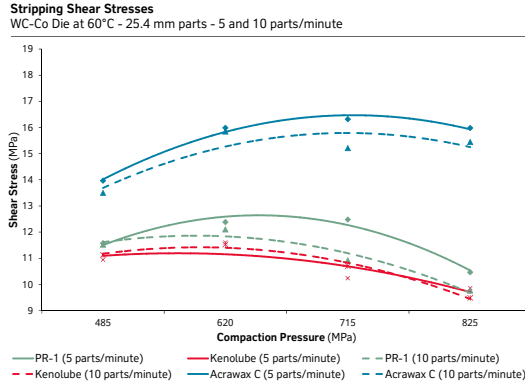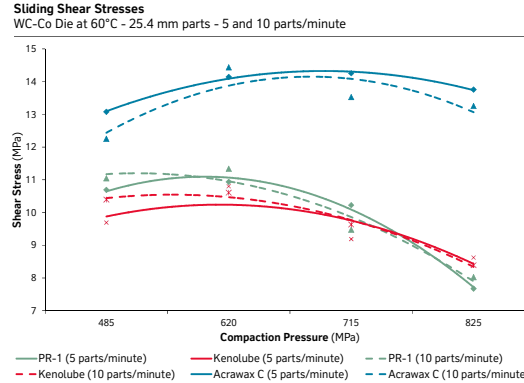

Fig. 7. Stripping shear stresses for lubricants PR-1, Acrawax C and Kenolube at 60°C.

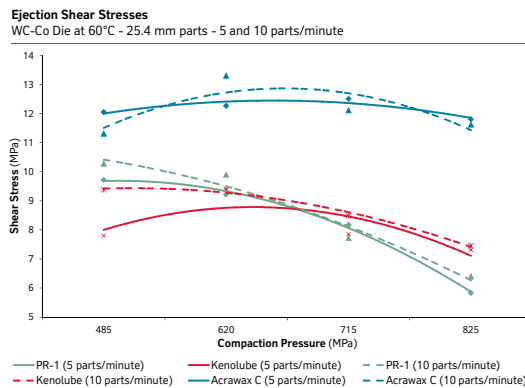Fig. 8. Sliding shear stresses for lubricants PR-1, Acrawax C and Kenolube at 60°C.

Fig. 9. Ejection shear stresses for lubricants PR-1, Acrawax C and Kenolube at 60°C.

Evaluation of the Resistance to Warm and Humid Atmospheric Environments

Fig. 10 shows the effect on the apparent density of a 30 minutes exposure to the high humidity, high temperature environment. The measurements were done after a 24 hour rest period at ambient (yet controlled) temperature and relative humidity. The powder mix with lubricant PR-1 has a low apparent density, 2.95 g cm^{-3} after 30 minutes of blending in the warm and humid environment. After 24 hours of blending, the apparent density sharply increased by about 0.18 g cm^{-3} to 3.13 g cm^{-3}.   The lubricant PR-3 has a much higher apparent density after 30 minutes compared to PR-1 at 3.19 g cm^{-3}. The increase in apparent density after the 24 hour blending period was limited to about 0.08 g cm^{-3}. The Kenolube powder mix had a high apparent density of 3.27 g cm^{-3} after 30 minutes of blending. However, after being blended for 24 hours in the atmospheric chamber, its apparent density significantly dropped to 3.06 g cm^{-3}, a 0.21 g cm^{-3} decrease.

The impact of the exposure to high temperatures and high humidity levels on the flow rate is shown in Fig. 11. The Kenolube powder mix had an excellent flow rate of 25.7 seconds per 50 g after being exposed to the warm and humid environment for 30 minutes. However, after a 24 hour exposure, the flow rate had degraded so much that it was impossible to record any flow on a Hall funnel. The mix containing the lubricant PR-1 had no measurable flow after the 30 minute exposure. Not surprisingly, the PR-1 mix showed no flow as well after the 24 hour exposure period. The powder mix made from the lubricant PR-3 had a good Hall flow rate of 27.9 seconds per 50 g after being blended for 30 minutes in the atmospheric chamber. Furthermore, this value was maintained with a reading of 27.6 seconds per 50 g after the 24 hour exposure period. This result indicates that the manufacturing procedure of a lubricant might impact its ability to cope with environmental conditions where temperature and humidity levels are high.
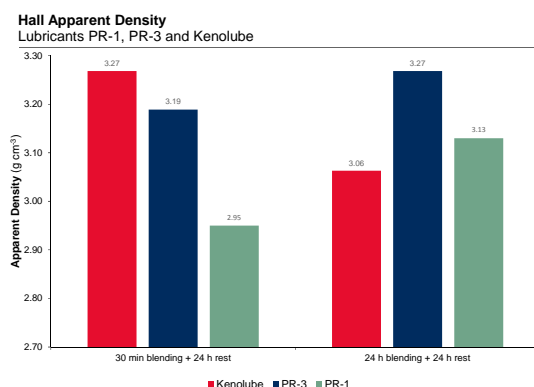
Hall Apparent Density
Lubricants PR-1, PR-3 and Kenolube

Fig. 10. Apparent density of lubricants PR-1, PR-3 and Kenolube based mixes measured after 30 minutes and 24 hours of exposure at 60°C and 60% RH.
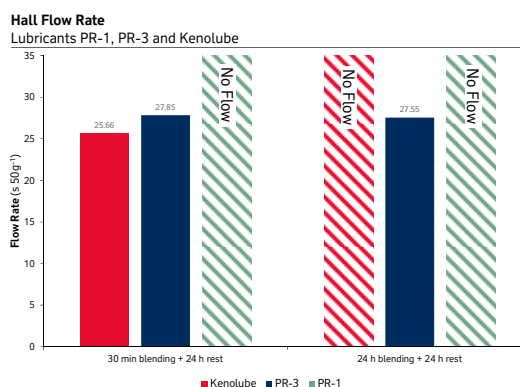
Hall Flow Rate
Lubricants PR-1, PR-3 and Kenolube

Fig. 11. Hall flow of lubricants PR-1, PR-3 and Kenolube based mixes measured after 30 minutes and 24 hours of exposure at 60C and 60% RH.

Conclusion

In a previous paper, the novel lubricant PR-1 was introduced as a potential Kenolube substitute because it had demonstrated excellent ejection behaviour both with conventional and warm die compaction. The effect of other compaction parameters – such as the stroke rate and the height of the parts pressed – was not investigated and it was consequently impossible to determinate if that formulation could satisfy the criteria of a high-performance lubricant. Furthermore, the apparent density of the powder mix was lower compared to Kenolube, unless a different flowing agent admixing method was used. The resistance to warm and humid environments was not yet completed.

As the research and development work pursued on this family of lubricants, other lubricant manufacturing techniques were investigated. It was therefore necessary to assess their effectiveness. The effects of more stringent compaction parameters on the ejection behaviour (stroke rate, taller parts) were also tested. It appears that the PR-1 family of lubricant and its derivatives PR-1E, PR-2 and PR-3 can perform very well with tall parts and at higher shear stresses induced by a rapid stroke rate. The apparent density of a powder mix containing the lubricant PR-1 is lower compared to the Kenolube benchmark. This is a consequence of the addition of a flowing agent. The PR-1 derivative PR-3 has a much higher apparent density than its relative, PR-1. Furthermore, this apparent density value appears to remain stable, even after long blending and exposure to aggressive environments. More development work will be required to optimise the apparent density, particularly on the scale-up effect from small, laboratory scale blenders, to larger industrial equipment.

Perhaps one of the most significant improvements achieved through the enhancements made to the PR-1 family of lubricants is the flow behaviour of the powder mixes. In effect, the lubricant PR-3 achieved a very rapid flow, comparable to lubricants containing metallic stearates, yet, it does not contain any. Furthermore, a 24 hour exposure to a warm and humid environment did not affect the flow rate, which remained similar. This formulation could therefore be used to manufacture premix blends that are more stable and more reliable than ever before.

The comparison with the lubricant Acrawax C demonstrates that in order to satisfy the requirements of a high-performance lubricant, a complex balance of parameters must be controlled. Both the particle size distribution and the manufacturing technique appear to have a significant impact on the ejection behaviour as well as the physical properties of the powder mixes. It was also demonstrated that the lubricants PR-1 and its derivatives can perform as well as Kenolube over a wide range of compaction conditions.

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