

## **Effect of Atmospheric Humidity and Temperature on the Flowability of Lubricated Powder Metallurgy Mixes**

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### **ABSTRACT**

Good powder mix flowability is required in high-volume powder metallurgy (P/M) manufacturing in order to ensure a uniform and consistent filling of the die cavities and, in turn, high productivity, low rejection rates, part integrity and consistent part-to part characteristics such as dimensional change. Apart from the particle size and the shape of the metallic powders and additives, lubricants, even though admixed in small quantities (< 1.5 weight %), have a significant impact on the flow characteristics of powder blend formulations. In addition to that, the blending parameters and the atmospheric conditions such as powder temperature and atmosphere temperature and humidity, may also affect significantly the flow of powder mixes. For instance, it is known that some mixes produced during humid and hot summer days may behave differently.

In this paper, the effect of relative humidity and temperature on the flow of conventional P/M mixes containing different types of lubricants was studied. Typically, amide wax, Kenolube, zinc stearate as well as Caplube L were admixed in a V-blender enclosed in an environmental chamber set at different humidity and temperature levels to simulate different atmospheric and processing conditions. The sensitivity to such conditions of the different lubricants was assessed and the influence on the flow of the powder mixes was measured.

### **INTRODUCTION**

Good powder mix flowability is required in high-volume powder metallurgy (P/M) manufacturing in order to ensure a uniform and consistent filling of the die cavities and, in turn, high productivity, low rejection rates, part integrity and consistent part-to part characteristics such as dimensional change. Intrinsic parameters that affect the flow are the particle size and shape of the metallic powders, as well as their surface properties and nature of additives that can influence the interparticulate friction. For instances, lubricants such as zinc stearate or ethylene-bis stearamide, even though admixed in small quantities (< 1.5 wt%) have a strong effect on the flow behaviour of the metallic powder mixes. However, extrinsic parameters such as blending and storage parameters as well as the environmental conditions such as powder temperature reached in the blender, the atmospheric temperature and relative humidity, could also have a great impact on powder mix flowability by affecting also the interparticulate friction. Indeed, it is well known in the PM industry that the behaviour of some production-scale mix formulations changes when produced during humid and hot summer days. Their behaviour on the press can also change with time. Indeed, as observed by Hausner more than thirty years ago for some given powder mix formulations, the flow, die filling and compressibility behaviour of samples from a given lot of powder mix was changing with time and atmospheric conditions [1].

The problem of flowability observed with some mixes at high humidity level may be explained by the occurrence of strong capillary forces that increase significantly the cohesion between particles. At low relative humidity, the moisture associated with the particles is adsorbed water vapour. As the relative humidity increases, the thickness of the adsorbed layer increases, until eventually condensation occurs at the contact points, generating liquid bridges [2]. The effect of humidity is however different depending on the type of metal powders but also on the particle size distribution for the same metal powder. For measurements performed at room temperature, Peterson et al. [3] reported a first critical humidity level near 60% and a threshold humidity level of 85% for water-atomized iron powder. Above this threshold value, a decrease of the measured apparent density and an increase of the flow time were observed for all the powder size fractions, but in general finer powders (<50 $\mu\text{m}$ ) were more sensitive to humidity. Irreversible behaviour of AD and flow properties was exhibited once the powder was exposed to humidity level higher than the threshold value. Peterson suggested that this irreversible phenomenon is caused by a sharp increase in the amount of condensed moisture on the particle surfaces that could promote corrosion in presence of surface contaminants and enhance interparticular friction. Regarding aluminum powders, reducing the sensitivity of these powders to humidity was recently shown by applying an hydrophobic coating at the surface of these powders that decrease the amount of moisture adsorbed onto the particle surface and in turn decrease the contribution of the capillary forces acting on the powder [4].

If the influence of atmospheric conditions on the flowability of powders is well known in particular for pharmaceutical, fertilizer and food applications, relatively few studies have been published specifically on metal powders admixed or not with alloying elements or additives, graphite and lubricant. As reported earlier, Hausner published thirty years ago preliminary experimental observations on metal powders that clearly indicated that environmental factors such as humidity and/or temperature influenced the interparticulate friction conditions in a powder mass, and therefore, the behaviour of powder properties such as flow and apparent density [1]. The same author also reported that ‘‘there are good reasons to believe that humidity affects powder particles, depending on the type and amount of lubricant added’’. One of the reasons that may explain the little amount of work done to evaluate the effect of blending temperature and humidity on mix flowability is the difficulty to reproduce in a laboratory scale the results achieved in much larger blenders under real production conditions. It was clearly one of the main objectives of that study to establish laboratory procedures and conditions that can reproduce problems encountered at the production scale.

Thus, in this paper, the effect of relative humidity and temperature on the flow of conventional P/M mixes containing different types of lubricants was studied. Typically, amide wax, Kenolube, zinc stearate as well as Caplube L were admixed in a V-blender enclosed in an environmental chamber set at different humidity and temperature levels to simulate different atmospheric and processing conditions. In this study a special procedure was develop to reproduce problems encountered under real production conditions. The sensitivity to such conditions of the different lubricants was assessed and the influence on the flow of the powder mixes was measured.

## **EXPERIMENTAL PROCEDURE**

### ***Description of lubricants used and characterization performed***

Four different lubricants were used in this study: Kenolube as well as Acrawax C, zinc stearate and Caplube L. Table 1 described their nature, as given in their material safety data sheet [5,6], suppliers and particle size distribution measured on a Coulter laser diffractometer, model LS230.

**Table 1: Description and characterization of lubricants used in this study.**

|               | Nature                            | Supplier           | Particle size distribution ( $\mu\text{m}$ ) |      |      |
|---------------|-----------------------------------|--------------------|--|------|------|
|               |                                   |                    | D10  | D50  | D90  |
| Acrawax C     | EBS or ethylene bis-stearamide    | Lonza              | 1.0  | 5.9  | 16.2 |
| Zinc stearate | ZnSt                              | H.L. Blachford Ltd | 1.3  | 8.0  | 24.1 |
| Kenolube P11  | Amid wax + ZnSt                   | Hoganas AB         | 2.0  | 22.6 | 60.3 |
| Caplube L     | Mixture containing metallic salts | H.L. Blachford Ltd | 3.3  | 21.1 | 76.6 |

The phase transitions of the four lubricants as a function of temperature were characterized on a Setaram (Caluire, France) differential thermal Analyzer (DTA) under argon atmosphere at a heating rate  $2^{\circ}\text{C}/\text{min}$ . The sensitivity of the lubricants to humidity were evaluated through the determination of their equilibrium sorption isotherm using a closed system in which a micro-balance weights the samples as they are exposed to different relative humidity by mixing dry and saturated air (DVS gravimetric sorption analyzer from Surface Measurement Systems, NA, USA).

#### *Description of powder mixes and blending conditions used*

The effect of blending parameters and the atmospheric conditions, such as powder temperature and atmosphere temperature and humidity, on the flowability of ferrous powder compositions containing these four lubricants was evaluated in a Blue M environmental chamber with humidity and temperature control in which a V-Type blender was installed. Typically, 2.2lbs powder mixes consisting of ATOMET 1001HP steel powder admixed with 0.6 wt% graphite, 0.3 wt% MnS and 0.8 wt% of lubricant were prepared under different conditions of temperature and levels of humidity. In all cases, the iron powder admixed with the graphite and MnS powders were first allowed to reach the targeted temperature and humidity (~1 hour) before introduction of the lubricant. Mixing was then performed during 24 hours. Samples were collected after 30 minutes of mixing and after 24 hours. The 24 hr mixing time used is much higher than what is used in production scale but, due to scale-up effect, it was found to reproduce well the behavior and problems encountered during production of such mixes. Flow and apparent density measurements were performed according to MPIF Standards 03 and 04 [7], on samples 24 hours after being collected and maintained at ambient temperature  $\sim 22^{\circ}\text{C}$  and 30%RH.

The blending temperature and humidity used are described in Table 2. It is worth mentioning that blending was done at temperatures where no partial melting of lubricant is observed ( $60^{\circ}\text{C}$  and lower) and at  $80^{\circ}\text{C}$ , where partial melting of Kenolube and Caplube L is obtained as it will be discussed in the next section.

**Table 2: Conditions used as a function of the lubricant type**

| Conditions  | Acrawax C atomized | Zinc Stearate | Kenolube P11 | Caplube L |
|---|--------------------|---------------|--------------|-----------|
| $20^{\circ}\text{C} / 50\%\text{RH}$              |                    |               | X            |           |
| $30^{\circ}\text{C} / 90\%\text{RH}$              |                    |               | X            |           |
| $50^{\circ}\text{C} / 95\%\text{RH}$              |                    |               | X            |           |
| $60^{\circ}\text{C} / 20\%\text{RH}$              |                    |               | X            |           |
| $60^{\circ}\text{C} / 67\%\text{RH}$              | X                  | X             | X            | X         |
| $80^{\circ}\text{C} / \sim 1\text{-}2\%\text{RH}$ | X                  | X             | X            | X         |
| $80^{\circ}\text{C} / 30\%\text{RH}$              |                    |               | X            |           |

## RESULTS

### 1. Characterization of lubricants

The different lubricants used in this study was characterized by differential thermal analysis (DTA) presented in Figure 1. The Acrawax C ethylene bis-stearamide wax has a typical melting endotherm at  $\sim 145^{\circ}\text{C}$  attributed to the bis-amide compound, while the endotherms at  $\sim 97^{\circ}\text{C}$  and  $\sim 68^{\circ}\text{C}$  should correspond to the mono-amide compound and stearic acid respectively, issued from the synthesis of the bis-amide wax. Zinc stearate has only one endotherm at  $\sim 125^{\circ}\text{C}$  corresponding to the phase transition of this compound. The Kenolube and Caplube L DTA curves show several endotherms validating that these lubricants are mixtures of different compounds, but containing metallic stearates as disclosed in their material safety data sheet. The first melting endotherm peaks for Kenolube and Caplube L are respectively at  $\sim 71^{\circ}\text{C}$  and  $\sim 76^{\circ}\text{C}$ . A minor peak could also be seen at slightly lower temperature ( $\sim 63^{\circ}\text{C}$ ) for Caplube L confirming the presence of a minor amount of lower melting wax.

In this study, blending temperature was intentionally set below and over the partial melting of Kenolube and Caplube L in order to be able to differentiate the effect of humidity and temperature on the flow behavior of powder mixes from the effect of a partial melting of lubricant during mixing. Results for mixing below and above  $60^{\circ}\text{C}$  will be presented separately in the next sections.

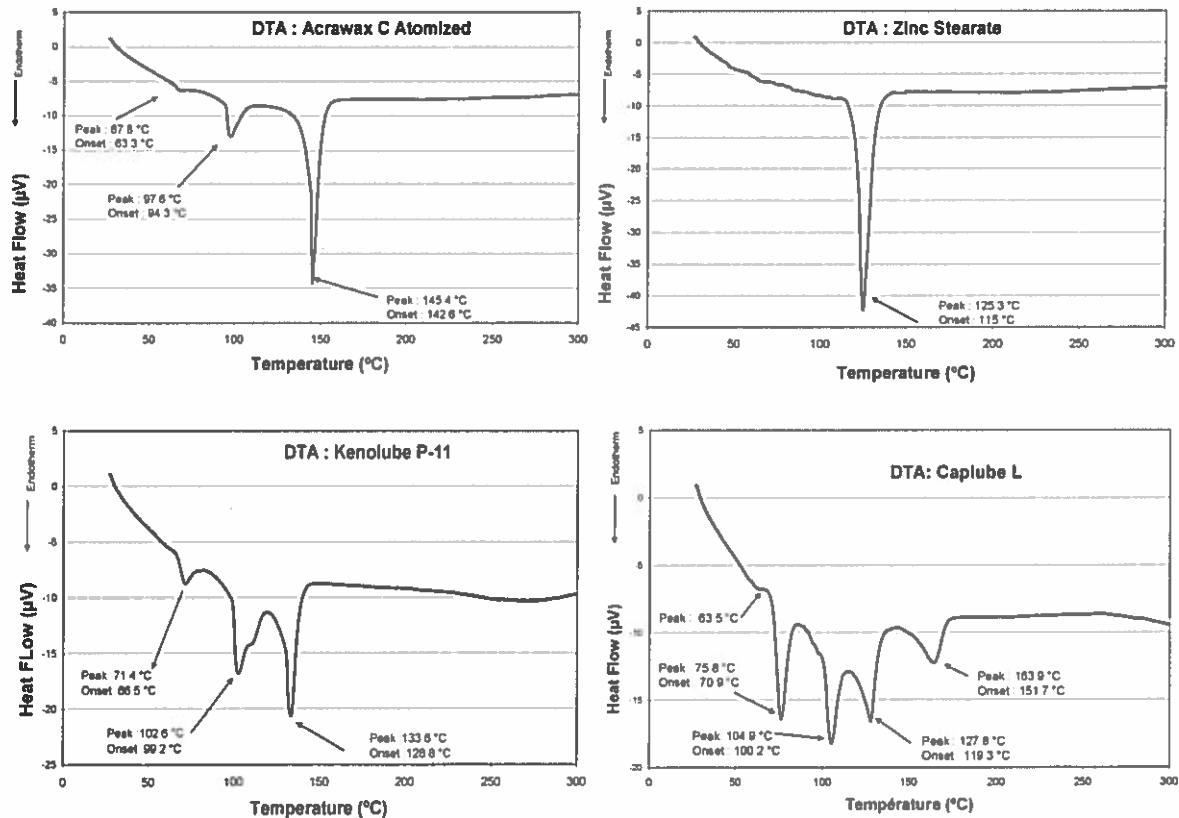


Figure 1: DTA of the four lubricants studied

## 2. Effect of environmental and processing conditions on the flow behaviour of metal powder mixes

### Mixing temperatures below or equal to 60°C

The effects of environmental and processing conditions were first evaluated on metal powder mixes containing 0.8 wt% Kenolube and admixed in a V-blender during 30 minutes and 24 hours in the environmental chamber. The use of an unusual and relatively long mixing time of 24 hours was necessary to duplicate the behaviour of various lubricants and reproduce flowability problems periodically observed in much larger production blending equipment. Flow rate and apparent density as measured after 24 hrs are presented in Figure 2 a) and b). When a 30 min mixing time was used, excellent flow rate (~28s/50g) and very stable apparent density (~3.3 g/cc) were achieved whatever the temperature and relative humidity used. However, results were highly dependent on the blending conditions used when a much longer mixing time was used. For mixes processed at temperature of 30°C or lower, whatever the relative humidity, and 60°C and low relative humidity (20%RH), only a slight deterioration of flow rate was achieved. In these conditions, similar or slightly higher apparent densities were measured. However, when a temperature of 50°C or higher combined with very humid conditions were used, the powder mixes did not flow in the Hall funnel. Apparent density, as evaluated in a Carney funnel due to the non-flowing behaviour of mixes, was much lower than achieved in the other conditions, especially at 50°C and 95%RH, where an apparent density of about 2.9 g/cc was measured.

The flow results obtained for mixes with Kenolube are illustrated in a different manner in Figure 3. Indeed, in this figure, the flow behaviour of each mix/condition is given as a function of the calculated corresponding dew point temperature. Mixes with good flow are illustrated with green stars while mixes with no flow are illustrated with red stars. The dew point is the temperature at which the air is fully saturated with moisture. At a given barometric pressure, independent of temperature, the dew point is a useful measure of water vapour in the air. In comparison, the relative humidity (%RH) is defined as the ratio between the actual water vapour pressure and the saturation vapour pressure over water, which varies as a function of temperature. As long as the dew point remained low (10 or 30°C), the powder mixture gave very good flow, while at dew point temperatures of about 50°C, the powder mixture did not flow on a Hall flowmeter. It is noteworthy that the degradation of flow achieved at dew point of about 50°C was an irreversible phenomenon, i.e. flow rate did not recover even after a long period of time under normal atmospheric conditions.

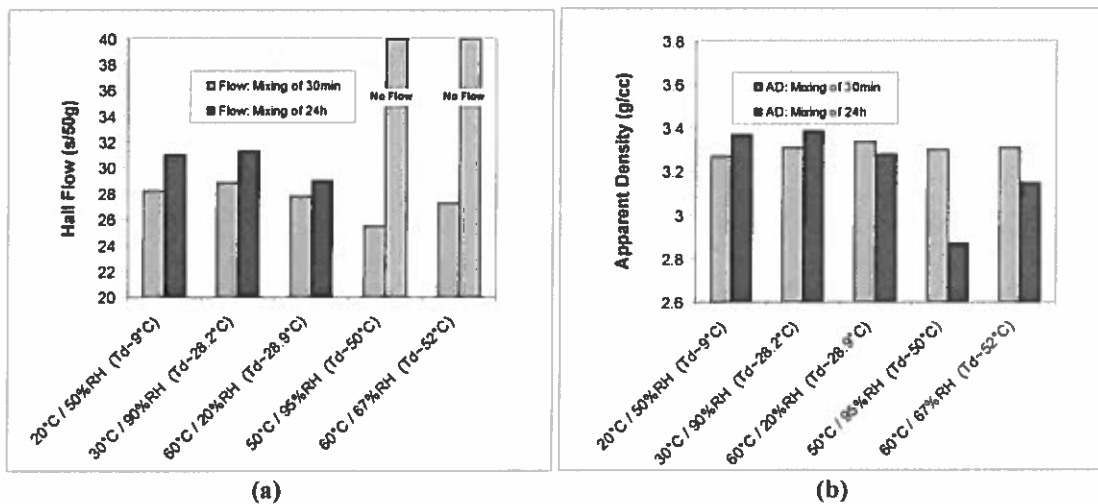
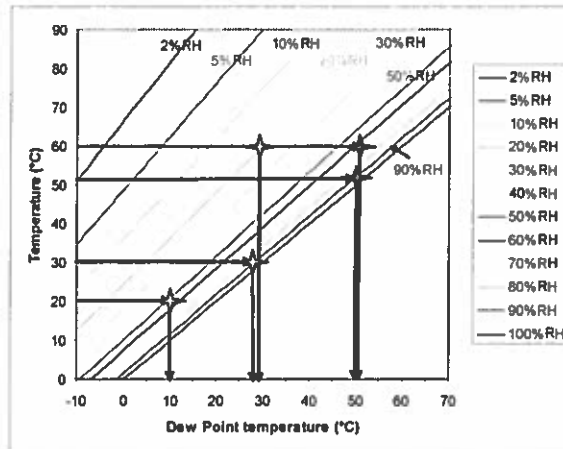


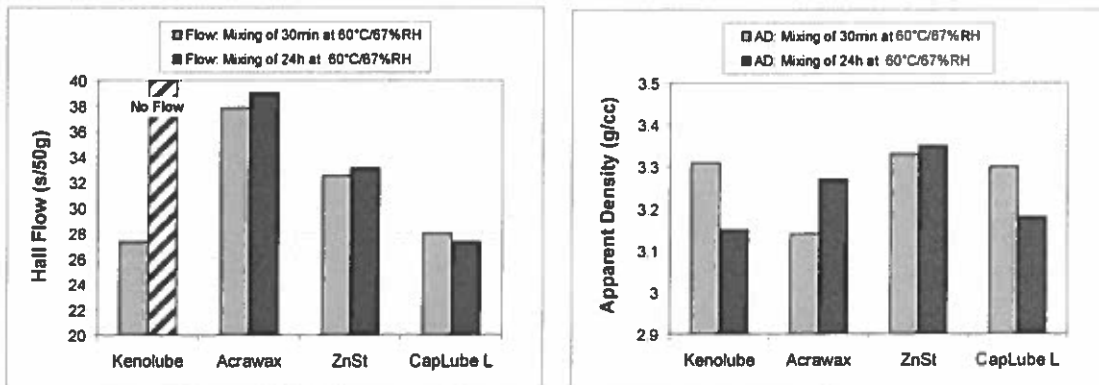
Figure 2: Effect of temperature and relative humidity on a) Hall Flow and b) Apparent Density for mixes containing Kenolube lubricant



**Figure 3: Dependency of Flow behaviour of powder mixes containing Kenolube with dew point temperature (Flow: green star; No Flow: red star)**

The behaviour of powder mixes with Kenolube was compared to that of powder mixes admixed with other lubricants in the same high dew point mixing conditions (Dew point of ~ 50°C, or 60°C/67 %RH). The flow rate and apparent density as measured after 30 min and 24 hrs of mixing time are illustrated in Figures 4 a) and b) respectively. It should be noted again that flow and apparent density were measured 24 hrs after completion of mixing. First, it is seen in Figure 4a that a flow rate was measured for all mixes after 30 min of mixing time, even if flow rate varied from one lubricant to the other, as expected. However, only mix with Kenolube did not flow after 24 hours of mixing time, all the others giving flow rate quite similar to what was measured after 30 min of mixing time. This clearly shows that the sensitivity of Kenolube to the admixing time and atmospheric conditions is much higher than the other lubricants, with a significant deterioration of flow when mixing time is increased from 30 minutes to 24 hours. This observation duplicates well the behaviour of mixes containing Kenolube in larger production scale blenders, where flow tends to deteriorate as blending time is increased. Of course, this behaviour is observed at much lower blending time.

On the other hand, for 30 min of mixing time, a similar apparent density (~3g/cc) was measured for the mixes containing the Kenolube, ZnSt and Caplube L lubricants, while the mix containing Acrawax show a lower value of 3.14g/cc (Figure 4b). However, for a longer mixing time of 24h, different apparent densities were measured depending on the type of lubricant: apparent density remained constant for ZnSt mix, while it decreased for the Kenolube and Caplube L powder mixes reaching respectively 3.15 and 3.18 g/cc, and it increased for the Acrawax mix up to 3.27 g/cc.



**Figure 4: a) Hall Flow and b) Apparent density of metal powder mixes containing the four lubricants prepared at 60°C and 67%RH.**

### Mixing temperatures above 60 °C

The temperature of powder mixes in large production scale blender may reach temperatures above 65°C due to friction and mechanical working. That is why tests were carried out with a temperature of 80°C to try to reproduce phenomena observed in production. However, as discussed previously, depending on lubricant type, partial melting of lubricant during mixing could occur for temperature slightly above 60°C. In particular, Kenolube presents a melting endotherm at ~71°C, while Caplube L presents one minor endotherm at ~64°C and another larger endotherm at ~76°C. Acrawax presents one minor endotherm at ~68°C. Therefore, results were treated separately in this part to be able to differentiate the effect of humidity and temperature on the flow behavior of powder mixes from the additional effect of partial melting of lubricant.

First, it is noteworthy that 1-3 mm white balls agglomerates of lubricants were observed, whatever the mixing time, in the powder mixes containing the Kenolube and Caplube L lubricants, indicating a partial agglomeration of these lubricants when mixing is done at 80°C. Mixes with Acrawax also contained few lubricant agglomerates, but their size and quantity were much lower than with Kenolube and Caplube L. As mentioned previously, partial melting of these lubricants at this temperature should explain this agglomeration phenomenon. The type and small size of blender used (1 liter V-blender) that generates a low shear mixing might also partly explain the fact that these agglomerates were not dispersed in the powder mix. However, it is worth mentioning that such lubricant agglomerates can be occasionally formed in larger production mixer, especially with Kenolube, indicating that precautions must be taken when admixing these lubricants.

Flow rate and apparent density of mixes processed at 80°C as measured after 24 hrs are presented in Figure 5 a) and b). Mixes with Kenolube were prepared at low and high relative humidity (~1%RH and 30%RH) while the other lubricated mixes were only prepared at low relative humidity (~1-2%RH). It should be noted that the dew points calculated for mixes produced at 1-2 and 30% RH are approximately 10 and 50°C respectively, which corresponds quite well to the dew points calculated for mixing tests done at 60°C or lower. When a 30 min mixing time was used, all the mixes show an excellent flow rate, even better than at 30°C and 60°C, the lower flow rate being measured for mixes with Kenolube and Caplube L (~27s/50g). The flow rate of mix with Acrawax improved from 38s/ 50g (60°C) to 34s/50g (80°C). Regarding apparent density, Kenolube mixes kept a good and stable value of ~3.3 g/cc whatever the relative humidity used. However, higher apparent densities (>3.3g/cc) were measured for mixes admixed with Acrawax and ZnSt, while the apparent density decreased for mixes admixed with Caplube L from 3.3g/cc (60°C) to 3.14g/cc (80°C).

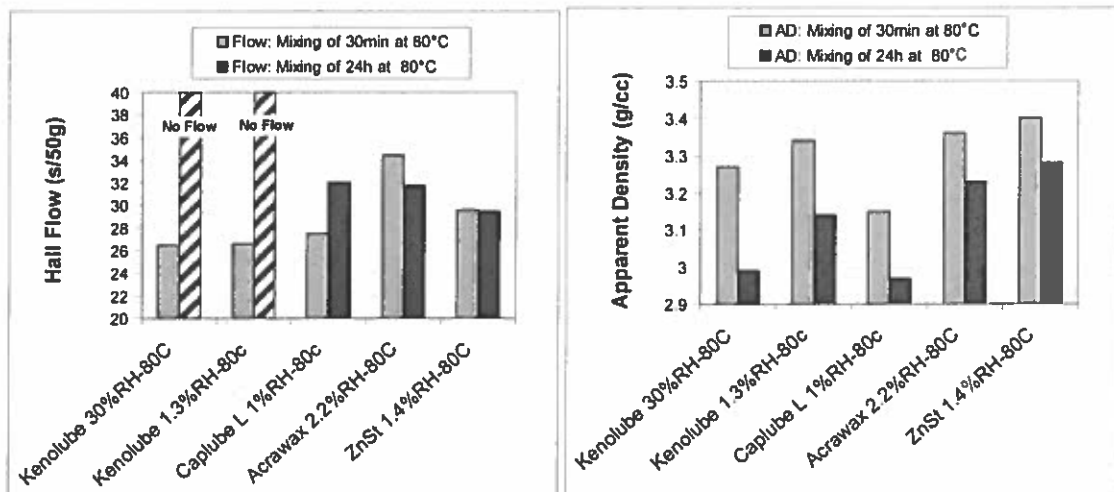


Figure 5: a) Hall Flow and b) Apparent density of metal powder mixes prepared at 80°C.

When a much longer mixing time (24h) was used, much more variations were observed depending on type of lubricants. Mixes with Kenolube did not flow in the Hall funnel whatever the relative humidity used. Mixes with Caplube L did not flow just at the exit of the blender, but recovered its flowability when measured 24 hours after, giving a flow rate of 32s/50g. In the case of Acrawax, a slight improvement of flow rate (32s/50g vs 34s/50g) was obtained while no change in flow rate was measured with ZnSt (~30s/50g). Regarding the apparent density, all the mixes showed a decrease of their apparent density as the mixing time increased. The higher significant decrease ( $> 0.2 \text{ g/cc}$ ) was observed for the non-free flowing mixes with Kenolube that were evaluated in a Carney funnel, and in particular at high humidity level (80°C and 30%RH), where an apparent density of about 3.0 g/cc was measured. A significant decrease (0.18 g/cc) of apparent density by increasing the mixing time was observed for the mixes with Caplube L down to about 3.0g/cc. Finally, a 0.12g/cc decrease of apparent density was observed with mixes containing Acrawax and ZnSt, with apparent densities respectively of 3.23 and 3.28 g/cc.

Figure 6 illustrates the flow behavior as a function of dew point for all the tests carried out in this study for Kenolube-containing mixes.

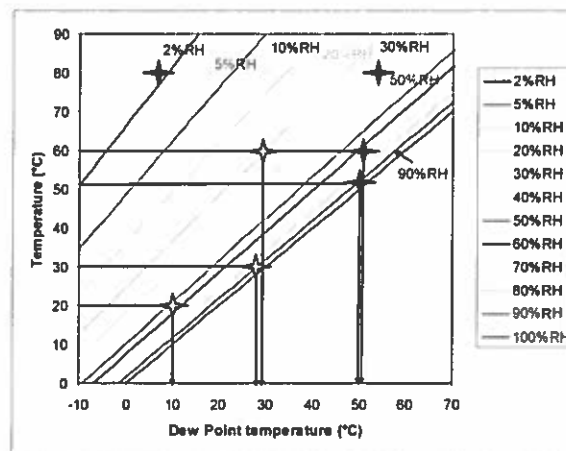


Figure 6: Flow behaviour of Kenolube-containing mixes as a function of dew point and temperature

## DISCUSSION

As compared to results published on water atomized powders by Peterson [3], it is clear that, in presence of lubricants and other additives, the mix formulation investigated showed a lower sensitivity to humidity and thus a higher threshold humidity level above which flow and apparent density are affected. Indeed, at room temperature, Peterson observed a threshold humidity level of 85%, while in our study, excellent flow as well as a high apparent density were still obtained at 30°C and 90%RH for powder mix admixed with Kenolube. In fact, for this mix, a elevated dew point of 50°C, achieved by a combination of high temperature and appropriate level of humidity, was required to degrade both the flow and apparent density. For the other lubricants (ZnSt, Acrawax and Caplube L), excellent flow and high apparent density were still obtained at such a high dew point. This should be explained by the hydrophobic character of the different lubricants which are able to be smeared at the surface of metal powders during mixing, and act as a coating making them water repellent, and reducing the amount of moisture adsorbed on the powder. At very high humidity level, however, condensed liquids will be present, but as shown by Stevens [4], two hydrophobic surfaces joined by a liquid bridge would be less likely to agglomerate by capillary forces and therefore should also be easier to separate.



If the sensitivity to humidity of lubricated mixes was shown to be less than water atomized powders only, it remains that some differences between lubricants were observed. Indeed, for blending temperatures below or equal to 60°C, only Kenolube showed some deterioration of its flow properties under high dew point conditions. In order to better understand the behaviour of these lubricants, the response of these lubricants to humidity was investigated. These results are discussed in the following paragraphs.

The ability of a solid to adsorb water vapour at a particular relative humidity and temperature is usually expressed as an equilibrium sorption isotherm and depends on its chemical affinity for the solid and the number of available sites for interaction. The moisture sorption isotherms at 30°C of the four lubricants are presented in Figure 7a. The first observation is that the overall moisture uptake for the four lubricants between 10 to 90%RH remains very low (below 0.2%) confirming their hydrophobic character. For comparison purpose, crystalline magnesium stearate shows a moisture uptake of ~1% at 90%RH and materials used in tablets pharmaceutical formulations have a wide range of hygroscopicity with moisture uptake from as low as 0.1% to 50% at 90%RH [8]. Even though the lubricants studied are mainly non-hygroscopic, they have different moisture sorption behaviour. First, both Acrawax C and Caplube L showed the lower hygroscopicity among the four lubricants at relative humidity under 65%, with a gradual weight increase reaching ~0.04% at 65% RH. Over 65 %RH, Acrawax still continued to gain weight gradually and at the same rate but gain in weight became much more important for Caplube L, which reached ~0.16% at 90%RH versus 0.06% for Acrawax. This indicates a greater sensitivity of Caplube L to moisture adsorption at high relative humidity. The sorption isotherm of Kenolube shows that moisture is adsorbed constantly up to 65%RH where gain in weight reached 0.06%, but moisture pick-up became more important above 65%RH as it was the case with Caplube L, and reached 0.12% at 90%RH. Finally, the sorption isotherm of zinc stearate shows a significant uptake of humidity between 0 and 30%RH (0.06% at 30%RH), but above 30%RH, moisture pick-up rate is much lower, with a moisture uptake of 0.07% and 0.09% respectively at 65%RH and 90 %RH. From these moisture sorption isotherms, we can conclude that both Caplube L and Kenolube showed the highest sensitivity to humidity at high humidity levels and should therefore be more susceptible to variations of flow behaviour when humidity level changes. On the other hand, Acrawax and ZnSt appeared as the less sensitive lubricants to humidity. This result is in good agreement with the behaviour of mixes containing these two lubricants, which showed very little sensitivity to humidity.

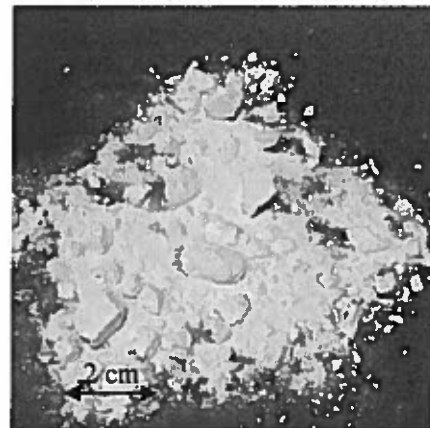
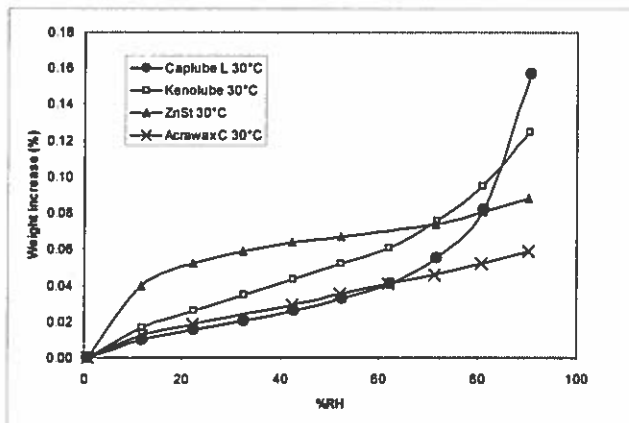


Figure 7: a) Moisture sorption isotherms of lubricants at 30°C and b) agglomerates of lubricants (Kenolube and Caplube L) at 60°C /67%RH (conditioned 24 hours)

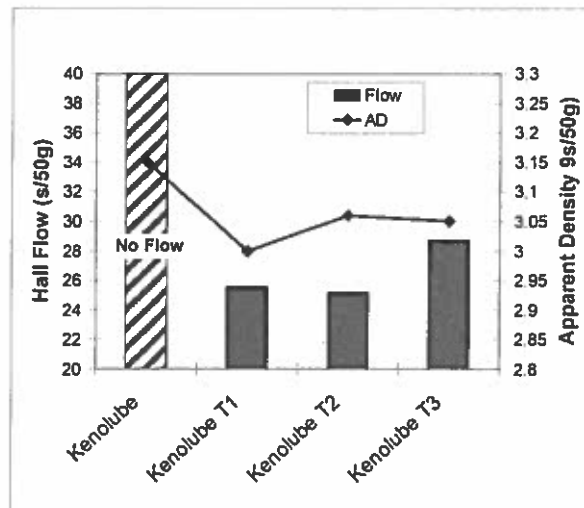
The lower sensitivity of Acrawax and ZnSt to humidity was also confirmed by a conditioning test. This test consists in placing a certain quantity of different lubricant powders in the humidity and temperature controlled chamber for 24 hours. When conditioned during 24 hours at 60°C and 67%RH, both Acrawax

and ZnSt kept their powdery appearance while significant changes in the appearance and texture were observed with Kenolube and Caplube L. Indeed, large lumps or agglomerates were observed for these two lubricants as shown in Figure 7 b). These lumps remained however soft and were easy to desagglomerate with a spatula. Formation of these lumps also observed in pharmaceutical ingredients or food powders could be explained by the higher adsorption of moisture of these lubricants that could lead to the formation of liquid bridges at high humidity level increasing the cohesion of the powder. When the conditioning of these lubricants was performed at a slightly higher temperature (66°C /60%RH) where partial melting could occur, the cohesion strength of these lumps increased and they were much more difficult to desagglomerate. This result explains the presence of lubricant balls or agglomerates in mixes prepared at 80°C.

If the behaviour of mixes containing Acrawax and ZnSt can be easily explained with the moisture sorption and lubricant conditioning test results, it is much more difficult to use them to explain the behaviour of mixes containing Kenolube and Caplube L. Indeed, even if both Kenolube and Caplube L presented a similar and higher sensitivity to humidity based on their moisture sorption isotherms and their appearance and texture after conditioning in the environmental chamber, the flowability of the powder mixes containing these lubricants behave in a different way in high humidity level conditions. Indeed, only mix with Kenolube clearly presented a clear deterioration of its flow properties under high dew point and long mixing time conditions. Mix with Caplube L produced under the same mixing conditions showed no particular deterioration of its flow rate. Even though this result appears surprising, it was confirmed twice by re-producing a second lot under the same conditions. This could be explained by a change in the moisture sorption behaviour of one of these lubricants. In that regard, moisture adsorption test at 50-60°C would be necessary to try to correlate our results with the amount of moisture adsorbed. Indeed, the decrease of flowability at high humidity level should be due to the increase of the cohesion between particles when condensation occurred with the formation of liquid bridges that generate strong capillary forces preventing the flow of the powder mixes. An attempt to obtain the moisture sorption isotherm at 50°C was done to try to confirm this hypothesis, but the laboratory was not able to obtain reproducible results, likely due to the incapacity of the equipment to run such a test at that temperature.

### **PRELIMINARY DEVELOPMENT TO ELIMINATE FLOW ISSUE FOR MIXES PRODUCED UNDER HIGH TEMPERATURE/HIGH HUMIDITY CONDITIONS**

One major objective of that study was to develop a method in laboratory to reproduce problems encountered at the production scale. The method and procedure developed in that study met that objective. Another major objective of that study was to investigate different avenues to eliminate the flow issue encountered with powder mixes containing Kenolube under very humid conditions. While further trying to understand the reasons for the particular behaviour of the Kenolube lubricant, few preliminary avenues were explored and are presented below. Three treatments were evaluated called Kenolube T1, T2 and T3. Details of these treatments could not be disclosed for proprietary reasons. However, the avenues investigated are based on approaches similar to those used particularly in the food or pharmaceutical industries to reduce the sensitivity to humidity of powdery ingredients. These approaches included the use of free flow or anti-caking additives, which have the function to either adsorb excess moisture or reduce the moisture adsorption, or act as a protective coating layer. Figure 8 shows that such approaches enable to obtain powder mixes that maintain excellent flow behaviour even if produced under high dew point conditions (Dew point ~ 50°C, or 60°C/67 %RH) as compared to the powder mix containing only the Kenolube lubricant processed in a conventional way. Optimization is however still required to reduce the negative impact of these treatments on the apparent density of the powder mixes. However, it should be pointed out that apparent density remained at 3.0 g/cm<sup>3</sup> or higher. Obviously, validation of these methods under real production conditions would be required to validate their real benefit.



**Figure 8: Effect of additives and processing treatments on the flowability behavior of powder mixes admixed with Kenolube at 60°C/67 %RH**

## CONCLUSION

In this paper, the effect of relative humidity and temperature on the flow of P/M mixes containing different types of conventional lubricants was studied. Indeed, it is well known in the P/M industry that the behaviour of some production-scale mix formulations changes when produced during humid and hot summer days. The first challenge was to develop a method in laboratory to reproduce problems encountered at the production scale, to be able in a second time to identify avenues to maintain the flowability of these mixes under very humid and hot conditions. This was done using an environmental chamber with humidity and temperature control and by using an unusual and relatively long mixing time of 24 hours that was found to duplicate well the flow behaviour and problems periodically observed in much larger production blending equipment with some lubricated powder mixes.

First, the lubricated powder mixes with zinc stearate and ethylene bis-stearamide (Acrawax) were not affected by humidity and temperature, even in very humid conditions (Dew point ~ 50°C, or 60°C/67 %RH). These results are in good agreement with the low sensitivity to humidity of these lubricants alone, as measured by the moisture sorption and lubricant conditioning tests. At higher temperature (~80°C), slightly better flow and higher apparent density were even measured for these powder mixes. These results reproduce well the behaviour of these mixes at the production scale.

On the other hand, mixes with Kenolube clearly presented a clear deterioration of their flow properties under high dew point and long mixing time conditions. Higher temperature (~80°C) also affected negatively the flowability of these mixes. Moisture sorption isotherm of this lubricant indicated a significant increase of moisture adsorption at high relative humidity. The presence of lumps or agglomerates when this lubricant is conditioned in the environmental chamber at a high humidity level confirmed also the higher sensitivity of this lubricant to humidity that could explain the behaviour of the powder mixes containing this lubricant. Again, these results duplicated well the behaviour of mixes containing Kenolube observed in larger production scale blenders.

Regarding the Caplube L lubricant, while presenting an apparent similar sensitivity to humidity than the Kenolube, the flowability of powder mixes containing this lubricant were not affected by humidity and temperature, even in very humid conditions, if the temperature remains below its melting temperature. However, at temperatures higher than 60°C, the performances of the powder mixes containing this lubricant were deteriorated.

Finally, few preliminary avenues were explored to eliminate the flow issue encountered with powder mixes containing the Kenolube lubricant under very humid conditions. These avenues based on approaches similar to those used particularly in the food or pharmaceutical industries to reduce the sensitivity to humidity of powdery ingredients enabled to obtain powder mixes that maintain excellent flow behaviour even if produced under high dew point conditions. Obviously, validation of these methods under real production conditions would be required to validate their real benefit.

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