A feasibility study to investigate caking in washing powder formulations using a Freeman FT4 powder rheometer [Conference paper]

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A FEASIBILITY STUDY TO INVESTIGATE CAKING IN WASHING POWDER FORMULATIONS USING A FREEMAN FT4 POWDER RHEOMETER

M.C. Leaper¹, E. Fisk¹ and R Browne²

¹. Department of Chemical Engineering, Loughborough University, Loughborough, Leicestershire, LE11 3TU, United Kingdom.
². McBride plc, Park Road, Barrow-in-Furness, Cumbria, LA14 4BN, United Kingdom.

Abstract – Using conventional powder flow analysis methods to quantify the formation of a crust on the surface layer of a powder formed from moisture ingress is a challenge, as cake formation is not uniform throughout the powder sample. This study examined the feasibility of using a dynamic flow tester to compare the caking tendencies of washing powder formulations. A Freeman FT4 powder rheometer was used to investigate crusting caused by 80% relative humidity in washing powder formulations at room temperature. Formulations of 27%, 31%, 33% and 37% sodium carbonate were exposed for 7 days to high humidity before being tested using a twisted blade powder rheometer. Successive test cycles showed the breakdown of agglomerates caused by caking, with the greatest effect on formulations of 27% and 37%. Visual inspection of these powders indicated a hard caking layer of approximately a third of the depth, beneath which free-flowing powder was present. Variations from weak caking, caused by interparticle liquid bridges, were also detected using this method. The study demonstrates the viability of this methodology for optimising new formulations for caking resistance.

1. INTRODUCTION

In recent years, formulations within the laundry powder sector have changed significantly in response to legislation against the use of phosphate-based ingredients [1-2]. These are known as builders, and reduce the effect of calcium and magnesium ions in hard water, enhancing the ability of surfactants to remove grease and dirt. A key substitute for phosphate builders is sodium carbonate, which is cheap and plentiful. However, sodium carbonate in an amorphous form has a high affinity for moisture and is prone to caking. The extra challenges posed by this have prompted several systematic studies, often combining uniaxial tests and Discrete Element Modelling (DEM) [3-7]. Other studies have examined changes in single particle properties as an indicator of trends in bulk powder behaviour [8-9].

There has been a focus on scenarios where particles have been compressed into a single block of agglomerate, sometimes with humid air being passed through the entire structure, and caking has been quantified on the basis of the compact strength. However, where caking occurs only as a crust because of moisture migration alone, a different approach is required. This study explored the feasibility of quantifying unconsolidated crusting with changes in formulation and humidity using a Freeman FT4 powder rheometer.

The Freeman FT4 dynamic test system consists of a twisted blade that rotates first downwards and then upwards through a column of powder, with the resistance over the distance covered used to calculate the energy required to deform the powder, as shown in Figure 1; this is called the Basic Flow Energy (BFE) and can be used to compare a new powder against the behaviour
of a standard powder [10-11]; it is usually within the range of 500-5000mJ. The test has often been performed in combination with a shear cell test using the same equipment. Conventionally a full dynamic test consists of 11 cycles where the blade tip rotates at 100 mm/s for 8 cycles and then decreases to 70, 40 and 10 mm/s for the final three cycles.

![Figure 1: The basic principle of the Freeman FT4 powder rheometer [10]](image)

Powders which show a strong tendency to de-agglomerate show a decrease in BFE with successive cycles, along with a reduction in the rate of decrease; the ratio of the BFE in cycle 1 to cycle 7 is defined as the stability of the powder. If the stability value is below 0.9, this indicates a significant tendency for the powder to de-agglomerate. The presence of surface crusting could cause this result. Using this methodology, this study aimed to measure the de-agglomeration of caked material that had been exposed to 80% relative humidity, with the expectation that only the top layer had been caked. The number of cycles was increased to 25 with a constant tip speed of 100 mm/s, enabling any subsequent degradation of the primary particles to be detected.

2. MATERIALS AND METHOD

2.1 Test Materials

The test materials were ideal detergent formulations where 47% by mass of the powder consisted of a combination of sodium carbonate and sodium sulphate. Table 1 summarises the combinations used in the study:

<table>
<thead>
<tr>
<th>% Sodium Carbonate by mass</th>
<th>% Sodium Sulphate by mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>27</td>
<td>20.52</td>
</tr>
<tr>
<td>31</td>
<td>16.52</td>
</tr>
<tr>
<td>33</td>
<td>14.52</td>
</tr>
<tr>
<td>37</td>
<td>10.52</td>
</tr>
</tbody>
</table>

Table 1: Formulation of test samples by mass

Sodium sulphate is a filler within the formulation, reducing the costs and enabling better flow properties. Figure 2 shows the particle size distributions of formulations containing 27% and
37% sodium carbonate prior to the investigation, obtained by sieving, with the difference being attributed to the variations in the proportions of sodium carbonate and sodium sulphate.

Figure 2: Particle size distributions of test formulations containing 27% sodium carbonate and 37% sodium carbonate respectively, obtained using sieves.

2.2 Test Method

The BFE for the test materials was obtained using the FT4 dynamic rheometer method described by Leturia et al. [11]. This involved filling a 50mm split cell with the test powder, as shown in Figure 3. The powder was then brought to a consistent initial packing configuration by exposing it to one cycle of the rotating blade. After this, the top part of the powder was removed by sliding the top portion of the split cell.

Figure 3: How the sample is prepared using the split cell [10].

At this point, the BFE test could be performed immediately in the case of an “initial state” test or the sample could be placed in controlled environment to assess the effect of humidity. For this study, the samples were placed in an 80% RH environment at 20°C, with the maximum exposure time being 7 days. This approach also enabled multiple samples to be exposed simultaneously. The samples were then exposed to repeated cycles with the rotating blade, with the BFE measured for each successive cycle.
3. **RESULTS AND DISCUSSION**

Figure 4 shows the Basic Flow Energy plots of the formulations at before exposure to high humidity. The plots show little difference between the different formulations 31-37%, although there is evidence of some initial agglomeration of the powder at room conditions. The values shown are the average of three experiments for clarity. It also shows that 27% sodium carbonate shows slightly more resistance than the other formulations. It also indicates that after 25 cycles there is no degradation present.

![Figure 4: BFE values for formulations containing different concentrations of sodium carbonate by mass.](image)

Figure 5 compares the initial values in Figure 4 with the equivalent systems after 7 days of the split cell being exposed to 80% RH.

![Figure 5: A comparison of BFE values for the test formulations after 0 days and 7 days at 80% RH. The 7 day results are denoted by _7.](image)
Figure 5 shows that the formulations at 27% and 37% sodium carbonate were affected the most by exposure to high humidity, although all formulations showed a higher BFE after 7 days. Visual inspection of the 27% and 37% samples after 7 days indicated that the top 35% of a container of powder was agglomerated and required almost complete inversion to remove the powder; below this agglomerated layer the powder was still free-flowing.

During the FT4 BFE test, the increased resistance to the blade was caused by two mechanisms:

i) The crust formed on the top of the powder due to the moisture uptake and chemical reaction between ingredients induced by the extra moisture, i.e. hard caking. This was at its strongest with the initial cycle, but the crust was then mixed into the powder with progressive cycles, transferring the main resistance from the surface to the bulk of the powder. These agglomerates that were dispersed through powder were responsible for continuing the characteristic curve of de-agglomeration.

ii) Inter-particle bridges of moisture also held particles together with a weak force, contributing where hard caking had not occurred. The moisture could also be dispersed throughout the bed with progressive cycling. Figure 4 clearly shows that all of the formulations had an immediate affinity for moisture and these bridges were formed at atmospheric conditions in the powder prior to the test. An absence of the stronger agglomerates accounted for the lower strength of the powder that had not been exposed to high humidity.

The results also show that 31% and 33% sodium carbonate formulations are less prone to crusting at high humidity, suggesting an increased resistance to caking. The interactions between the ingredients within the formulations, moisture and carbon dioxide are complex, giving products such as sodium percarbonate and sodium sesquicarbonate. However, these results show that at an optimised combination of ingredients, the effects of moisture-induced caking can be minimised.

4. **CONCLUSION**

Using the Freeman FT4 to assess surface crusting is useful for the comparison of multiple samples and indicates conditions which would require further investigation using other methods. The concentration combinations of 31% sodium carbonate/16.5% sodium sulphate and 33% sodium carbonate/14.5% sodium sulphate showed more resistance to high humidity at room temperature. Formulations with more and less sodium carbonate showed an increased tendency to moisture uptake and caking, with sufficient strength to form a solid crust that could only be removed by inverting the container. However, further study is required to assess the effect of other ingredients and the effect of temperature.

5. **REFERENCES**


6. **ACKNOWLEDGEMENTS**

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