



## Caking of Lemon Flavour Powdered Drink Using DVS Humidity Cycling

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***This paper demonstrates the effect of relative humidity pulse length and cycling methods at different temperatures.***

### Introduction

The moisture sorption properties of foods are critical for their shelf-life stability. This is especially true for powdered products which are vulnerable to either temperature or humidity shocks. Products can cake, become sticky and collapse thus rendering them unsaleable.

### Method

Lemon flavour Kool Aid (a powdered beverage popular in North America) was purchased from a grocery store in the United States. Lemon was chosen as a relatively inert flavour unlikely to contain any components that will plasticise the carrier matrix.

### Results

Figure 1 shows gravimetric data for Kool Aid at two different temperatures. The powder was initially equilibrated at 20% RH for two hours followed by a thirty minute pulse of humidity at 70%RH. This is followed by a return to 20% RH. In one case, the powder is subjected to a second burst of relative humidity. The relative humidity cycle is shown in black in Figure 1. The percentage weight change for the powder is

shown for two different temperatures. The 25°C test is shown in blue, the 35°C test is shown in red. At the lower temperature, the sample takes up nearly 2% moisture during the relative humidity pulse. Although most of this is released, the powder retains an additional 0.4% moisture even after returning to its previous equilibrium relative humidity. This situation worsens with the second burst of humidity and it appears that additional moisture will be retained afterwards. At 35°C, the moisture uptake is nearly 5% during the 30 minutes spent at 70% RH. This is sufficient to change the state of the powder: it had gone sticky at the end of the test. The moisture release is very slow.

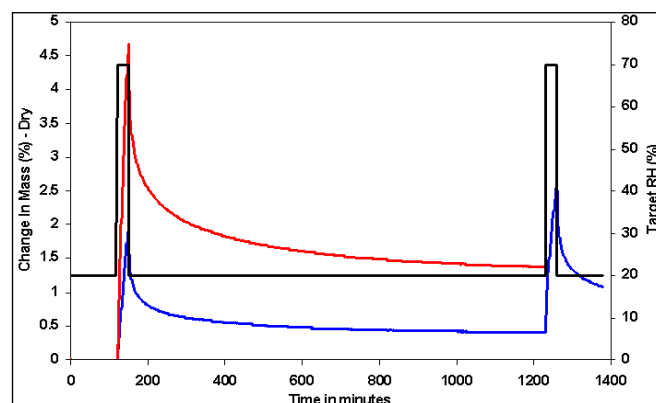


Figure 1. Effect of relative humidity spike on lemon flavour Kool Aid powder



This approach is developed further in Figure 2. On this occasion, the temperature was maintained at 25°C throughout the test and the time of exposure at 70%RH was varied. This test was undertaken a total of five times on fresh sample. After an initial exposure at 20% RH for equilibration, the briefest exposure time was just two minutes (the percentage mass change is shown in red in Figure 2). The moisture uptake is negligible and the sample returns to equilibrium rapidly. When the exposure time is increased to five minutes (dark blue line), the moisture uptake is a few tenths of a percent but the powder does seem to hold on to some of this water irreversibly. This phenomenon can be followed with longer exposure times of thirty minutes (green line), sixty minutes (purple line) and three hours (light blue line). As the exposure time increases, the moisture uptake increases and the powder retains more water. One hour and longer exposure at 70% RH cakes the sample.

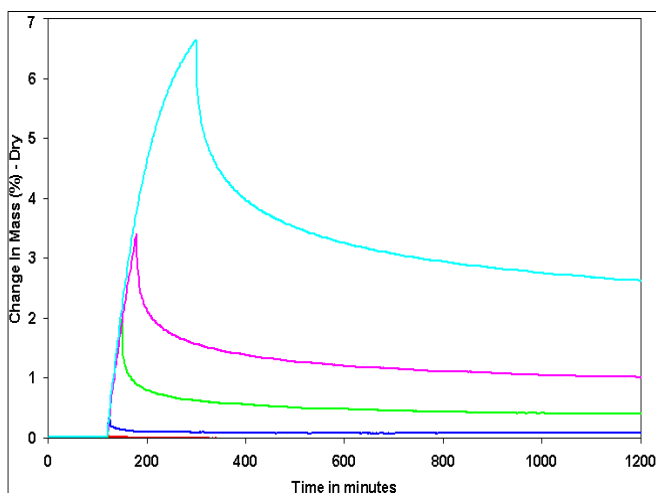


Figure 2. Water pickup by lemon flavour Kool Aid at 25°C exposed to different length 70%RH pulses.

An interesting alternative is to examine the stability of the powder against repeated relative humidity cycles. This is an interesting feature of

DVS performance and is useful for illustrating the effects of cumulative damage due to relative humidity. This could simulate the daily fluctuations in ambient relative humidities in some countries or the repeated movement of stock into and out of a warehouse environment. Humidities were chosen that would span a sufficient range to severely challenge the sample but not lead to collapse during a single event. As shown in Figure 3, the humidities selected were 20% and 70% RH and the ramp rate was 20% RH per hour. Twelve cycles were completed (shown in blue) and the response of the sample is shown in red. It is clear that the powder gains more moisture as a result of each relative humidity cycle.

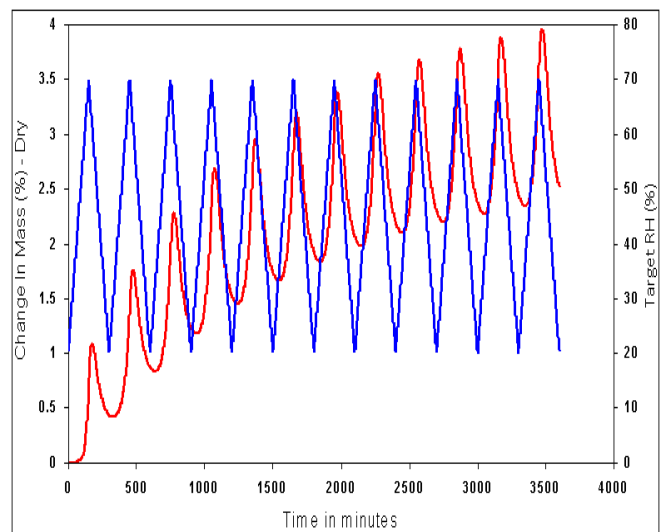
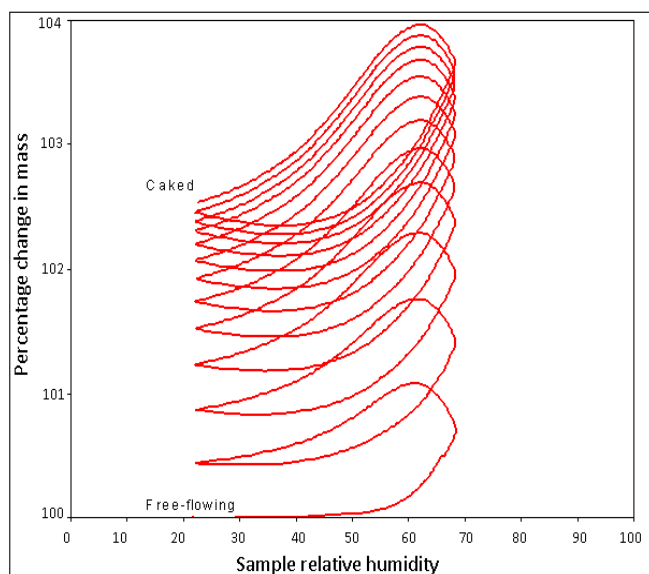


Figure 3. Water pickup during relative humidity cycling.

This is also re-plotted in Figure 4. The hysteresis between sorption and desorption is a way to visualise the product stability, especially since the data from Figure 3 can be converted to a series of hysteresis loops. Repeated cycling changes the physicochemical state from free flowing to caked.



*Figure 4. Multiple hysteresis phenomenon for Kool Aid powder*

## Conclusion

The flexibility of the DVS software enables caking measurements to be undertaken easily. The stability and shelf life can be characterised.

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