The hand squeeze test. Is this the right way?
BACKGROUND

Company “A” produce a range of tablets as food supplements from natural materials, plant extracts and other natural food sources. Most tablet formulations work well and company “A” has a good reputation for their products. Generally they do not encounter problems with the tablets breaking or disintegrating during manufacture, packing or storage.

However, one product using a particular plant extract tablet was constantly giving problems. The manufacturing process consisted, in part, of manual hand mixing by a group of four operators of all the formulation ingredients in a large pot. The mixing was continued until the product appeared to be homogeneous. As this was a time consuming and labour intensive process “overmixing” was not an issue. This same process was used for other products for which this low “intensity” of mixing seemed to be appropriate and gave good results.

THE PROCESS

However the rheological properties of this “hand-made” granulation in relation to the mixing intensity or the energy supplied in the mixing process had never been considered.

Using the Caleva Mixer Torque Rheometer (MTR-3) the rheological properties of this particular granulation were examined in a quantitative way to see if extending the granulation time or intensity of this product would have a significant impact on the consistency of the formulation and properties of the final product. With some formulations, mixing intensity can have a significant effect on granulation properties. From a practical standpoint this was a relatively simple and quick task using the Mixer Torque Rheometer.
The assessment was made utilizing a 20g sample of the ingredients that make up the dry powder mixed together with the appropriate quantity of liquid binder (in this case, water). The instrument was used to measure the consistency of the material in the bowl as the granulation progressed. Idealised results the pattern of what was obtained are shown over the page.

THE RESULTS

The results (Figure 1) show that with this particular formulation the consistency remained at a fairly low level for a significant period after all the constituents of the formulation appeared to be uniformly mixed but with additional mixing the consistency rose rapidly.

![Diagram](image)

**Figure 1.**
A diagrammatic representation of results obtained from the MTR showing the consistency of a nutritional formulation with an extended granulation time.

The results showed that in this particular case the granulation regime is of paramount importance to achieve the required product quality. With the additional granulation time the consistency of the formulation showed a dramatic increase in the measured consistency. The cohesiveness between the particles in the mix became significantly stronger demonstrating that additional and continued work done to the apparently homogeneous mix were essential to achieve a formulation with sufficient cohesiveness to produce an acceptable tablet.
CONCLUSIONS

The main conclusions that can be drawn from this case study are:-

1. Quantifiable measurements of formulation consistency are desirable, and should be considered an essential part of formulation development. Lack of information of what is happening in the formulation during granulation can be problematic as demonstrated in this case study.

2. The granulation program can be a significant factor in establishing formulation properties. When used as an integral part of formulation development processes, the Caleva Mixer Torque Rheometer can be instrumental in avoiding downstream problems in production. This collection of quantitative data is recommended as a standard feature of the development process.

The Caleva Mixer Torque Rheometer. The only way to quantify the rheology of a wet mass

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