



SCIENTIFIC AND ENGINEERING TEST RESULTS

EXERCISE: Logic-related Questions About the Learning Report

Answer the following questions about the logic and content of the before and after Learning Reports that follow. See how long it takes you to answer these basic questions in the initial report. Then see how quickly you can find them in the revised report.

1. What are the technical objectives for this research?
2. What criteria will the author use to decide if those objectives are met?
3. Why is it important to achieve those technical objectives?
4. What did we learn from the research relative to those objectives, and on what are those learnings based?
5. What should we do next?

Before and After Technical Reports

Original Report:

Title: Study of Bermuda Mixtures in 2000 ml Glass Reactor

Description of Testing: Bermuda components were combined in a 2000 ml glass reactor, duplicating order of addition and ratios for the Bermuda XP production at Phoenix. The components were heated and cooled (nil perfume and with perfume) while making visual observations, measuring particle size by Snapshot, and recording resistance to shear by torque on the agitator shaft with a pitched blade turbine. No baffles were used. The trials included two perfume candidates, Zappo and White Meteor.

Bermuda base was added to the reactor and heated to 90°C. Clearsolv was then added and the mix reheated to 90-95° and the Dimax was added. This matrix was then heated to 110°. The reactor jacket temperature was quickly dropped to 55° to begin the cooling cycle. The matrix was cooled until agitation was hardened to the degree agitation was no longer attainable, and then reheated to 105°. Perfume was then added, the matrix reheated to 110° and the cooling cycle repeated.

Discussion:

Phase Transition

When cooling the matrix from 110° (nil perfume), visible clouds appear at about 80°C. At 68° it is opaque white, and at 60° it is unflowable (Fig. 1-3). During this phase transition, Snapshot recorded the presence of solids, nearly all less than 120 microns (Fig. 4). On reheating the matrix, melting appeared complete at 80°C. When perfume was added, and the hot mix was again cooled, the cloud point shifted down to around 66°. While the product continued to go through a phase change (as the matrix did), there was no response on the Snapshot probe (Fig 1-3).

Torque

The measurement of solids by Snapshot during cooling of the matrix (nil perfume) was accompanied by an increase in torque measured at the agitator shaft. When the matrix with perfume was cooled, there was little increase in torque even though there was a phase transition (Fig. 5).

Dimax Addition

In the Phoenix trials, Dimax particles were observed in the matrix, at temperatures as high as 110°. In the initial trials in the beaker, this same behavior was observed with agitation at 400 rpm. When agitation was increased to 550 rpm, the Dimax dispersed almost immediately. The higher agitation did increase the amount of observed air in the system. The level of observed air dropped significantly with the addition of perfume, but gradually increased again through time under agitation.

By changing the order of addition to Bermuda base first, and Dimax second (no Clearsolv), the Dimax melts and disperses quickly at 195°C with gentle stirring. This was done offline in a smaller beaker trial and was not confirmed in the 2000 ml glass reactor.

Phase Separation

In a separate trial, agitation was stopped at increments of 05°C during the cooling cycle (matrix + perfume) from 85° to 65°. During these five-minute holds, no phase separation was noted, including no Dimax observed. At 65°, the normal



clouding began to appear with or without agitation.

Heat Transfer

During cooling, the rate of temperature change is about the same for the matrix as it is for the matrix + perfume. While the data suggests there may be some exotherms for both the matrix and the matrix + perfume, a more controlled run for Calorimetry purposes is suggested. The two perfume systems behaved similarly to each other.

The highest cooling rates were seen at the highest dT between the jacket and the product, and the rate decreased asymptotically as the dT decreased (Fig. 1-3).

Recommendations:

The lack of Snapshot counts during the phase transition of the matrix + perfume mix suggests a real difference in the way this transition is occurring. By observing samples of both systems (matrix, matrix + perfume) under a heated-stage microscope at varying temperatures, an understanding of this phenomenon is possible. The lack of torque increase for the matrix perfume system may be accounted for by Ms. Maple's conclusion that the system is shear-thinning.

The shift down in cloud point for the matrix + perfume system may help in scale-up design. Since the degradation of perfume is a concern, determining the lowest practical temperature in the filling system can be used to reduce perfume loss.

More controlled glass reactor runs can help define the heat transfer properties, including heats of crystallization, if they are present.

Order of addition and/or mixing shear should be investigated as means to keep Dimax dispersed.

(Several attachments included)



Revised Technical Report

Processing Temperatures for Bermuda XP

This report summarizes results of testing to establish preliminary temperature minimums needed to process Bermuda XP with two leading perfume candidates (Zappo and White Meteor). Specifically, we determined minimum temp before and after perfume addition needed to ensure full dispersion of components, in-process phase stability, and pumpability. We also identified potential ways of reducing temperatures before perfume addition.

Processing Bermuda XP at the lowest possible temperatures is important because it will reduce manufacturing cost by reducing energy use and processing time needed to heat and cool the mixture. It will also help protect product quality and performance by reducing perfume degradation during processing.

BACKGROUND

Bermuda XP, the leading candidate to replace Bermuda AX, requires significantly different processing conditions than AX because XP uses a significantly different matrix to deliver perfumes. XP trials at Phoenix have shown that processing temperature is a critical variable, and that the XP matrix behaves differently after perfumes are added.

METHOD OVERVIEW

We conducted a series of bench-scale experiments monitoring ingredient dispersion, phase stability, and bulk viscosity of a simplified Bermuda XP base vs. temperature.

Mixer: The mixing vessel for most tests was a jacketed, baffle-free 2000 ml glass reactor with a pitched-blade turbine that has accurately simulated Bermuda mixing in the past.

Dispersion: We monitored ingredient dispersion with Snapshot, and used visual observation to detect phase stability.

Torque: Torque on the agitator shaft served as proxy for bulk viscosity.

In a typical batch,

1. Bermuda base was added to the reactor and heated to 90°C.
2. Clearsolv was then added and the mix reheated to 90-95°C before the Dimax was added.
3. This matrix was then heated to 110°C.
4. The reactor jacket temperature was quickly dropped to 55° to begin the cooling cycle.
5. The matrix was cooled until agitation was no longer possible, and then reheated to 105°.
6. Perfume was then added, the matrix reheated to 110°C, and the cooling cycle repeated.

Attachment 1 describes the method in more detail.

KEY FINDINGS – Temperatures Before Perfume Addition

- 1. Processing temperatures up to 110°C may be needed before perfume addition, limited by the ability to disperse Dimax.** With the current order of addition and agitation level, Snapshot showed undispersed Dimax particles up to 110°C, at which point the Dimax melted and dispersed (Fig. 2). This was also consistent with observations during previous testing at Phoenix. Since full Dimax dispersion is needed for the product to perform, this establishes a minimum processing temperature under these conditions.
- 2. Neither phase stability nor pumpability limits processing temps under these conditions.** All batches remained phase-stable over the range of conditions tested, and bulk viscosity remained in the pumpable range at temperatures well below 110°C (Fig 3).
- 3. Some reduction in processing temperature may be possible with a revised order of addition or with increased agitation.** In a smaller scale experiment, Dimax dispersed at 95°C when added before Clearsol. In another experiment, increasing agitation from 400 rpm to 550 rpm resulted in almost immediate Dimax dispersion at 85°C, although with higher levels of entrained air in the mixture.

KEY FINDINGS – Temperatures After Perfume Addition

- 1. After perfume addition, temperatures as low as 65-70°C may be possible.** Dispersion, phase stability, and pumpability all look good in this temperature range.
 - Dispersion lasted until product was cooled to 66°C after perfume addition, as indicated by Snapshot readout and confirmed by the absence of cloudiness.
 - A separate trial showed that larger-scale phase stability was also acceptable down to about 70°C. In this trial, agitation was stopped at increments of 5°C during the cooling cycle (matrix + perfume) from 85° to 65°C. During these five-minute holds, no phase separation was noted, including no Dimax separation. At 65°C, the normal clouding began to appear with or without agitation.
 - Pumpability was also acceptable down to 65°C. As the matrix + perfume mix was cooled, viscosity remained nearly constant (Fig. 5).
- 2. Both perfumes behaved similarly in all tests.**

NEXT STEPS

1. By August 1, Corporate Engineering will evaluate the potential for the revised order of addition and increased agitation, separately and in combination, to allow full Dimax dispersion at lower temperatures. This includes assessing the impact of entrained air from increased agitation.
2. If temperature reductions are possible, we will confirm that phase stability and pumpability are still acceptable at the new conditions.
3. We will continue to monitor temperature requirements after perfume addition.

(Several attachments included)