



Brookfield Viscosity Test Development for IMX Formulations (Abstract # 17284)

NDIA Insensitive Munitions & Energetic Materials Technology Symposium 2015

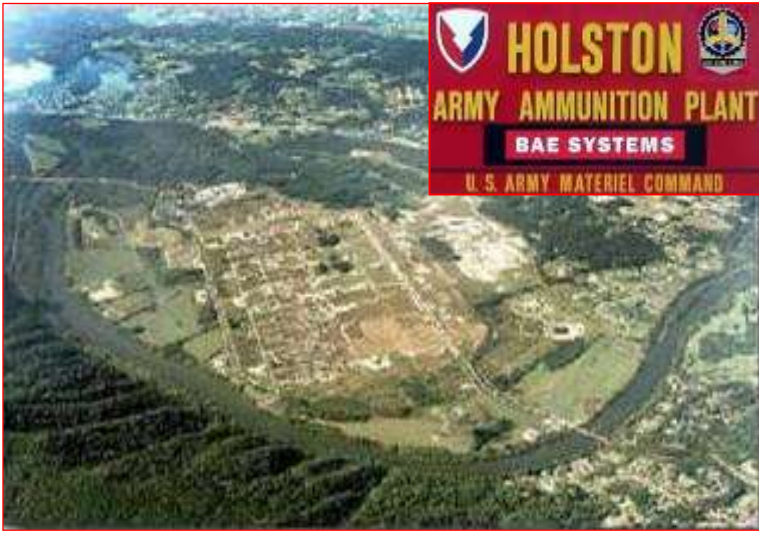


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Presentation Outline

Brookfield Viscosity Test Development for IMX Formulations

- Background
- Program Objectives
- Technical Approach
- Analysis and Evaluation
- Summary
- Future Work
- Conclusion



Brookfield Viscosity Development - Background

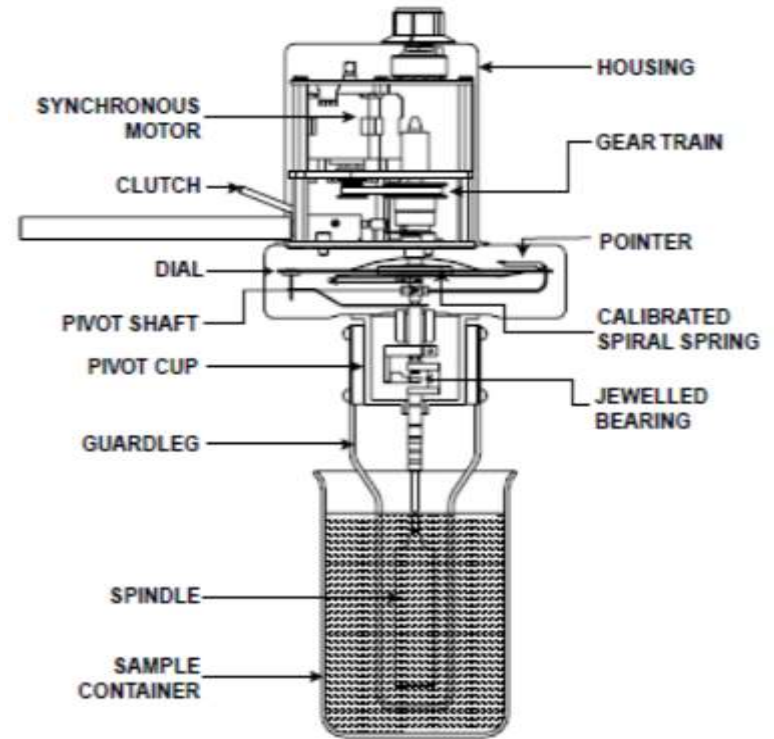
- Efflux Viscosity
 - Originally developed for TNT based formulations (Composition B)
 - Variability: Operator to Operator
 - Measurement system (Timer)
 - Procedure (Interpretation)





Brookfield Viscosity Development - Background

- Rotational Viscometry
 - Measure the torque of a spindle immersed in a material spinning at a constant speed
 - Torque of the melt pour sample over a set duration determines the viscosity of the sample





Brookfield Viscosity Development – Program Objectives

- Development of Test Method for measuring Brookfield Viscosity
- Gage R&R study with 3 test sites
 - Repeatability & Reproducibility



Why is Viscosity Important for Melt Pour Formulations?

- Quality Control
 - Raw Materials – Indirect measurement of product consistency
- Processability
 - Direct assessment of how well the Formulation will perform in loading operations



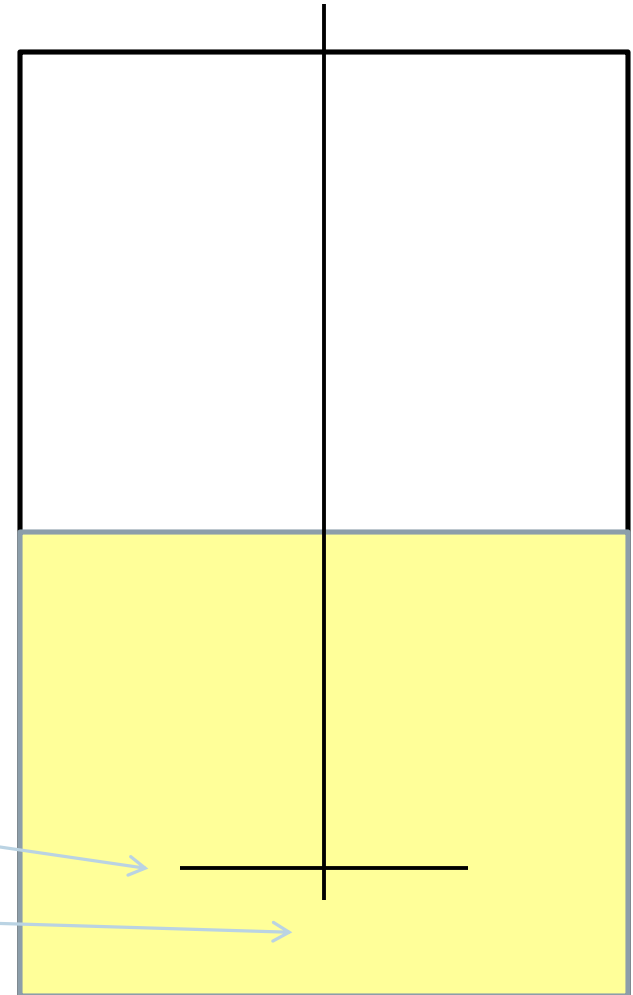
Brookfield Viscosity Development – Technical Approach

Sample Preparation:

- Spindle is placed so that material surrounds the spindle completely as recommended by Brookfield Engineering
- Additionally enough material is used to submerge the spindle to the correct level
 - The spindles have a notch indicating correct level
 - The T-Bar Spindles do not have this requirement

Space between wall and spindle

Space between bottom and spindle

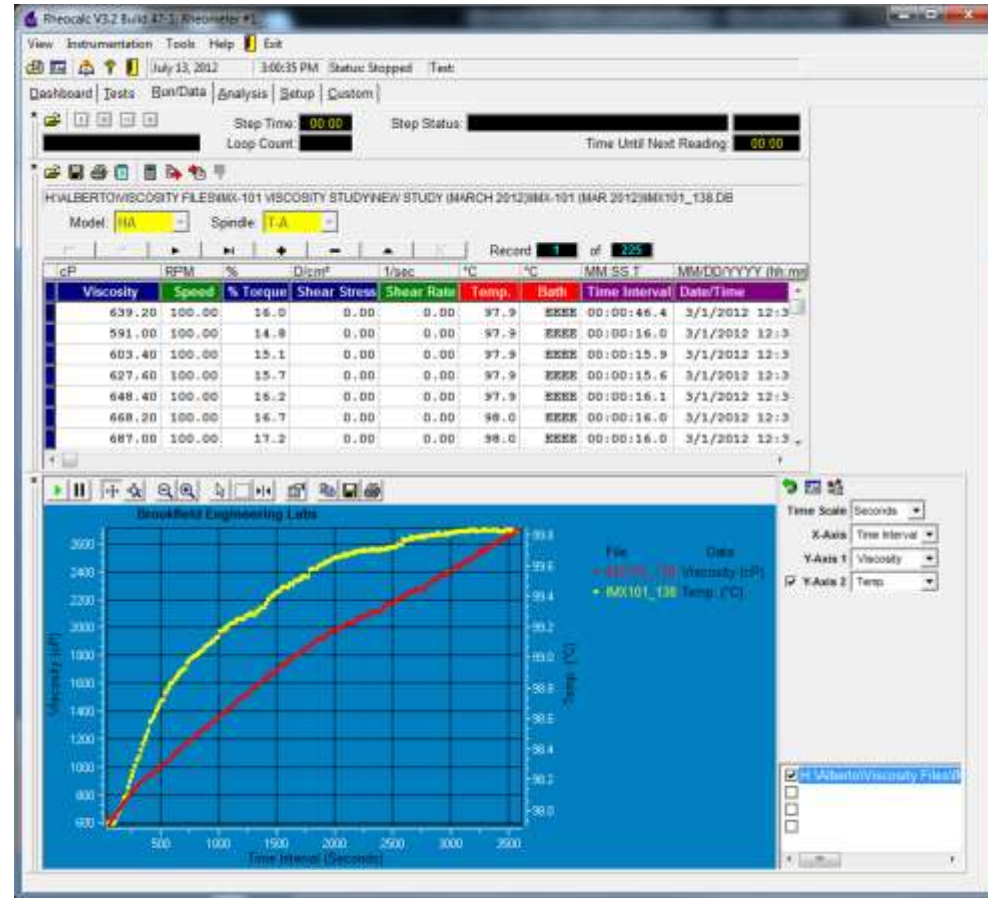




Brookfield Viscosity Development – Technical Approach

Data Collection – Rheocalc 32

- Software used to automate data collection
 - Data is available continuously
 - Collected 1 Data point per second
 - Data from 15 – 45 seconds averaged and used as initial viscosity value
 - What is recorded?
 - Calculated viscosity
 - Speed
 - % Torque
 - Shear Stress
 - Shear Rate
 - Temperature
 - Time Interval
 - Real Time Plot of Viscosity and Temperature over Time



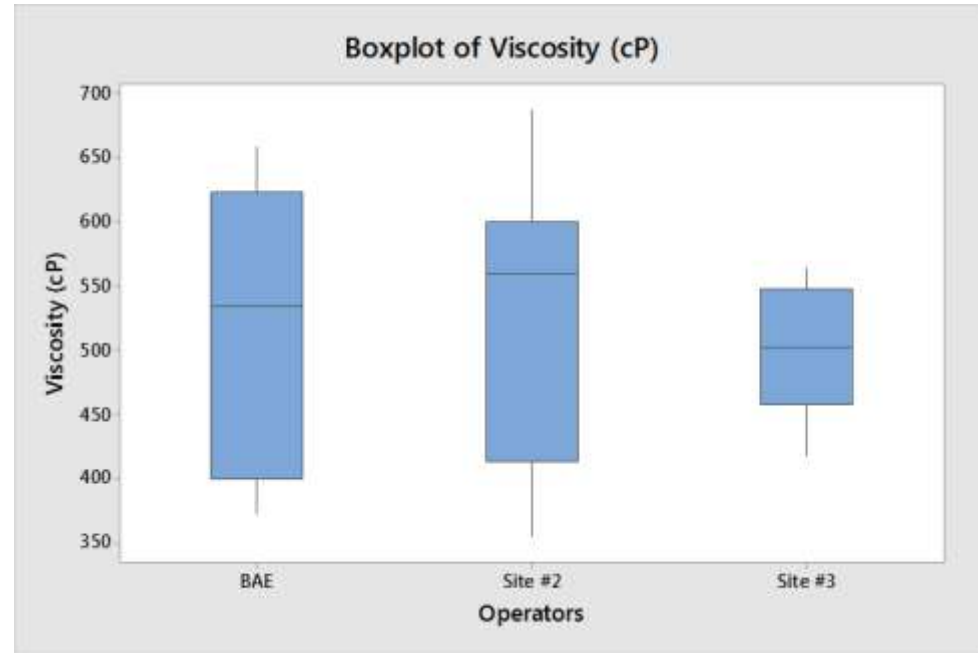


Brookfield Viscosity Development – Analysis and Evaluation

Gauge R&D Study

- Boxplot shows variability between sites

The overall distribution of data for Site #3 is significantly lower in value than the values obtain by the other sites



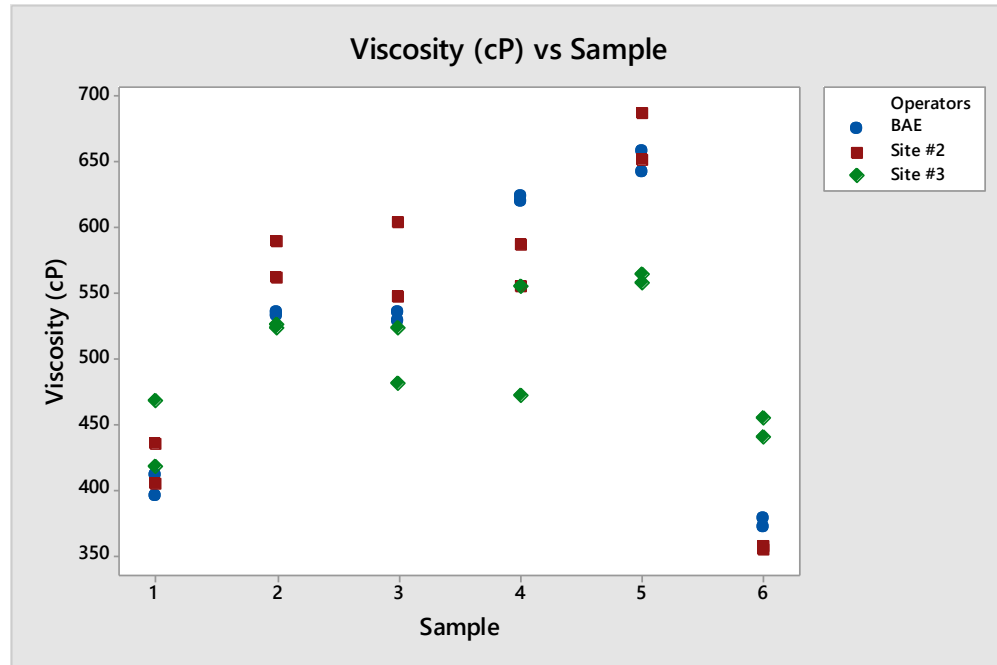


Brookfield Viscosity Development – Analysis and Evaluation

Gauge R&D Study

- Scatterplot shows variability between sites

Differences in the measured viscosities for samples 4, 5, and 6 contribute to the lower overall distribution of the data collected by Site #3



Further examination of the data also indicates a difference in the temperature of the samples during the data collection

Site	Site #1, BAE	Site #2	Site #3
Average Temperature (°C)	100.6	98.8	106.4

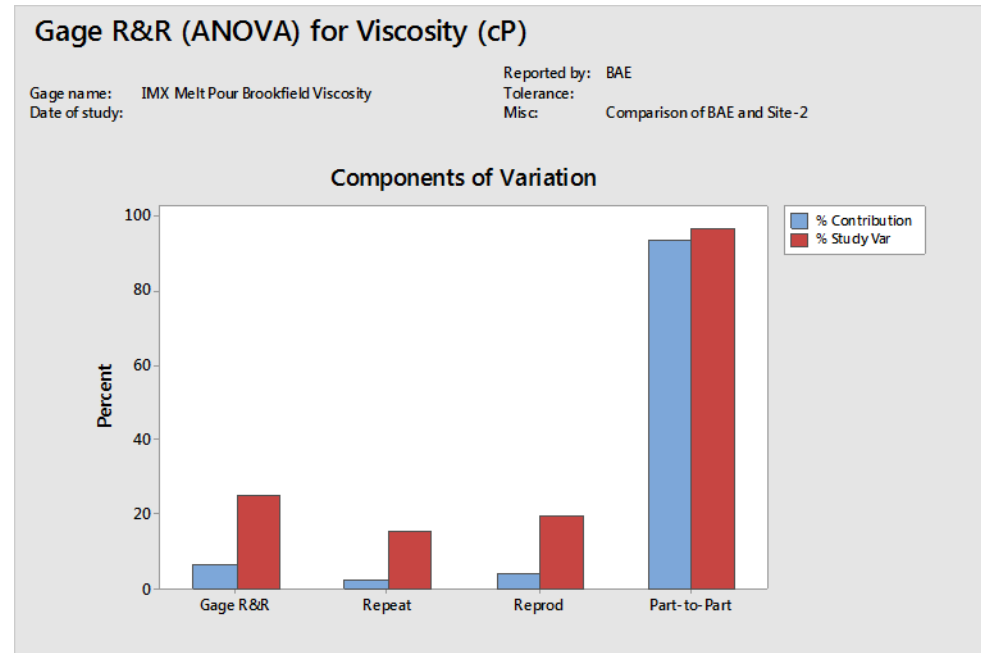


Brookfield Viscosity Development – Analysis and Evaluation

Analysis of Variance (ANOVA)

Part-to-part contribution is much higher than that of Repeatability & Reproducibility

This indicates that the measurement system has little variation within laboratory (repeatability) and between laboratory (reproducibility) measurements



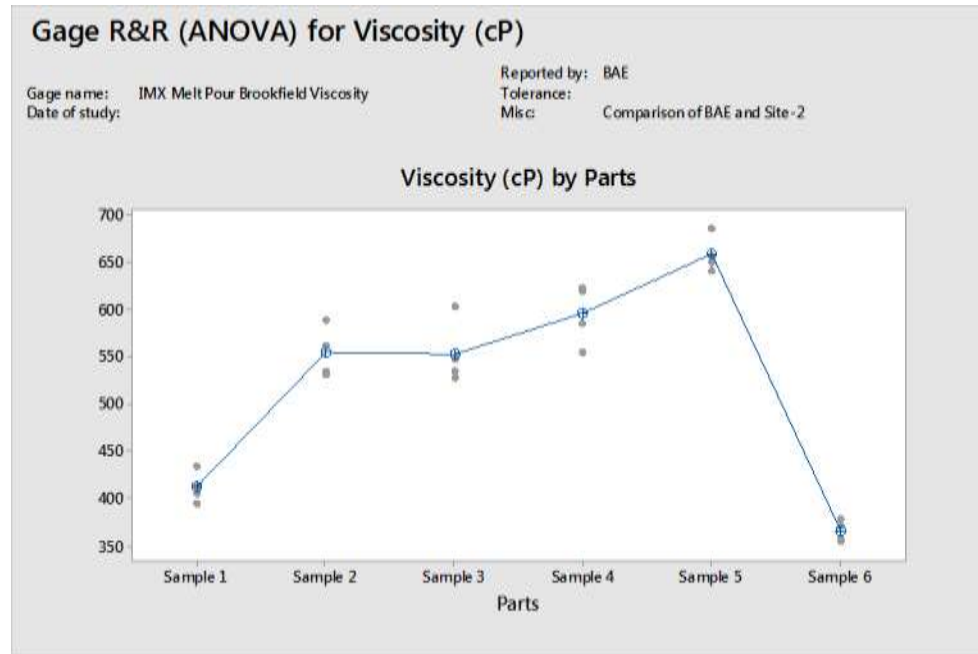


Brookfield Viscosity Development – Analysis and Evaluation

The measurement by part graph summarizes the repeatability of the measurements.

A tight grouping of values for each part indicates that the repeatability for the individual parts is fairly high and contributes only slightly to the variation in the measurement system

Additionally it shows that the variations in the measurements are mainly occurring due to variations in the parts themselves.



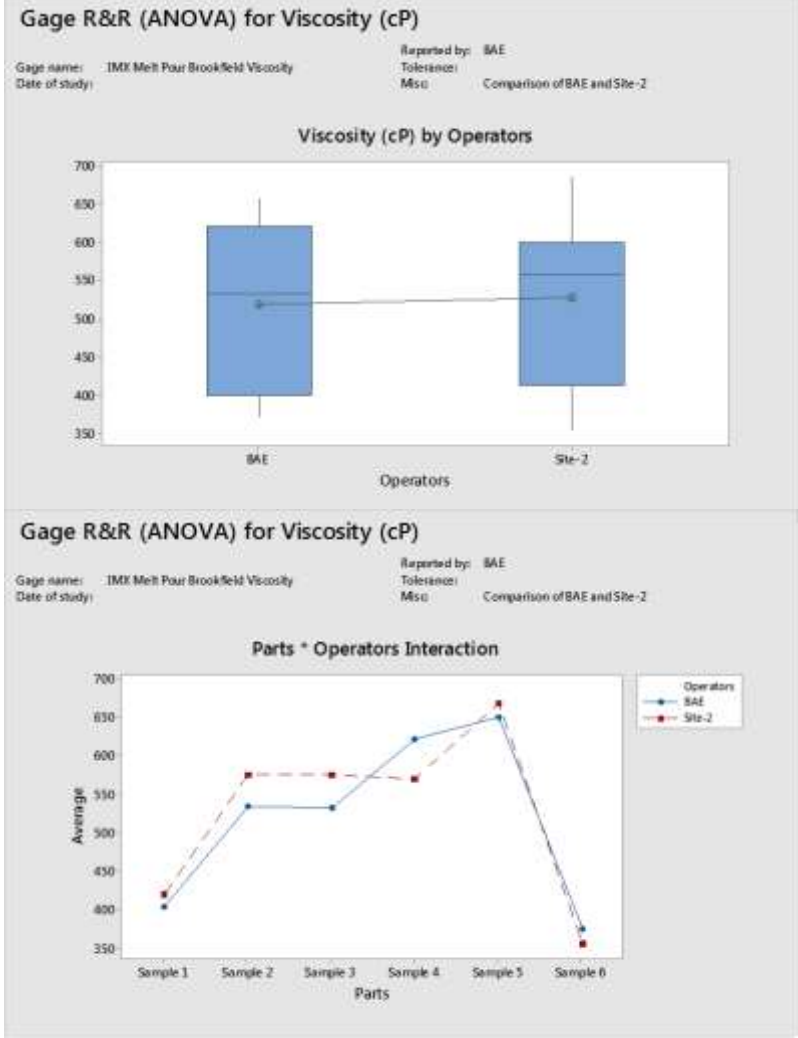


Brookfield Viscosity Development – Analysis and Evaluation

- Slight variations in the viscosity occur between the two sites.
- Variation is quite low
- The main source of variation in the operators is due to part-to-part variations
- The results in these two graphs together strengthen the conclusion that the largest contribution to the variation is in variations in the part (batch).

Two-Way ANOVA Table With Interaction

Source	DF	SS	MS	F	P
Parts	5	252735	50546.9	37.4703	0.001
Operators	1	428	427.6	0.3170	0.598
Parts * Operators	5	6745	1349.0	4.2204	0.019
Repeatability	12	3836	319.6		
Total	23	263743			





Brookfield Viscosity Development – Summary

- Largest variability is found in part-to-part variation (Batch)
- 6.35% = Total gage R&R contribution
($< 9\%$ = measurement system is adequate)





Brookfield Viscosity Development— Future Work

Improvements:

- Minimizing sample temperature variations
- Better standardized data collection
- Additional sites for improved Gage R&D
- Efflux Correlation





Conclusion

Use of the Brookfield Viscometer shows promise as an alternative to Efflux Viscosity as the gage R&R study indicates.





Questions?

