MASTER

LX-13 COMPONENT DEVELOPMENT SCALE UP EVALUATION

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MANUFACTURING ENGINEERING DIVISION

Normal Process Development
Endeavor No. 102

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ABSTRACT

The intermediate phase of the continuous PETN process development has been completed. After the initial development of the process on a small scale, the intermediate step was to determine if the process will scale up to a production lot size and still maintain adequate performance. Preliminary data indicate that this process is feasible on a production scale. Three 18-kg lots (production size) showed good repeatable performance, firing on all tracks while maintaining acceptable extrudability.

INTRODUCTION

The Development Division is working in conjunction with the Manufacturing Engineering Division to develop new methods for preparing quantities of PETN for use in the formulation of LX-13. In the past, difficulties were encountered in production when the raw material lot of PETN was changed. Many lots of LX-13 have been rejected because of changes in properties due to the differences in parent lots of PETN. The problem may possibly be alleviated by continuously producing a consistent product. The development approach, using a continuous mixed-suspension mixed-removal recrystallizer, has been tested and found to produce acceptable PETN on a small scale(1). More work was needed to further refine processing parameters and to allow scale up to production scale batches. Therefore, two small (4 kg) and three large (18 kg) PETN batches were formulated at a low temperature to further evaluate the process.

DISCUSSION

Two small (4 kg) PETN batches were formulated to compare with previous small PETN batches. Three large (18 kg) PETN batches were then formulated to reproduce the performance of the small batches on a larger scale. Preliminary data indicate that the larger batches' performance is reasonably close to that of the smaller batches.

The five PETN batches were precipitated by the continuous method for evaluation use. All batches were made at reduced temperatures using thermally purified PETN as a starting material. Formulation parameters for these batches are summarized in Table I. All PETN batches were dried in a recirculating air steam-heated oven at 66 C for 36 hours. Samples were obtained and air permeametry [$FSSS (S_o[P])$] and gas adsorption [Perkin-Elmer ($S_o[G])$] surface areas were obtained for each batch.

LX-13 lots were formulated in production equipment from each of the PETN batches listed and were subsequently tested for extrudability and firing performance. The results of these tests are presented in Table II. A typical production LX-13 lot made from batch-precipitated PETN (the last batch in Table II) is included for comparison.

(1) J. C. Adams, A. G. Osborn, T. L. Stallings, "LX-13 Processing," MHSMP-76-17A, January - March 1976

The first two LX-13 lots listed in Table II show a decided improvement in extrudability with 25.9 mm (6040-01) and 28.2 mm (6041-01) being recorded in the 15-mil tracks. PETN Batch 6040-01 was an attempt to reproduce another batch that did not mill due to excessive moisture(1). Batch 6041-01 was an experimental batch made to test the effect of a lower temperature (4 C) on the performance of the LX-13. Batch No. 6041-01 had the highest extrudability observed on the 15-mil track (28.2 mm) for thermally-purified PETN. This LX-13 lot also fired successfully. Indeed, all four tracks fired at over 7.3 km/s, with the 35-mil track firing at 7.41 km/sec.

Three 18 kg batches which correspond to the production PETN batches (the third, fourth and fifth batches listed in Table II) were produced using thermally purified PETN with a constant water-to-HE solution ratio. Each batch was produced at the ratio of 3.28 cm water to 1 cm solution at the low temperature (4 C) with the small (32 mm OD) impeller, slow impeller speed (1000 RPM) and low flow rates. PETN batch number 6048-01, the third batch listed, gave acceptable extrudability (20.3 mm in the 15-mil track) with detonation velocities ranging from 7.301 km/s in the 15-mil track to 7.425 km/s in the 35-mil track. Batch 6125-01, the fourth batch listed, gave very nearly the same performance as Batch 6048-01. Batch 6127-02, the fifth batch listed, also performed similarly to Batch 6048-01, with the only exception that of a low velocity of 7.178 km/s in the 15-mil track.

As discussed last quarter(1) the detonation velocity figures given do not necessarily need to meet a given value, but they must be consistent and reliable. The extrudability figures given should approximate those of a typical production lot such as the one shown in Table II (the last lot listed), but some allowance can be made if the detonation velocity figures are consistent and the DV test block fires reliably on all tracks.

The detonation velocity values for the last five batches are consistent with, but in most cases higher than, the production lot listed. The one low figure (for Batch 6127-01 in the 15-mil track) is less than 1% lower than the production lot.

CONCLUSIONS AND COMMENTS

The results from the last several runs have been very encouraging—a steady improvement in firing performance of the LX-13 has been made with little effect on the extrudability of the material. The last and final stage of this project will be to further refine the processing parameters for producing large quantities of PETN in a single run and establish the reliability and reproducibility of the process. Several large batches (18 kg) from different lots of mil spec PETN will be made to attempt to duplicate the performance of Batches 6040-01 and 6041-01, which not only had very high extrudability for thermally purified PETN, but also fired extremely well. Other objectives will be to further refine the operating conditions to obtain consistent surface areas and to train production personnel in operating the equipment.

A full particle characterization (e.g., Zeiss Analysis, etc) will be made of those PETN batches for which previous photomicrographs are available and for any future batches of PETN to determine an optimum particle size distribution. An attempt will be made to correlate processing parameters to the particle size distribution using the data generated above.

Further work needs to be done in upgrading the continuous process, in training production personnel to operate the equipment, and in designing a facility for use of the equipment. The transition and training of personnel should present no unusual problems, since the materials (PETN, reagent grade acetone, and distilled water) are very similar to WR component materials already being used in production batch processing.

With the scale up to production size lots completed this project will be transferred from Process Development to Process Engineering to accomplish the refinements listed above.

Table I. Continuous Process PETN Formulation Parameters

PETN Lot Number	Lot Size (kg)	Acetone Flow Rate (cm ³ /s)	Distilled Water Flow Rate (cm ³ /s)	Water Temperature (C)	Impeller Diameter (mm)	Impeller Speed (RPM)	PETN Surface Areas (m ² /kg)	
							Air Permeametry FSSS(S ₀ [P])	Gas Adsorption Perkin-Elmer [S ₀ (G)]
6040-01	4.0	13.42	46.67	11	32	2000	440	865
6041-01	4.0	8.08	26.47	4	32	1000	390	845
6048-01	18.0	8.08	26.47	4	32	1000	390	690
6125-01	18.0	8.08	26.47	4	32	1000	405	1300
6127-01	18.0	8.08	26.47	4	32	1000	325	725

Table II. Continuous Process PETN Evaluation

Lot Number		Lot Size (kg)	PETN Surface Areas (m ² /kg)		Wt. % Volatiles	Length Extruded Down the Track (mm)			Firing Performance Detonation Velocity (km/s)			
			Air Permeametry FSSS(S ₀ [P])	Gas Adsorption Perkin-Elmer [S ₀ (G)]		0.015"	0.020"	0.035"	0.015"	0.020"	0.035"	0.080"
PETN	LX-13											
6040-01	20-76-0428-222	4.0	440	865	0.02	25.9	35.2	82.0	7.301	7.302	7.364	7.336
6041-01	20-76-0630-223	4.0	390	845	0.01	28.2	42.6	105.8	7.301	7.318	7.410	7.387
6048-01	20-76-0504-224	18.0	390	690	0.01	20.3	25.9	77.2	7.301	7.308	7.425	7.360
6125-01	20-76-0713-226	18.0	405	1300	-	19.1	28.9	78.1	7.261	7.290	7.374	7.362
6127-01	20-76-0907-229	18.0	325	725	-	20.1	28.7	84.3	7.178	7.314	7.416	7.340
10-73-1120-116 ^a	20-73-0294-103	18.0	605	905	0.01	34.3	41.7	77.7	7.230	7.250	7.265	7.265

^aProduction lot