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LX-13 Processing

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DEVELOPMENT DIVISION

JANUARY - MARCH 1976

Normal Process Development
Endeavor No. 102

MASTER

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SUMMARY

Results were obtained from two LX-13 lots formulated from PETN precipitated at a reduced temperature by the continuous method. The firing performance of the LX-13 was significantly improved while maintaining acceptable extrudability. Burning was complete in all of the tracks.

Three additional PETN batches have been precipitated at reduced temperatures in order to further evaluate the effect of reduced temperatures on extrudability and detonability. One of these PETN batches was scaled up to the production PETN batch size of 18 kg.

INTRODUCTION

Work has been in progress to develop a process for continuously precipitating PETN which is used for making LX-13. The process has been developed to the point that precipitation conditions can be varied to obtain either the desired extrudability or detonability of LX-13. Difficulties have arisen, however, in achieving the proper conditions for obtaining both the desired extrudability and detonability simultaneously. These properties seem to have opposing effects. Changes made to improve one property are detrimental to the other and vice versa. Processing parameters which have been studied include acetone/PETN and water flow rates, impeller type and speed, and water temperature. Water temperature has been controlled at 21 C; however, last quarter batches of PETN were precipitated at a reduced temperature (8 C) . Results were encouraging—the trend was toward improved detonability while maintaining adequate extrudability. Additional improvement was required, however, because burning was not complete in all of the tracks.

DISCUSSION

DETONATION VELOCITY TESTING

Three PETN batches precipitated previously by the continuous method(1) were dried in a recirculating steam oven at 66 C for a period of 36 hours. Gas adsorption [Perkin-Elmer (S_0[G])] and air permeametry [Fisher Sub-Sieve Sizer (FSSS[S_0(P)])] surface areas were obtained and the PETN batches were formulated by the production process into LX-13 lots. Two of the three PETN batches (5304-01 and 5304-03) were successfully formulated. The third (5304-02) did not transfer during the roll milling process. It was subsequently analyzed for moisture content and found to contain an excessive amount. Therefore, it was destroyed without testing. Extrudability and detonability were measured for LX-13 made from the two successful batches of PETN. The results for the

(1) J. C. Adams, A. G. Osborn and T. L. Stallings, LX-13 Processing, October - December 1975, MHSMP-76-5B.

two batches of PETN are presented in Table I (the first two batches listed). Also presented for comparison purposes are two LX-13 batches reported previously and a typical LX-13 lot (20-73-1120-116) prepared from a batch-precipitated production lot of PETN. Formulation parameters for the PETN batches 5304-01, 5304-02, and 5304-03 are repeated in Table II for ease of reference.

The extrudability data in Table I indicate that the larger impeller (64 mm vs 32 mm) and the low flow rates (8.08 cm^3/sec vs 13.42 cm^3/sec for acetone, 26.35 cm^3/sec vs 46.67 cm^3/sec for water) affected the extrudability slightly at the reduced temperature. Batch No. 5304-03 made with the 64 mm impeller at the low flow was not as extrudable as Batch No. 5304-01 made at the high flow rates with the 32 mm impeller. This is consistent with experience at the higher temperature (21 C) as presented in previous quarterly reports(*1,2*). The values of extrudability in the 15-mil track of 24.4 mm (5304-01) and 19.3 mm (5304-03) are low but would be considered acceptable if the LX-13 fires successfully and detonation velocities are recorded on all tracks.

Firing was successful for both the LX-13 lots; all four tracks fired for each lot with detonation velocities ranging from 7.238 km/sec to 7.354 km/sec for PETN Lot No. 5304-01 and 7.197 km/sec to 7.348 km/sec for No. 5304-03. Typical velocities for a Production LX-13 lot fall in a range of 7.230 km/sec to 7.265 km/sec as shown in Table I. Detonation velocities are not required to be within a certain range. What is important is that the firing for any given lot be reliable and consistent.

PETN FORMULATION

Three additional PETN batches were precipitated by the continuous method at reduced temperatures in a continuation of the partial factorial experiment outlined in the July - September report(*2*). The first batch, No. 6040-01, was a duplicate of Batch 5304-02, which could not be milled due to high moisture content. The second, No. 6041-01, was an experimental batch conducted to test the new heat exchanger design and evaluate an even lower processing temperature (4C vs 8 C) at the lower flow rates and rpm. The third batch, No. 6048-01, was a scale-up to the production PETN batch size of 18 kg. The low flow rates, small (32 mm) impeller, low agitation (1000 rpm) and low temperature (4 C) were used. All batches were dried in a recirculating steam oven at 66 C for 36 hours. Samples were taken and air permeametry [FSSS(S_o[P])] and gas adsorption [Perkin-Elmer(S_o[G])] surface areas and moisture analyses were measured; photomicrographs at a magnification of 160X were obtained. The parameters used for the three batches, the moisture content, and resulting surface areas are summarized in Table II (the last three batches listed). The photomicrographs are included in Fig. 1. LX-13 lots are currently being formulated from these PETN batches.

(2) J. C. Adams, A. G. Osborn and T. L. Stallings, LX-13 Processing, July - September 1975, MHSMP-75-40A.

CONCLUSIONS, COMMENTS AND FUTURE WORK

The extrudability and detonability of the LX-13 manufactured from continuously-precipitated PETN can be improved substantially by precipitation at reduced temperatures. The detonability was greatly improved over previous batches. The slightly higher detonation velocities recorded may be due to the thermally-purified PETN which is used as the starting material for the continuous precipitation process. A normal lot of LX-13 formulated from a thermally purified PETN batch precipitated by the batch process detonated in the 7.3 km/sec range. As noted in the discussion the velocities recorded are not required to meet a standard but rather to give consistent and reliable detonation velocities.

The manufacture of large batches of PETN by the continuous method appears to present no unusual problems. Some time savings can be realized over present manufacturing methods, if large lots can be manufactured at one time. Only one operator is necessary to monitor the operation and tend the material for the continuous method, while two operators are required for precipitating the PETN in 5-1/2 to 6 kg batches and blending into a single 18 kg lot.

The PETN manufactured by the continuous process at reduced temperature appears to perform nearly as well as the PETN precipitated in 5-1/2 to 6 kg batches and then blended into a single 18 kg lot when formulated into LX-13. Further experiments need to be performed to evaluate the effect of scale-up on the subsequent LX-13 performance and to confirm behavior of the LX-13 formulated from PETN precipitated at reduced temperatures. Parameters to be evaluated will include batch size, drying method and feed method.

Table I. PETN/LX-13 Extrudability/Detonability

Batch Number		PETN Surface Area (m^2/kg)		PETN Batch Size	%	Length Extruded Down the Track (mm)			Detonation Velocity (km/sec)			
PETN	LX-13	FSSS [S_o(P)]	Perkin-Elmer [S_o(G)]	(kg)	Volatiles	0.015"	0.020"	0.035"	0.015"	0.020"	0.035"	0.080"
5304-01	20-75-1230-213	355	825	4.0	-	24.4	35.6	114.3	7.238	7.286	7.354	7.353
5304-03	20-76-0108-215	375	855	4.0	0.03	19.3	27.5	87.1	7.197	7.271	7.335	7.348
5259-02	20-75-1025-207	410	840	4.0	0.02	24.6	31.5	105.6	PB [a]	7.225	7.347	7.346
5259-01	20-75-1024-208	500	1170	4.0	0.01	18.7	40.4	81.7	7.206[b]	7.252	7.356	7.332
c	20-73-1120-116	605	870	18.0	0.01	34.3	41.7	77.7	7.230	7.250	7.265	7.265

[a] *Partial Burn - Burns stopped at 5th port on first DV block and 2nd port on second DV block*

[b] *Burn stopped at 7th port on first block and 2nd block fired*

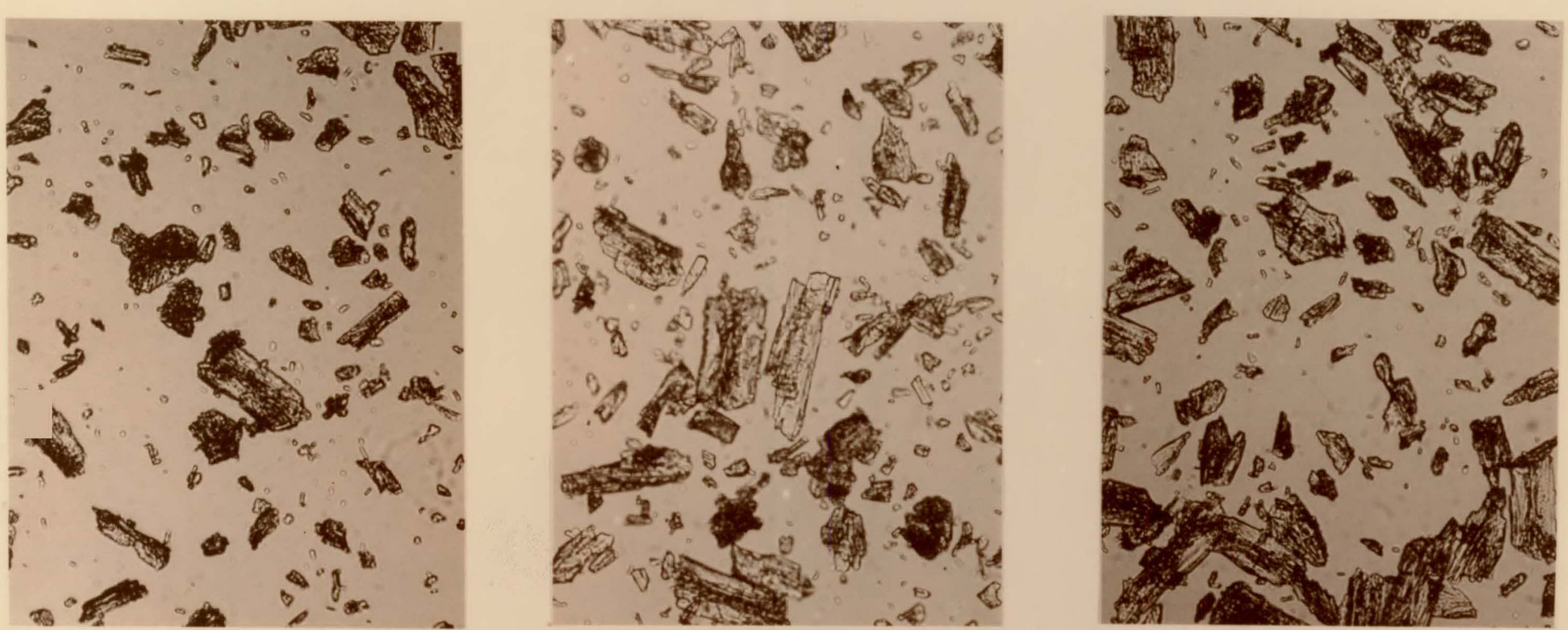
[c] *PETN Batch No. 10-73-0294-103 - A batch-precipitated PETN lot*

Table II. PETN Continuous Precipitation Parameters

PETN Batch No.	Batch Size (kg)	Acetone Flow Rate (cm^3/sec)	Water Flow Rate (cm^3/sec)	Water Temperature (C)	Impeller Diameter (mm)	Agitator Speed (Rev./Min)	Moisture Content (Wt % Volatiles)	PETN Surface Area (m^2/kg) FSSS [S_o(P)]	Perkin-Elmer [S_o(G)]
5304-01	4.0	13.42	46.67	11	32	1000	*a*	355	825
5304-02[b]	4.0	13.42	46.67	11	32	2000	6.6	375	835
5304-03	4.0	8.08	26.35	8	64	1000	0.03	375	855
6040-01	4.0	13.42	46.67	11	32	2000	0.02	420	865
6041-01	4.0	8.08	26.35	4	32	1000	0.01	390	845
6048-01	18.2	8.08	26.35	4	32	1000	0.01	390	690

[a] *Not obtained - Dried at 66 C for 36 hours*

[b] *Destroyed - Too wet to mill*



PETN Batch 6040-304C-01
$S_o(P) = 420\ m^2/kg$
$S_o(G) = 865\ m^2/kg$
4 kg Batch

PETN Batch 6041-304C-01
$S_o(P) = 390\ m^2/kg$
$S_o(G) = 845\ m^2/kg$
4 kg Batch

PETN Batch 6048-304C-01
$S_o(P) = 390\ m^2/kg$
$S_o(G) = 690\ m^2/kg$
18 kg Batch

Fig. 1. Photomicrographs of Samples of Formulated PETN (160X Magnification)