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TATB DRY AMINATION FINE PARTICLE PROCESS

W. T. Quinlin
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DEVELOPMENT DIVISION

JULY - SEPTEMBER 1977

Process Development
Endeavor No. 106

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ABSTRACT

A process was developed on the pilot plant scale which can be used to reproduce fine particle TATB by dry amination. A series of 10 batches, with each batch yielding from 15 to 20 kg was produced to evaluate the process. The total chlorine content of the batches was generally about 0.5% but one of the batches had a total chlorine content higher than expected (1.05%).

DISCUSSION

The need for a fine particle size process for TATB is related to the requirement of booster pellets. Several approaches for an amination process were considered, including introduction of ammonia by sparging, low temperature reaction condition and agitating the reaction at a reduced rate from that normally used to produce coarse particle TATB. The latter was selected because of equipment considerations and past experience(1) with this type of process.

The amination process for the production of TATB involves dissolving sym-trichlorotrinitrobenzene (TCTNB)

in toluene and reacting it with gaseous ammonia. The reaction takes place at the liquid interface, and at 150 C TATB is only slightly soluble in toluene. With mild agitation the fine TATB particles fall below the reaction zone and their size is preserved. However, when the agitation is reduced throughput is significantly reduced. Initial work established a process that met two criteria: a reaction time of less than 8 hours for the production of 35 to 40 pounds of TATB in a 100-gallon vessel and a product containing a minimum of 75% of the particles passing a 20 μ m sieve.

The final series of 10 batches showed that the process was capable of reproducing batches which had 82% $\pm$ 5% of their particles passing a 20 μ m sieve.

The total chlorine content of the batches ranged from 0.37 to 0.60% with

(1) W. T. Quinlin, V. H. Evans & Z. L. Estes, TATB Pilot Plant Activities FY 75, MHSMP-74-35L.

an average of 0.47% omitting the high chlorine containing batch (1.05%) from calculation. A sigma of 0.07% was found for the nine batches.

EXPERIMENTAL

All the amination reactions were conducted in a 100-gallon stainless steel reactor heated with 75 psi steam to the jacket. The reactor was fitted with an ammonia inlet, a vent tube for azeotroping water from the system and a thermowell.

The TCTNB was first dissolved in toluene in a feed vessel and passed through a 1.5 μ m in-line filter during transfer to the reactor. The solution was then heated to 140 C and the water azeotroped from the system. The ullage was then backfilled with ammonia and the temperature brought to 150 C. The reaction time varied between 6 and 8 hours with completion indicated by a drop in ammonia flow as measured by the mass flow meter, an increase in system pressure approaching the 60 psig of the ammonia regulator and a decrease in the heat generated as indicated by

the requirement of more steam to the reactor jacket.

Product isolation involved cooling the reaction mixture to 100 C, adding 40 l of water and vigorously stirring for 0.5 hours. The warm mixture was isolated in a plate and frame filter press, washed with 85 C water for 1 hour and steamed for 1 hour. The product was then dried at 115 C for a minimum of 16 hours prior to sampling and packaging.

Table I gives the yield, conditions and analysis for the 10 batches. The particle size data were determined by wet sieving through a stack of electro-form sieves. The total chlorine was determined by decomposition in a Parr bomb calorimeter and determining the chloride liberated by titration with silver nitrate.

CONCLUSIONS

A process has been developed for the reproducible production of fine particle TATB which should be applicable for booster pellets.

Table I. Amination Results

Batch No.	Yield TATB (kg)	Time (hrs)	Total Chlorine	Particle Size	
				<20 μ m (%)	<44 μ m (%)
7076	18.2	6.25	0.44	87.5	99.0
7080	19.1	6.25	1.05*	77.9	96.5
7084	18.2	5.75	0.47	88.0	98.1
7087	15.4	6.00	0.40	77.1	98.2
7088	16.8	6.50	0.52	79.2	97.4
7094	17.2	5.75	0.60	85.2	99.7
7242	16.8	6.75	0.45	88.8	98.3
7265	17.0	8.00	0.50	80.0	97.0
7270	17.7	6.50	0.37	85.7	97.7
7271	17.7	6.00	0.44	75.2	89.7
Mean			0.5	82.0	97.0
Standard Deviation			0.07	5.1	3.2

*Omitted from calculation of average.

Note: All batches were run using 26.3 kg TCTNB in 312-litre toluene at 154 C and 55 rpm. Concentration was at 0.084 kg/l.

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