Twin Screw Extrusion of Black Powder with Sodium CarboxyMethyl Cellulose (CMC) Binder

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Background

In 2003, US Army RDECOM-ARDEC Twin Screw Extruder (TSE) team (Picatinny Arsenal, NJ) was enlisted by Program Executive Officer for Ammunition (PEO Ammo) and the Pyrotechnics Group (Picatinny Arsenal, NJ) to produce a black powder substitute (BPS). A Single Point Failure (SPF) situation exists within the US manufacturing of military grade black powder (BP). With use in over 100 Army end items, a shutdown in the production of black powder would have tremendous negative impact on, not only the Army, but the DoD community as a whole. In an effort to mediate the risk associated with sole-source production of BP a foreign comparison test (FCT) was initiated. Potential BP manufacturing sources outside the United States were solicited for this study. The Pyrotechnics Group, Picatinny Arsenal, NJ, was the lead on this project. A twin screw extruder processed black powder substitute was one alternative tested. In the end, TSE BPS fell short in performance as compared to GOEX but the initial goal of the TSE facility was met – to show processability of a BP-type material with water soluble binder through a TSE. The continuation of this work is to provide a BPS with comparable performance characteristics as traditionally made BP.

Introduction

The current black powder formulation has existed for over 2000 years. The basic formulation has not changed since its first discovery in China. Current manufacturing methods can be traced to E. I. du Pont's process in the 1800's. BP manufacturing consists of seven basic steps. First, charcoal and sulfur are combined and pulverized in a ball mill. This 'pulverized rifle' is then combined with water and oxidizer, KNO3, in a wheel mill. Next, the material is pressed through hydraulic presses to further consolidate the constituents. This dense cake then gets broken up and granulated via corning mill operations. Finally the material is sieved into appropriate classes, glazed with graphite, and dried. These operations are highly labor intensive and provide numerous opportunities for incident.

The TSE processing method will involve the elimination of the first four processing steps. The TSE process is a fully remote operation in which 2 -3 pounds of material are processed continuously at a throughput of up to 35 lb/hr. This will eliminate the high operator interaction with the live material and, therefore, increase overall safety of the operation. Today, TSE BPS is 85-90% of the performance of GOEX black powder.

Objective

The objective of the program was initially to show that a TSE could produce a black powder type material with a water soluble binder system. Upon completion of this objective a new goal was formed – to produce a black powder type material with similar characteristic to that of conventional black powder.

Experimental

To achieve the stated objectives a process development map detailing the approach was devised. This science-based development plan includes simulant identification, rheological characterization, mathematical modeling, model validation, inert and live processing, and product analysis.

Simulant Identification

In order for initial processing of a Black Powder substitute (BPS) through a TSE, an inert formulation must first be determined. This inert formulation provides a means to safely verify the process without the chance of incident. Only upon completion and verification of the initial runs and process condition will the live material be processed.

In order to determine an inert formulation numerous parameters were characterized. These included both mechanical properties (rheology, structure, and surface wettability) and physical properties (solubility, density, and melting point). In the case of BPS, the oxidizer (KNO_3) is replaced with a non-oxygen carrying molecule. Table 1 shows the possible candidate replacements for KNO_3.

Compound	Solubility g/100ml H_2O 0 ^oC - 100 ^oC	Density g/cc	mp ^oC	Structure
KNO3	13.3 - 247.0	2.109	129	rhomb.
NaCl	**35.7 – 39.1**	**2.165**	**801**	**cub.**
NaHCO3	6.9 -16.4$^{(60)}$	2.16-2.22	-CO2$^{(270)}$	mono.
NH4Cl	29.7 – 75.8	1.527	355 sub	cub.
Li2SO4	26.1 – 23.0	2.221	860	mono.

Table 1 - List of simulant candidates for the replacement of KNO_3 in the inert formulation

Upon consideration of these simulants, sodium chloride (NaCl) was selected as the inert substitute for KNO_3. It was chosen due to its availability in the desired particle size and other material properties.

Recent evidence has shown pitting of the processing equipment. This suggests that sodium bicarbonate [$CHNaO_3$] may be a better inert simulant than NaCl. Future work will be done with this as the inert simulant for KNO_3.

Raw Ingredient Studies

To better understand the effects of raw ingredients on performance, studies on particle size and intimacy of contact were performed. Table 2 shows the particle sizes of various pre- and post-processed materials.

	Particle Size Distribution	
Material	GOEX	TSE
Preblend (pulverized rifle)	1 – 30 μm	Non ball milled: 1 – 200 μm Ball milled: 1 – 100 μm
KNO_3	--	--
Post processing BP	< 10 μm	100 μm

Table 2. Particle size distribution of pre-process and post- process BP and BPS

Rheological Characterization

The deformation characteristics of the material as it is processed is one of the most important processing considerations. Through the use of rheology one may understand these types of deformations and how the material will process through an extruder. The rheological data gathered by Stevens Institute of Technology (SIT) on the BPS is used to model conditions within the extruder. This model provides information on temperature, pressure and shear forces imparted on the material as it is being process in the extruder. This information is vital to assigning processing parameters and condition so as to process safely.

When initial rheology was done on the black powder formulation it was evident that the military specified formulation was not processable in the TSE. It was determined that a water soluble polymeric binder is needed to be added in order to increase 'flowability' and eliminate phase segregation. Sodium carboxymethyl cellulose (CMC) was selected as a processing aid for its ability to form a hydrogel 'carrier'. Based on inert and live material rheology it was determined that four weight percent CMC be added to the formulation to provide adequate processability.

Mathematical Modeling

RDECOM-ARDEC along with Stevens Institute of Technology provided processing parameters and profiles based on mathematical modeling. Rheological data gathered at SIT was input into the model. The screw configuration and die design are two processing parameters generated via modeling. Figure 1 shows the screw configuration used during the original phase of work. Along with that, a .2" diameter single strand die with conical converging angle of 14.4^{o} and straight land section with L/D ration of 2.5 was selected.

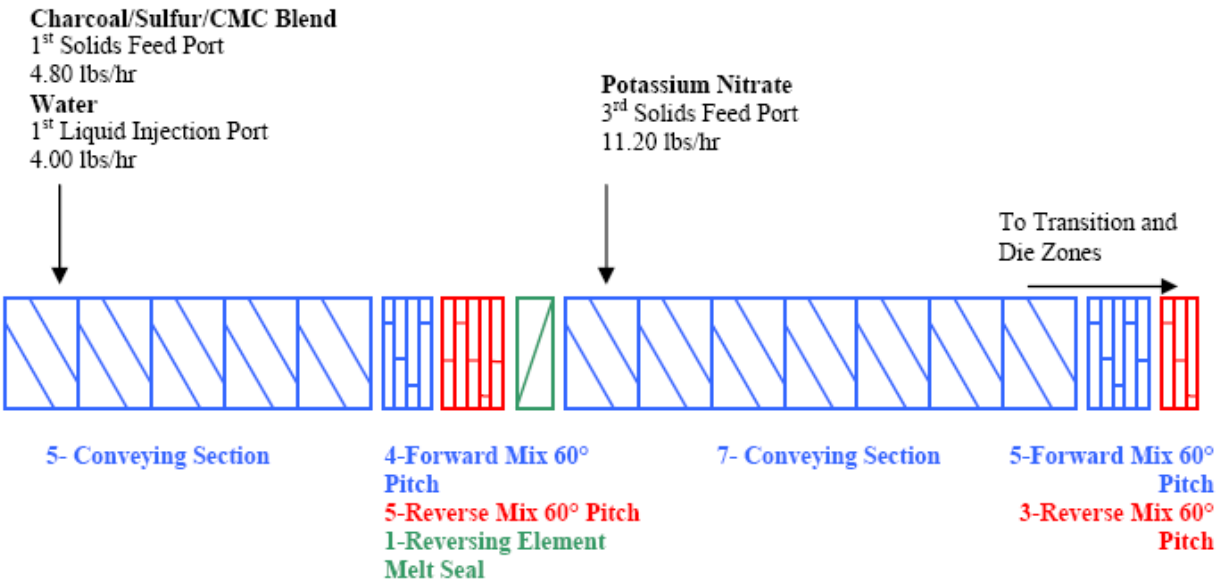

Figure 1. Screw configuration used for initial effort

Current Work

Current work to provide a BPS with similar performance characteristics to conventional BP has yielded numerous processing changes. The first of these changes is adjustment of the screw configuration. Feed order and feed rates have also been modified. Figure 2 shows these processing modifications.

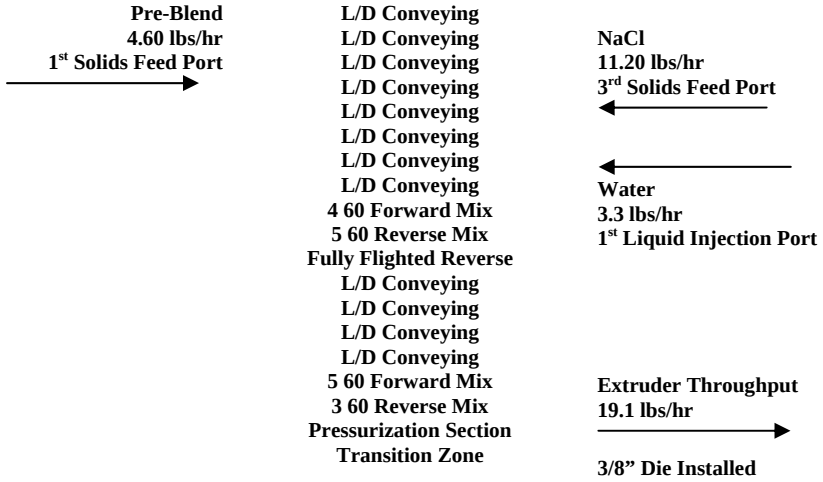

Figure 2. Screw configuration, feed order and feed rates with modifications

Other changes included the acquisition of GOEX pulverized rifle to replace non-ball milled preblend. Drying processes are currently being explored. Also, TSE BPS is being shipped to GOEX to undergo traditional post processing techniques. This will further quantify post-processing effects on end performance.

Along with that, performance standards for end items have never been established. This on going work will also focus on providing benchmarks for end item performance. The Pyrotechnics group plans to load TSE BPS into various end items in order to study performance output in relation to product functionality.

Testing

Various tests have been done on TSE BPS. These include closed bomb, bomb calorimetry, thermal analysis, moisture analysis, and scanning electron microscopy. Figures 3 and 4 show both closed bomb data and bomb calorimetry data comparing GOEX BP to TSE PBS and Wano (a German company studied in the FCT).

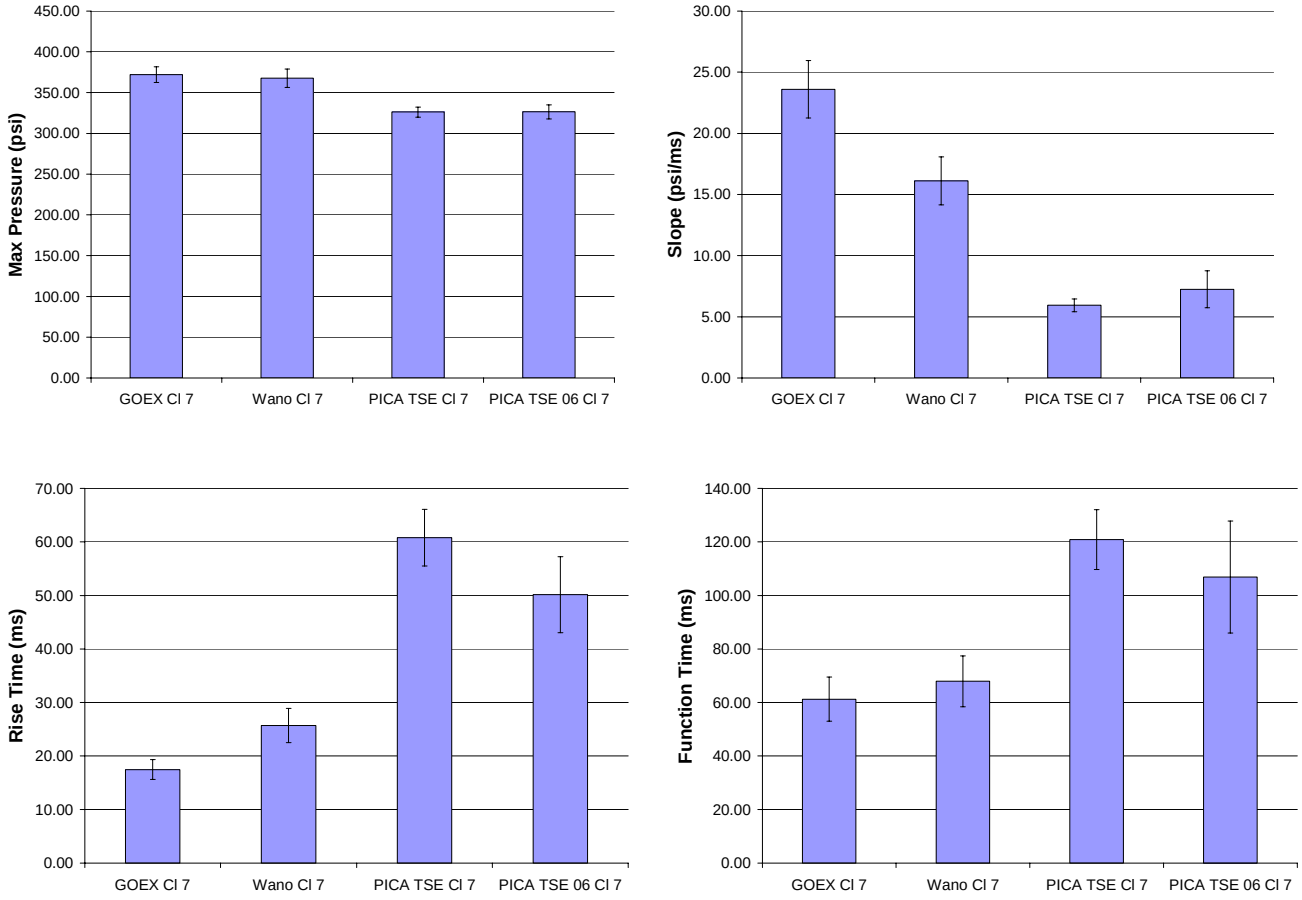

Figure 3. Closed Bomb data comparing GOEX BP to TSE BPS (PICA TSE Cl 7 = previous effort, PICA TSE 06 Cl7 = current efforts) and Wano

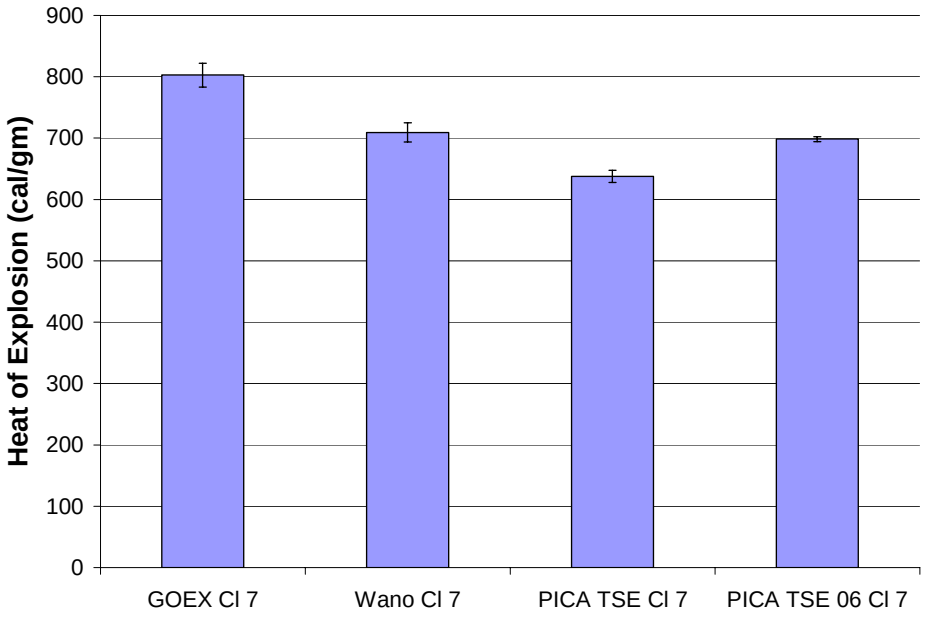

Figure 4. Bomb Calorimetry data comparing GOEX BP to TSE BPS (PICA TSE Cl 7 = previous effort, PICA TSE 06 Cl7 = current efforts) and Wano

Results indicate an increase in performance over previous work. However, TSE BPS still comes up short compared to GOEX. As it stands now, TSE BPS is 85-90% that of conventional BP.