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# **Application of Twin Screw Extrusion for Continuous Processing of Energetic Materials**

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**Abstract:** Continuous processing of energetic materials using a twin screw extruder is gaining importance as it is a safe and cost-effective alternative to conventional batch processing. The continuous process based on a twin screw extruder combines the capabilities of intensive mixing and high pressure extrusion. It is used for processing a variety of energetic materials, such as gun and rocket propellants, plastic bonded explosives, pyrotechnics, thermo-baric explosives, etc. The twin screw extruder process demands various safety features for the processing of energetic materials. Therefore, exhaustive characterisation of the energetic materials in terms of safety and rheology, coupled with characterisation of the mechanical components of the extruder, are essential for designing a safe continuous process. In this article, a technological overview of continuous processing for energetic materials is presented, along with its various features, process design methodology and safety issues.

**Keywords:** twin screw extruder, energetic materials, rheology, safety, residence time distribution

#### 1 Introduction

Energetic material formulations such as propellants, pyrotechnics and explosives are highly concentrated suspensions. The concentration of energetic particles is often close to the maximum packing fraction to maximise performance. The

manufacturing of such energetic materials involves various processes such as mixing, casting, extrusion, polymerisation, *etc*. The mixing of the ingredients is one of the most important and potentially hazardous operations in the manufacture of energetic materials. Conventionally, most of the processing methods for energetic materials are based on batch mixers rendering the entire process to a batch process. However, there is increasing interest in continuous processes for energetic materials because of its several advantages, the most important being increased safety due to the significantly smaller quantities of explosive material handled in the processor at any given time. The overall cost reduction, flexibility, complete automation, increased productivity, improved mixing efficiency with reduced waste, are some of the other advantages [1-5].

The early efforts with continuous processing include the processing of composite propellants based on Ko-kneader and Rotofeed deaerators for the Polaris programme in the 1960s [6], and processing of single and double base propellants by several European manufacturers in the 1970s [7]. In period 1994-1999, a joint programme 'Continuous Processing of Composite Propellants' (COPCO), between USA and France, demonstrated the capability of continuous processing of composite propellants using a twin screw extruder [8]. Societe National des Poudres et Explosifs (SNPE), has also been operating continuous production lines for extruded composite propellants for air bags since 1996 [9, 10]. Furthermore, SNPE has proposed the construction of a continuous mixing and casting facility for solid rocket motors for Ariane-5, with an estimated 20-30% reduction in production costs [11]. Thus, with these technological advances, there is an increasing confidence and interest in the continuous processing of energetic materials. Presently, it is being used for a variety of energetic materials, such as composite propellants, homogenous propellants (single, double and triple base), [4] high energy propellants (nitrate ester plasticised polyether, NEPE propellant) [12], energetic thermoplastic elastomers (ETPEs) [13], PBXs, decoy flares [14], thermobaric explosives [15], air bag propellants, etc.

In the following sections, the various components of a continuous processor, process development methodology, safety issues, residence time and shear distribution in the extruder and rheological issues affecting continuous processing, are presented in detail. In addition, a few specialised applications of continuous processing are discussed.

### Advantages of continuous processing:

- i. Increased safety due to significantly smaller quantities (~1 order of magnitude less) of energetic materials in the hazardous mixing zone at any given time.
- ii. Highly flexible systems with the capability of processing a variety of energetic materials, such as rocket propellants, gun propellants, PBXs, graded energetic materials, *etc*.
- iii. Reduction in overall cost due to the compact systems with fewer buildings, installations, and less wastage of material.
- iv. Improved quality control because of online monitoring and complete automation.
- v. Improved heat transfer due to higher surface to volume ratios compared to batch mixers, making it highly suitable for thermoplastic materials.
- vi. Intensive mixing because of higher surface to volume ratios, making it suitable for difficult to mix materials like nano-energetic materials.
- vii. For gun propellants, it replaces the mixing and extrusion steps in batch processes with a single step mixing and extrusion.
- viii. Reduced volatile organic compound (VOCs) emissions in the processing of gun propellants, rendering the process as a green process [2].
- ix. Continuous processing is highly suited for special applications, such as functionally graded material, co-layered materials, *etc*.
- x. Continuous processing increases the production capacity significantly vis-à-vis that of batch processing.
- xi. The pot life issue is eliminated in continuous processing because of the very short residence time. This allows the processing of materials with very fast curing reactions [9].

#### 2 Construction of a Continuous Processor

Continuous processors for energetic materials are based on screw extruders, usually twin screw compounders/extruders [1, 3]. The major components of a typical continuous processor are:

- a. twin screw continuous mixer;
- b. ingredient feeding system;
- c. on-line quality control;
- d. shaping device.

The general scheme for continuous processing of energetic materials is shown in Figure 1.

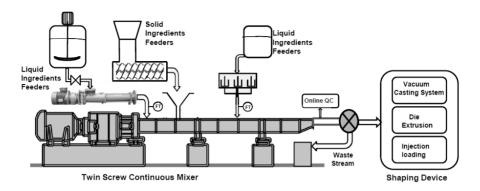


Figure 1. General arrangement of a continuous processor for energetic materials.

#### 2.1 Twin screw continuous mixer

Continuous mixers are based on a screw extruder, usually a twin screw extruder (TSE). The homogenous mixing and reduced friction in a TSE makes it more suitable for processing energetic materials. Co-rotating, fully intermeshing types of TSEs are inherently safe and effective for the processing of energetic materials because of the evenly distributed pressure across the periphery, uniform residence time and reduced sensitivity to the head pressure [1, 16]. TSEs have been used regularly in the plastics and polymer industries for a very long time; however, their application in the explosives industry needs due consideration for safety.

A typical design of a TSE consists of two screws rotating inside a closed housing called the barrel. The gap between the screw and the barrel wall is of the order of a few microns (of the order of 0.001\*D, where D is the diameter of the screw) to ensure a wiping action. However, for processing energetic materials with particulate matter, the gap is maintained at 2-3 times the maximum particle size of the powder for safety reasons [17]. These machines have different sections of the screw for different functions, such as feeding, melting, mixing, metering, venting and extrusion. The design of the screw is usually modular or segmented, as it offers flexibility and avoids the need to hold tight bore tolerances over a long barrel length. The screw elements are stacked one beside the other on a splined shaft. The screw design is expressed by the size (diameter) of the screw and the length to diameter (L/D) ratio of the screw, which varies from 7 to 30 depending on the application. Screw designs with short L/D ratios perform primarily mixing and kneading actions and do not generate extrusion pressure. Such mixers are also known as twin screw compounders. The majority of screw elements in these mixers are mixing and kneading elements. The barrels for TSEs have a clamshell or split design, with the two halves held together hydraulically or hinged for easy cleaning, with dowel pin assembly for alignment [2, 3, 18]. The barrel has various zones for temperature control and ports for feeding ingredients, venting, etc. The barrels are designed for varying pressure, up to 350 bar, for applications involving mixing followed by extrusion.

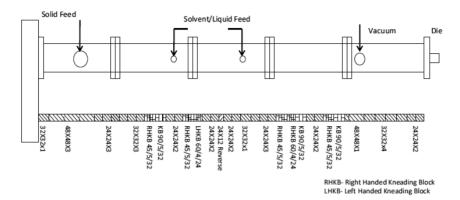
The mixing action in highly filled energetic materials does not encounter turbulence or eddy diffusion due to the very high viscosity. Also, molecular diffusion, being a very slow process, is insignificant. Hence, the major mixing action is distributive mixing (macro-mixing) and dispersive mixing (micromixing). Homogenous mixing of energetic materials requires both distributive and dispersive mixing [19]. The screw has several elements which impart distributive and dispersive mixing actions. Conveying elements are responsible for distributive mixing and kneading blocks imparts dispersive mixing. The standard kneading elements and conveying elements are shown in Figures 2 and 3. The kneading blocks are the main mixing elements, which consist of several discs staggered at an angle to one another. In some designs, the mixing elements are kneading paddles with various combinations of flat and helical paddles. The motion of these kneading blocks imparts intense shearing action and facilitates dispersive mixing. The screw elements in the vented section offer provision for vacuum application for deaeration or removal of volatiles. The arrangement of the various elements is optimised for product quality, safety and production rate. A schematic representation of a typical screw configuration is shown in Figure 4.



**Figure 2.** Kneading elements of a twin screw extruder.



Figure 3. Conveying elements of a twin screw extruder.



**Figure 4.** Schematic representation of the profile of a Twin Screw Extruder for the processing of gun propellant, used at High Energy Materials Research Laboratory, Pune, India.

## 2.2 Ingredient feeding system

The ingredients feeding system is an important component of a continuous processor. The feed streams of the liquid and solid ingredients are fed, with accurate metering, into the processor at various locations according to the mixing sequence. Accuracy and safety are the important criteria for the design of a feeding system. As the ingredients are energetic in nature, safety features such as non-sparking feed devices, explosion proof drives, explosion proof sensors, *etc.* must be incorporated.

## 2.2.1 Feeding of liquid ingredients

The various liquid ingredients used in energetic materials are polymeric binders, plasticisers, solvents and curing agents. In a few cases, such as composite propellants and PBXs, the polymeric binder and metallic powder mixed in a high speed disperser, constitute one liquid feed stream called the fuel premix [20, 21]. The minor ingredients of the formulations are usually blended with the major ingredients and fed to the extruder. The displacement for liquid ingredients is based on a volumetric feeding system, comprising a positive displacement pump. The various types of pumps used for high viscosity fluids, such as the fuel premix, are progressive cavity pumps, lobe pumps and gear pumps. The low viscosity fluids, such as plasticisers, curing agents and solvents, are pumped using internal gear pumps, diaphragm or piston pumps. A closed loop can be formed by measuring the mass flow of the liquid stream using a flow meter for better flow control.

## 2.2.2 Feeding of solid ingredients

Feeding of solid powder ingredients is based on gravimetric feeding [20] or volumetric feeding [14]. The flow characteristics and bulk density of the materials are important criteria for the selection of feeders. Volumetric feeding is a simple and economical method for materials with consistent bulk density, such as pellets, granules, *etc.* Gravimetric feeding, based on loss-in-weight (LIW) feeders, is an accurate method for materials with varying bulk density. The principle of operation of these feeders is based on varying weight where the feed rate is directly related to the change in weight. These feeders are also designed with an automatic refill capability, where, automated cranes are used to replace the bin on the feeder. During refilling of the feeder, the control mode of the feeder is switched from gravimetric to volumetric mode. Hence, these feeders are designed with minimal refill time and once refilling is over, gravimetric control resumes. In addition, a metal detection system is also used to check for the entry of ferrous materials into the extruder [22].

## 2.3 On-line quality control

On-line analysis of the quality of the product is an essential feature of a continuous process. On-line analysis tools, based on measurement of concentration of ingredients in the extrudate, are necessary to assess the quality of mixing and real time quality control with feedback control. It allows diversion of rejected materials into a waste stream. Furthermore, on-line measurements enable a detailed understanding of the effects of screw geometry and processing parameters on the product quality. Several techniques are used for online quality control. Near infra-red (NIR) spectroscopy [23], on-line X-ray based techniques [24] and on-line densitometry [25] are some of the techniques employed in continuous processing. The constituents of energetic materials, such as oxidisers, fuels, plasticisers, binders, etc., have near infrared spectral signatures that allow their quantification by NIR spectroscopic methods. In addition, NIR also allows the prediction of the physical and ballistic properties [23] of the finished product based on changes in the spectral response. On-line densitometers in composite propellant processing are based on a radiometric, non-contact type device consisting of a gamma ray source (138Ce), a scintillation detector and microprocessor. A gamma ray source is mounted on the external surface of the pipe or die through which the energetic material is passed, with the detector mounted on the opposite side. The detector measures the energy and, accordingly, the signal processed by the microprocessor is in terms of the density of the material.

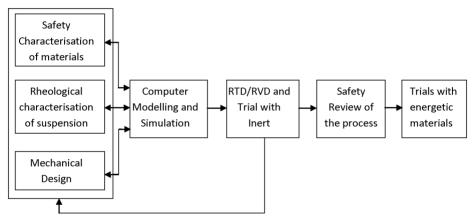
#### 2.4 Shaping device

The shaping device is the last component in the continuous processor for achieving the desired geometry of the energetic material. The shaping device consists of a die of desired shape for extruded propellant, injection loading unit for high explosives or a vacuum casting unit for cast propellant. For the application of extrusion, the final elements of the screw configuration are pressurisation elements to generate a high extrusion pressure. The downstream product handling system consists of a product take away system, with a guillotine cutter and specially guided vehicle [26] or conveyor belt to remove the collected product. In the case of composite propellant for large boosters, the continuous processor functions as a mixer and after mixing, the material is collected in a container. The material is subsequently cast into the rocket motor by vacuum casting. In such cases, the discharge of material from the screw extruder is generally vertically downwards and the screw is supported on the discharge side by outboard bearings, with suitable reverse screw elements to avoid ingress of material into the support bearings. NSWC (Naval Surface Warfare Centre), USA, have developed twin screw mixing coupled with a vacuum casting system to both mix and cast composite propellant continuously and remotely [27]. In this process, the casting system is positioned in front of the extruder and connected with pipe fittings. The material, after mixing, enters into the casting system where it is cast into the warheads. This process is claimed to reduce exposure of personnel, thus improving safety and quality, as well as reducing cost.

# 3 Process Development Methodology

In energetic materials processing, being a potentially hazardous operation, safety is paramount in the process development. This necessitates the exhaustive characterisation of the energetic materials coupled with simulation of the extruder [3, 26]. A schematic flow chart for continuous process development of energetic materials is shown in Figure 5. The process development starts with hazard analysis of the process. It involves safety characterisation tests such as sensitivity to friction, impact, shock and electrostatic discharge, autoignition temperature, decomposition pattern, *etc*. To simulate the friction and shearing in the TSEs, a liner tribometer, with high speed UV and IR cameras, has also been employed [28]. The rheological characterisation includes various material functions such as viscosity, normal stress differences and other flow behaviour. The important features with regard to mechanical design of the extruder are material of construction, clearance between screw and barrel

and manufacturing tolerances, drive system, seals, *etc*. Furthermore, various computer modelling and simulation tools [29] are used for detailed analysis of the extruder. These simulation tools are very important for predicting the distribution of various process parameters inside the extruder, which are critical to safety, such as temperature, pressure, *etc*. Trials with inert materials and residence time distribution studies are used to validate the computer models. The trials with inert materials help to assess the overall process configuration, mechanical design of the screw and other components, process limit alarms and trip set points. Finally, all of the safety measures are again reviewed before processing trials with live energetic materials can begin.



**Figure 5.** Flow chart for the development of a continuous process for energetic materials.

## 4 Safety Features of Continuous Processors

The various safety issues relevant to continuous processing are temperature control, pressure control, torque control, static electricity and mechanical construction. Although TSEs have less hold-up compared to batch mixers, work done on the material is more intense, resulting in increased risk. Few accidents of thermal decomposition of materials in a TSE due to thermal energy build-up because of improper die design and material rheology have been reported [30]. In a continuous processor, viscous dissipation heat due to intense shearing of the material in the narrow flow channels inside the extruder and dies, results in an increase in temperature of the material and hot spot formation [3, 26]. This necessitates precise control of the temperature in various sections of the

processor, which is achieved by providing maximum surface area to volume for the circulating heat exchange media. In addition, temperatures are measured in critical zones of the extruder and used for limit alarms and trip set points.

The other important safety issue is pressure control inside the extruder, as the process involves pressurisation through the die assembly. In such cases, excess pressure may result in intra-granular shear and ingress of material through the seals into the bearing or gearbox, leading to accidents [2]. The pressure control measures consist of pressure monitoring using pressure sensors at various locations and a pressure release system. A continuous processor employs a twin level pressure release system; automatic tripping of the extruder in case of over-pressure followed by hydraulic opening of the barrel [18]. These pressure release systems have response times of less than 1 second. Furthermore, the torque control in the extruder is provided by torque monitoring using direct torque sensors for a trip set point. Some designs also employ a safety clutch, tension bar or shear pin, which in case of over torque, automatically disengages the shaft from the drive [20]. To mitigate the concern of electrostatic discharge, all of the electrical fittings of the processor are explosion proof and intrinsically safe. The lubricating grease and rubber material of the mechanical seals are also made of electrically conducting materials.

The mechanical construction of the processor has an important bearing on safety. Generally, the clearance between the screw and the barrel are of the order of a few microns. Under certain conditions, there is the possibility of frictional ignition of the material due to metal to metal contact. Hence, the manufacturing tolerance for the screw and shaft and clearance between the screw and the barrel are very critical. In many designs, either the barrel lining or the screw elements are made of non-sparking materials (*e.g.* Cu-Be alloy) which reduces the possibility of frictional ignition.

Some of the other safety features include a solids feeder isolation system [22], which isolates the energetic material feeding stream from the extruder and increases the overall safety of the continuous processor. The feed isolation system consists of a baffled feed funnel to divert flame from the extruder and to reduce the flame travel upwards into the raw material feedstock. In addition, during processing of pyrotechnics, inert gas is also employed to reduce the oxidant concentration in static sensitive areas [14]. Continuous processors are also equipped with a remote control system and a number of flame detectors (UV/IR sensors) for maximum coverage of the process area, coupled with a quick response deluge system [2].

## 5 Residence Time and Shear Distributions (RTD & RSD)

The mechanism of mixing inside a continuous processor has been studied by various diagnostic techniques, such as particle tracking (X-ray based or positron emission particle tracking, PEPT) [31], residence time distribution (RTD) [32, 33], residence shear distribution (RSD) [34], etc., or by numerical simulation tools. The characterisation of a twin screw is often described by RTD, which gives information on the time over which the material fed into the extruder will reside in the extruder. The residence time inside the extruder determines the dispersive and distributive mixing and depends upon the screw configuration and process parameters such as speed of screw rotation, material properties, temperature, etc. The experimental RTD measurements involve the injection of a tracer, usually a coloured dye in the form of an impulse or step input and measuring its concentration (response) by an optical probe [32] or a dielectric response [35] at the exit of the extruder. The RTD response is expressed in terms of various age distribution functions. The RTD information is useful for various purposes, such as analysing the mixing process, the chance of degradation in the extruder, specifying the feed accuracies, scale-up and designing the process control algorithms [32, 35]. The RTD characterisation of a TSE can be used for investigating transient processing conditions for the evolution of gradient architectures [32, 33].

The intensity of mixing in an extruder also depends upon the amount of stress applied to the material. The RTD studies give information on the transport characteristics but do not give information about the stress history of the material. The stress history is essential for energetic materials for assessing the possibility of localised heating due to excessive shear, posing potential safety problems. Excessive shear also affects the microstructure of energetic materials and thus affects the product quality. The shear history inside the extruder is determined using a methodology called residence shear distribution (RSD). For characterising RSD, calibrated microencapsulated sensors (CAMES) are employed [36]. CAMES have an organic dye containing a core liquid encapsulated in the polymeric micro-capsules which rupture at predefined shear stresses depending on their diameter. Upon rupture, the die is released and is detected at the extruder outlet by optical methods. Thus, such RSD studies give the stress history experienced by the material inside the extruder. Hence, the characterisation of RTD along with RSD in the extruder is essential for optimising the screw geometry and process parameters.

## 6 Rheological Aspects of Energetic Materials

Energetic material suspensions are non-colloidal concentrated suspensions exhibiting complex rheological behaviour, expressed as pseudo-plasticity with yield stress, thixotropic and visco-elastic characteristics. They are affected by a number of factors, such as volume fraction of the solids, particle size and shape, the binder matrix, particle-particle and particle-matrix interactions, *etc*. The rheological characterisation is extremely important in designing the extruder, assessing safety during processing, mathematical modelling and optimization of the process parameters [3, 18]. The important rheological issues which pose special challenges during continuous processing are apparent slip behaviour, demixing due to migration of particles in a transverse direction, mat formation and binder filtration in the axial direction, die swell, *etc*. [37, 38].

Apparent slip results from the formation of a binder rich zone near the wall. The occurrence of slip in the flow of energetic suspensions complicates the measurement of the rheological properties and modelling of the flow behaviour. It also reduces the extrusion pressure for a given screw speed, affecting the properties of the final product. Other flow instabilities observed in energetic materials are particle jamming and binder filtration, when these materials are forced through the narrow flow channels of the screw or die. Such behaviour can lead to demixing of material, extrudate distortion and pulverisation of energetic particles, posing a safety threat. Furthermore, shear induced particle migration in the pressure driven flow of energetic materials leads to concentration gradients in the transverse direction. Such concentration gradients result in variations of important properties such as the burning rate of solid propellants in the transverse direction, commonly known as a hump effect [9, 39]. Furthermore, the visco-elastic material functions of energetic suspensions, such as normal force differences, are also important in understanding various effects such as die swelling and migration of particles during extrusion [38].

# 7 Specialised Applications of Continuous Processing

TSE based continuous processing has capabilities for various specialised applications, such as the manufacture of functionally graded materials (FGM). These materials possess a gradual variation in composition or microstructure and have the capability of multifunctional performance. Functionally graded rocket and gun propellants have the potential to replace geometrically complex grain configurations with a much simpler geometry, improving reliability while

meeting the same performance [32]. The dependence of various properties of energetic materials, such as burning rate as a function of the concentration of the feed stream [33, 40], forms the basis for the evolution of gradient architectures. In a steady state operation of a continuous processor, this is achieved by introducing dynamic changes in the ingredient feed rate, which results in changes in the composition and subsequently the properties of the extrudate. This combinatorial approach of transient operating conditions and/or feed conditions is also used for developing new energetic formulations. The inherent back-mixing in the extruder results in a gradual change in composition in response to abrupt changes in the feed rate of the ingredients. In addition, variations in the screw speed [40] and shear induced migration [41] are also known to produce such graded materials.

Nano materials are being increasingly used in energetic material for a variety of applications. However, their incorporation into energetic suspensions poses significant challenges in mixing and handling. Continuous processing, due to its intensive shearing, feed control and adaptability of additional mixing sections in the extruder, makes it suitable for processing nano materials. Nano metal incorporation into energetic materials by encapsulating them into various binders is also carried out using mini TSEs [42]. Furthermore, continuous processes, based on two TSEs, are used for the manufacture of co-layered energetic materials [43]. The process involves the processing of two different energetic materials using two TSEs, followed by extrusion through a special die. This process is advantageous as conventional methods involve multiple steps, higher cost, wastage and safety risks.

#### 8 Conclusions

The twin screw extruder based continuous process for energetic materials has tremendous potential to replace the conventional batch process. The reduced hazards in continuous processing enable the processing of conventional as well as new energetic materials with improved safety. The continuous process has been successfully implemented for many applications, such as extruded propellants for military and civil applications, with significant cost reductions and improved reliability. Furthermore, efforts are being made for the use of the continuous process for large scale applications such as booster motors for space shuttles. However, safety being of paramount consideration for energetic materials, a detailed hazard analysis of the twin screw extruder based process is essential for the process development. This may be accomplished by a complete understanding of the characterisation of energetic materials and the mechanical design of the extruder and other components, along with mathematical modelling and simulation.

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