Flood Feeding Small Batches on a Co-rotating Intermeshing Twin Screw Extruder.

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PURPOSE

Describe how to successfully process small batches on a co-rotating twin-screw extruder (TSE) utilizing a flood feeding method to evaluate formulation efficacy in an R&D environment. Analyze results from flood feeding for several different pharmaceutical grade materials. Evaluate flood feeding to help determine viability of a continuous extrusion process when only a small batch is available for testing. (Twin-screw extruders are typically starve fed and typically over-torque when flood fed.)

BACKGROUND

The two basic feeding methods of extruders include flood feeding and starve feeding. Flood feeding is typically used in single screw extrusion where the feed hopper is filled, and the extruder screw rpm determines the feed rate. Starve feeding is typically used in twin screw extrusion where a feeder is used to meter the material into the system at a predetermined rate where rpm and throughput are independent of each other. On a TSE, flood feeding is atypical because it will normally over-torque the machine.

Pharmaceutical processes in early development stages often require minute quantities of materials to be processed. There have been challenges evaluating small batches at these low quantities. Auger style feeders often require 200+ grams of material to reach equilibrium. Flood feeding a twin-screw extruder has previously not been evaluated to determine viability due to torque limitations. The shallow flight depth (1 mm) and high torque capacity of the zse-16 mm TSE, plus an appropriately designed feed section of the screw allows for operation like this.

METHOD(S)

The Leistritz zse-16 mm co-rotating TSE at 26:1 L/D (1.2 OD/ID, 72 Nm of torque capacity) was used with two different screw designs (figure 1 a&b). A hopper was placed at the feed barrel and a 50 g batch was input into the hopper and screws. The extruder screw rpms forwarded the materials for processing. Several conditions (table 1&2) of 50-300 rpm, and a flat temperature profiles of 160 °C were used. Materials with different particle sizes and bulk densities were chosen including HPMC, PVP (Kollidon VA64), EVA, and PE Purge (PE Purge) (table 3). The rate was checked with a 60 second catch sample for each material and rpm.

RESULT(S)

A screw designed to limit the intake of material using 10 mm pitch conveying elements while still creating the downstream effects of a starve fed process allows for the benefits of varying degree of fill and mixing efficiencies (figure 1a). HPMC and PVP, both powders, showed similar effects vs. rpm. at 300 rpm and higher. The high rpm created an agitation/propeller effect and aerated the powder preventing it from being taken away as efficiently. This can be seen with PVP from 150 to 300 rpm where the rate decreases from 9.2 g/min to 8.3 g/min. It can also be seen less drastically for the HPMC which only slightly increased from 15 g/min to 16.4 g/min. This is because the particle size and bulk density were both higher than the PVP. As bulk density increased with powdered EVA, 0.43 g/cc to 0.95 g/cc, the effect becomes negligible.

PE Purge with 1.3 mm particle size, was also tested. Because of the low surface area, the pellets were not affected in the same way at higher rpm and the rate consistently increased with higher screw rpms.

In comparison, a screw with a more traditional higher pitch feed section (figure 1b), was also evaluated. The rate did increase approximately 40% on average, as expected. With the higher pitch elements, the agitation/propeller effect that was seen using screw #1 did not have an effect and the rate continued to increase linearly. Overall, the residence times for both screw designs were comparable to similar conditions with a starve fed system where 4 g/min of PVP at 200 rpm produced a residence time of 130 seconds. All small batches of 50 grams were successfully tested and approximately 2-3 grams were lost on the screws for each. The extrudate was amorphous and the color indicated little to no degradation.

Table 1: Flood feeding results for screw #1.

	HPMC (Powder)		RT	PVP (Powder)		RT	EVA (Powder)		RT	PE Purge (MicroPellets)		RT
RPM	g/min	g/hr	sec	g/min	g/hr	sec	g/min	g/hr	sec	g/min	g/hr	sec
50	8.6	516	150	3.7	224	210	4.1	246	188	1.8	108	285
150	15	900	-	9.2	550	90	12.5	750	65	7.9	474	82
300	16.4	984	-	8.3	498	65	24.5	1470	30	11.9	714	40

Table 2: Flood feeding results for screw #2.

	PVP (Powder)		RT	EVA (Powder)		RT	PE Purge (MicroPellets)		RT
RPM	g/min	g/hr	sec	g/min	g/hr	sec	g/min	g/hr	sec
50	5	300	210	5.6	336	155	3.6	216	150
150	13.5	810	75	15.3	918	50	9.8	588	70
300	24.6	1476	35	30.1	1806	25	16.9	1014	35

Table 3: Material properties.

	Bulk Density (g/cc)	Particle Size (d90)	
HPMC	0.43	237	um
PVP	0.2 - 0.3	139	um
EVA	0.95	900	um
PE Purge	0.57	1.3	mm

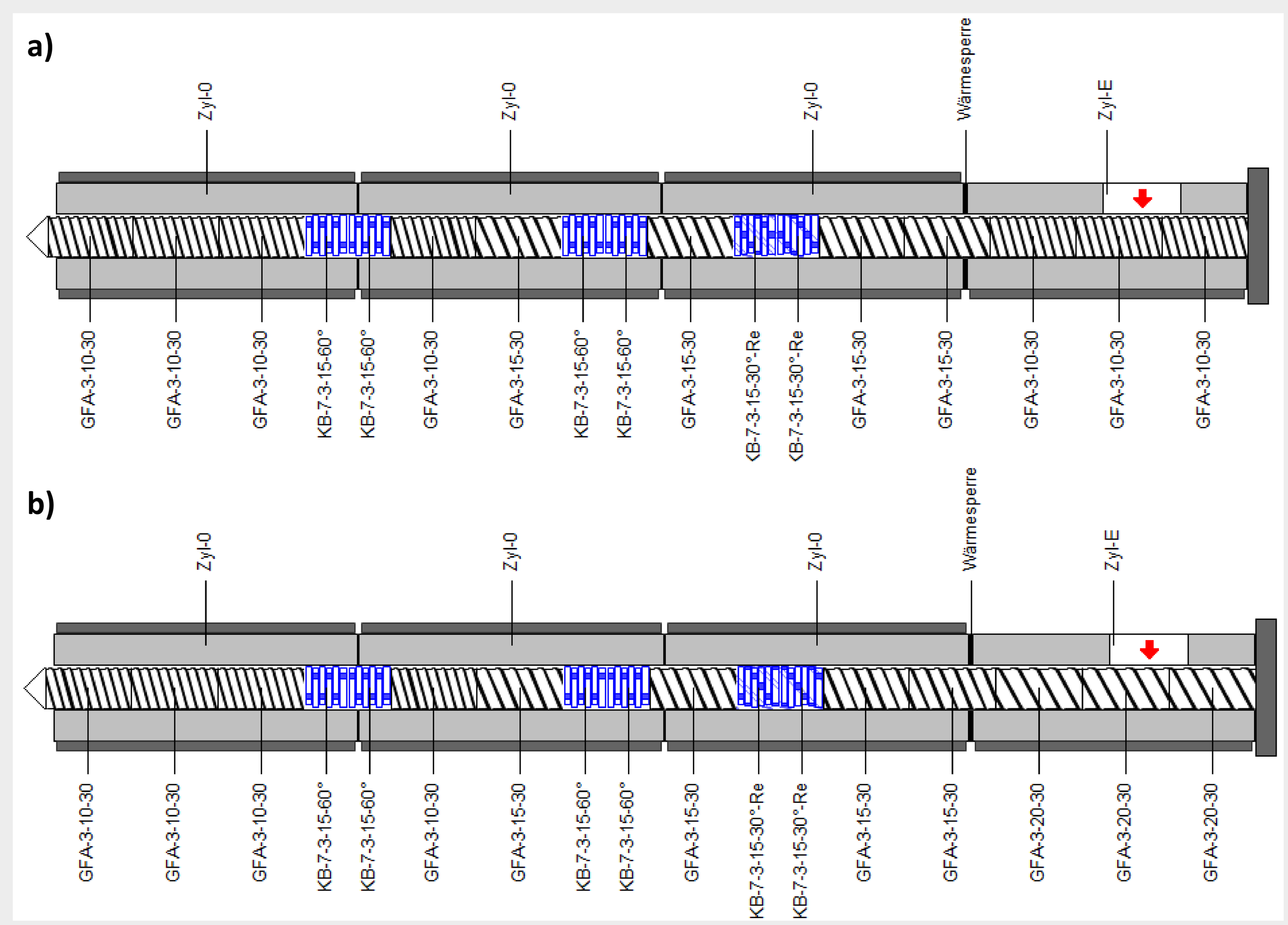

Figure 1 a&b: zse-16 mm co-rotating, intermeshing screw designs. a) Screw #1, b) Screw #2.

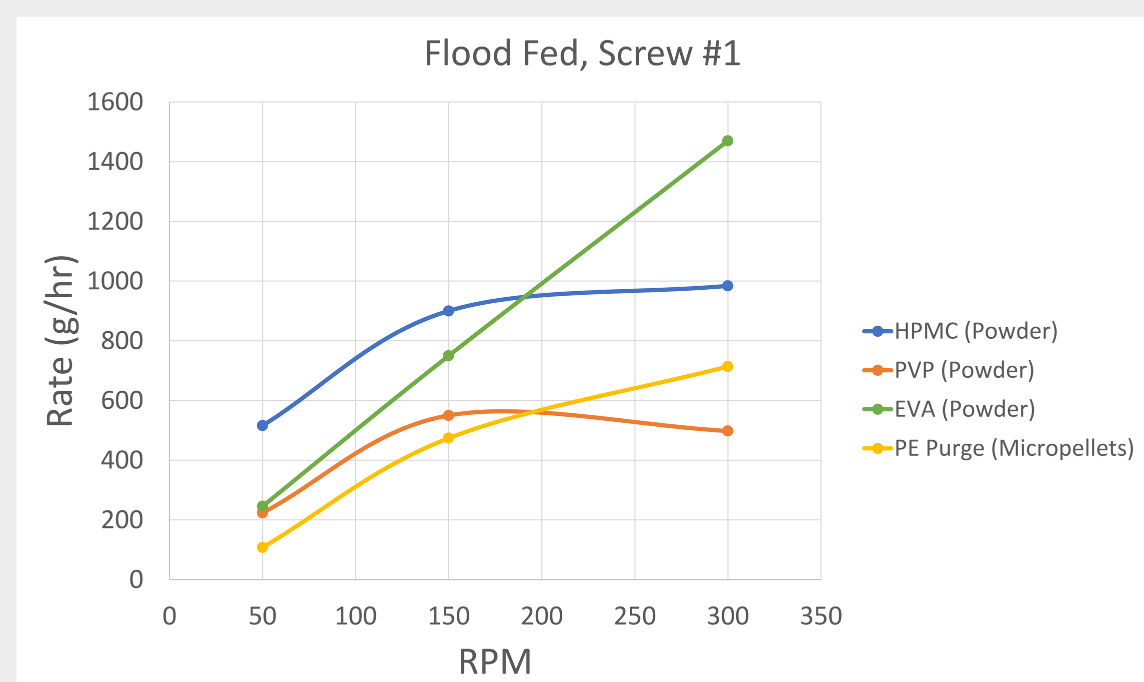

Figure 2: Feed rates of various polymers in a flood fed system with Screw #1.

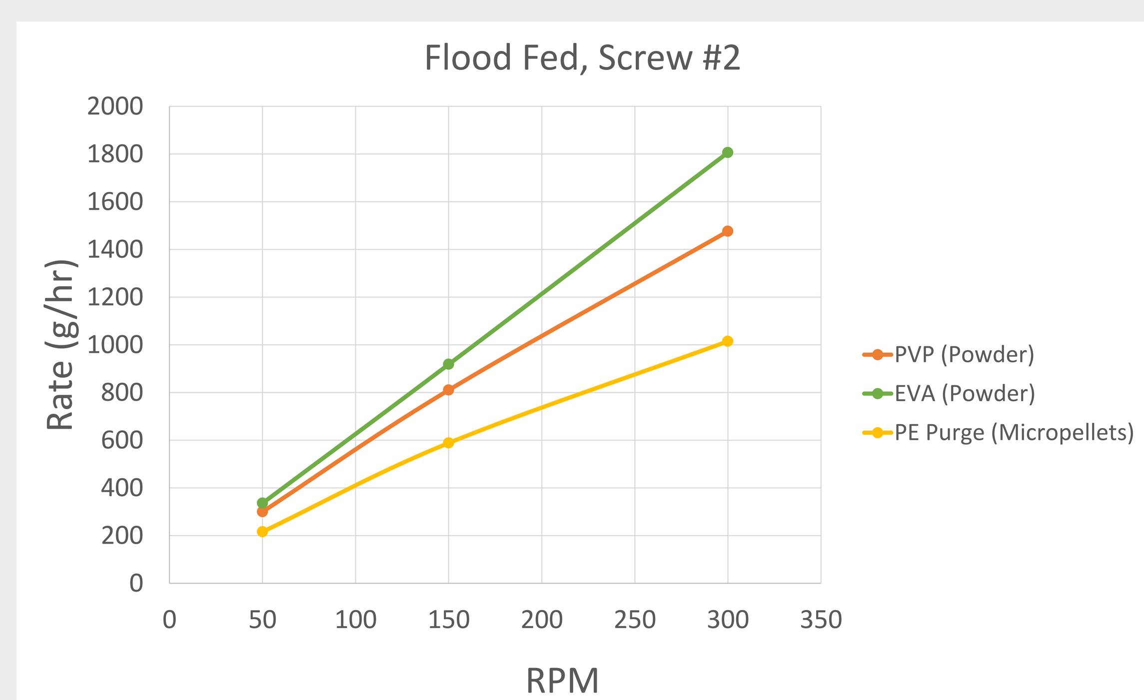

Figure 3: Feed rates of various polymers in a flood fed system with Screw #2.

CONCLUSION(S)

Implementing a flood feeding technique on a twin-screw extruder with a modified and atypical screw design effectively simulates a starve fed TSE system allowing for representative extrusion samples to be produced from small batches with near full batch utilization for sample evaluation. For samples produced at both low and high rpms, the degree of fill remained constant and the energy imparted from the screws repeatable and consistent. A simplified operating system combined with the reduction in waste inherent with auger style feeders presents a considerable benefit when only limited quantities of expensive excipient/APIs are available. Overall, this feeding method enables a quick and efficient methodology to process small volume pharmaceutical batches via twin screw extrusion.

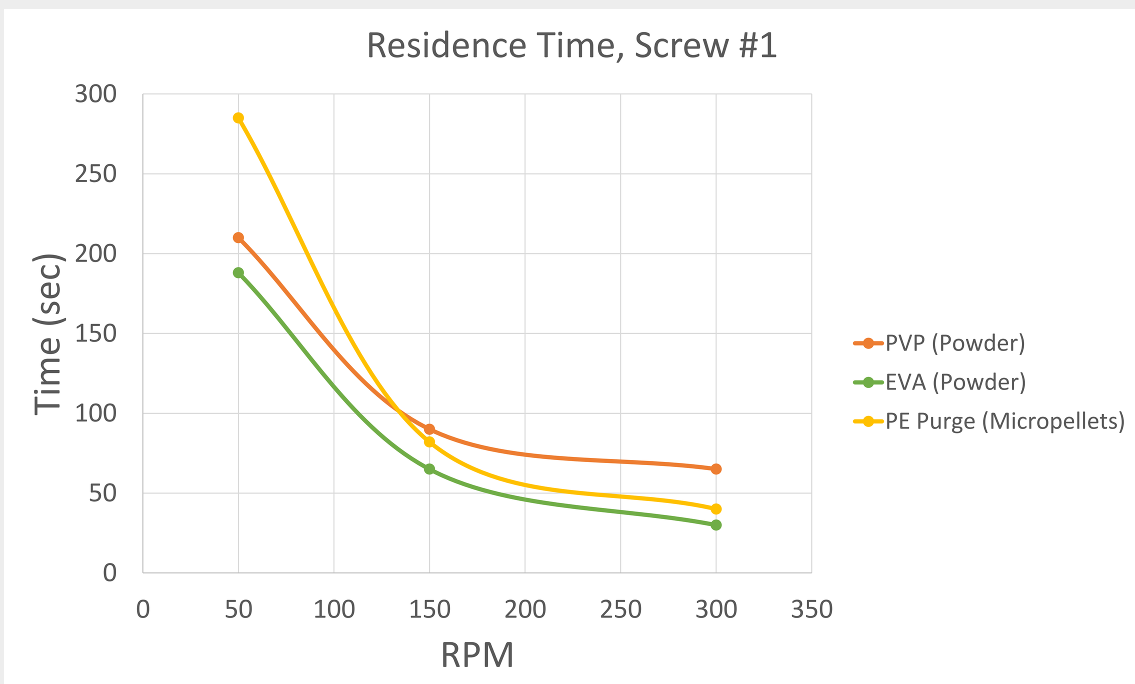

Figure 4: Residence Times using screw #1. (screw #2 trends are comparable)

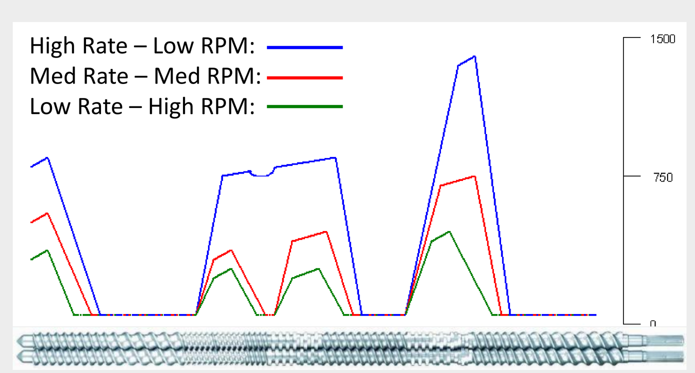

Figure 5: RPM vs. Degree of Fill.

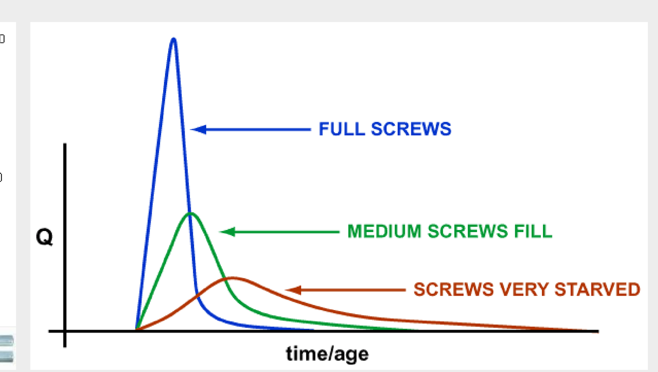

Figure 6: Residence Time vs. Degree of Fill.

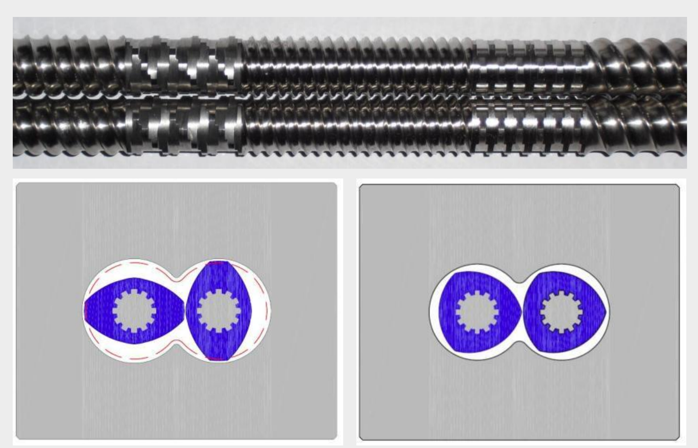

Figure 7: zse-16 mm screws with 1 mm flight depth and Bi-lobal vs. Tri-lobal screws.

Figure 8: Example of zse-16 mm extruder.

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