CONTINUOUS TWIN-SCREW MELT GRANULATION BINDER ADDITION METHODS.

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Introduction -

Twin-Screw Melt Granulation (TSMG) is an emerging processing method which enables continuous processing of powder blends to improve properties. Traditionally, TSMG is performed by feeding a pre-mix into the main port of one extruder. This study will compare three different sequences of binder addition methods including: 1 – pre-mixing, 2 – melt binder addition, and 3 – side stuffing API into a melt. The objective was to compare the effect of binder addition methods on granule formation stability and minimizing API particle size reduction.

Figure 1: Method 1 Pre-mixing.

Figure 2: Method 2 – Melt Binder Addition.

Figure 3: Method 3 – Side stuffing API.

Leistritz

Methods -

Granules containing 80% lactose monohydrate (LAC, solid particles) and 20% hydroxypropyl-cellulose (HPC, Klucel™ ELF grade, binder) were prepared. For method 1, the powders were pre-mixed and fed into a Leistritz ZSE 18 mm corotating twin-screw extruder (25:1 L/D, 3.2 cc/dia). For method 2, The LAC was fed into the main feed port of the ZSE 18 mm (25:1 L/D) and a nano-16 twin screw extruder was used to melt and pump the HPC barrel # 2 of the ZSE-18 process section. For Method 3, the HPC was fed into the main feed port of the ZSE 18 mm (40:1 L/D) and the LAC was fed into a side stuffer (barrel #4). RPM and feed rate were investigated for each method. Particle size of LAC in the granules was characterized using Sympatec® cuvette.

Results -

Each formulation and setup produced granules. The premix (method 1) was influenced by degree of fill (higher rate and lower rpm) which allowed more time for the binder to be fully activated and facilitated larger and more uniform granule growth. With melt addition (method 2), higher rpm and lower feed rate resulted in more uniform granule growth indicating more mixing was required to properly incorporate the melt. This method gave the most particle size retention of the three methods and the granule temperature was also reduced by 5 °C. The split feed (method 3) produced uniform granules regardless of rpm or rate conditions. The particle size of the LAC (22.3 µm) was reduced in all three addition methods. Method 1 mean diameter = 10.0 µm, method 2 = 12.3 µm, method 3 = 4.5 µm.

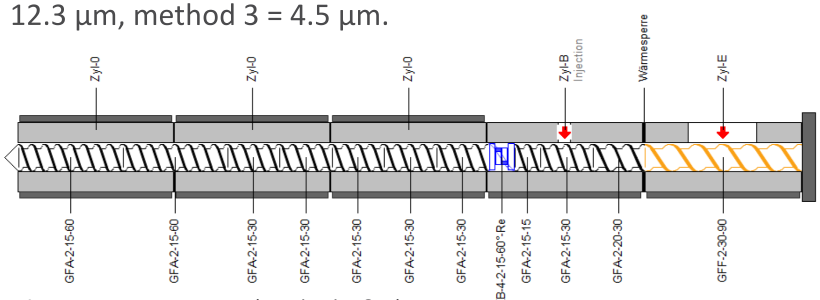

Figure 4: ZSE 18 mm HP (Method 1 & 2).

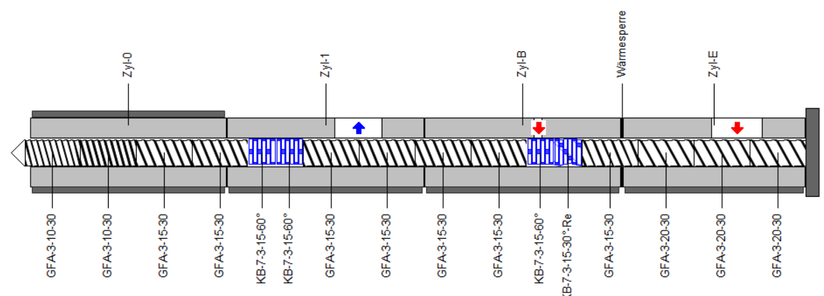

Figure 5: Nano 16 mm (Method 2 – used to melt HPC and melt feed into 18 mm, figure 4)

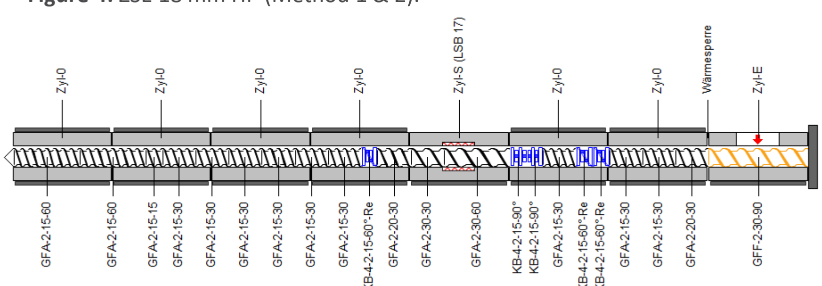

Figure 6: ZSE 18 mm HP (Method 3).

Conclusion/Implications -

The binder addition method in TSMG was impactful on processibility, showing that the traditional method of TSMG may not be the most efficient in preventing particle size reduction. The melt addition method resulted in the lowest particle reduction seemingly because the API was not exposed to the forces required to melt the binder and benefited from its lubricity after the HPC was properly melted and injected; only enough energy was used to mix and facilitate granule growth. Method 3 resulted in the highest particle size reduction due to high viscosity HPC causing particle attrition. With a different melt zone design this attrition could potentially be lessened.

Learning Objectives -

Explain the purpose of twin-screw melt granulation.; Describe the leading factor of granule growth for each method.; Analyze the reason for particle retention in method 2.

Table 1: Processing Results of Methods 1 - 3.

#	Parameters		Method #1 (Pre-mix)			Method #2 (liquid add.)			Method #3 (side stuffer)		
	Screw Speed (rpm)	Feed Rate (kg/hr)	Granule Temp. (°C)	Residence Time	Torque (%)	Granule Temp. (°C)	Residence Time	Torque (%)	Granule Temp. (°C)	Residence Time	Torque (%)
1	100	1	57	26	6	56	47	8	55	36	7
2	300	1	59	10	8	60	11	11	61	13	11
3	200	2	61	12	7	58	18	9	67	21	13
4	100	3	74	29	10	63	34	8	68	30	12
5	300	3	67	8	9	60	11	12	65	15	13