Investigation of a new Chill Roll with integrated mill for hot-melt extrusion with Soluplus®

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Purpose

To investigate a hot-melt extrusion process in a new complete setup starting from powder materials yielding in melt extruded powder for tableting in a single continuous process step.

Materials and Methods

Soluplus® is a polyvinyl caprolactam-polyvinyl acetate – polyethylene graft copolymer (13% PEG 6000 / 57% vinyl caprolactam / 30% vinyl acetate). It is a polymeric solubilizer with a glass transition temperature of 70°C. Soluplus® is available as free-flowing white to slightly yellowish granules.
Soluplus® was extruded on a Leistritz ZSE 18 HP-PH co-rotating twin-screw extruder in different process settings based on a statistical design-of-experiments plan. As downstream equipment a BBA Innova chill roll (type: CCRM-20/12PH) with integrated Frewitt conical sieve mill (type: Frewitt ConiWitt-150) we used. In the experimental plan extruders feed rate (2-5 kg/h) and its screw speed (100-400 rpm) were changed as independent variables to also influence the product melt temperature. In two different ANOVAs the impact of feed rate and screw speed versus in a second ANOVA the product melt temperature (150-190 °C) were investigated regarding their potential influence on the flake temperature, the flake thickness and the product powder temperature after processing through the chill roll and the conical sieve mill. The settings such as rotor speed and material-load for the mill were kept constant same as the settings for the chill roll. In a separate experimental series the chill roll speed was varied to investigate the impact on the resulting flake temperature and on the powder temperature after milling. The cooling water of the chill roll was kept at 14°C +/- 1°C.

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Figure 2: Leistritz ZSE 18 HP-PH extruding Soluplus® to the Chill roll

Extruder Setup

The extruder was setup in 40 L/D functional length. To redirect the melt flow after extrusion downwards the chill roll a special die head was used with extra heating for temperature control. The screw geometry can be seen in figure 1.

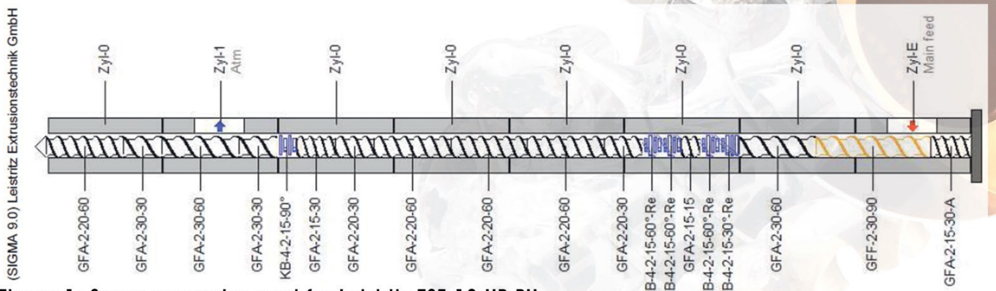

Figure 1: Screw geometry used for Leistritz ZSE 18 HP-PH

After the extruder the chill roll was attached as can be seen on figure 2. The melt was then spreaded over the width of the chill roll. After passing the chill roll, flakes were obtained which immediately were crushed by a breaker unit at the end of the chill roll and fall down into the mill.

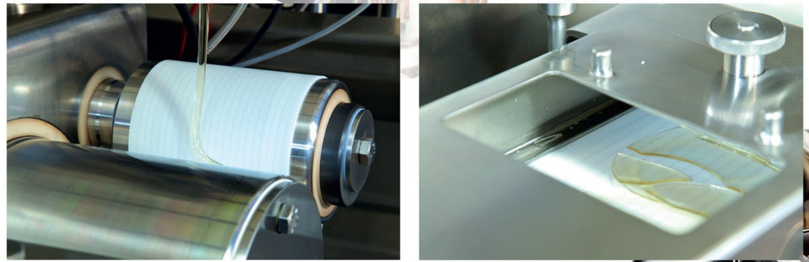

Figure 3&4: Detailed view of the chill roll

Results and Discussion

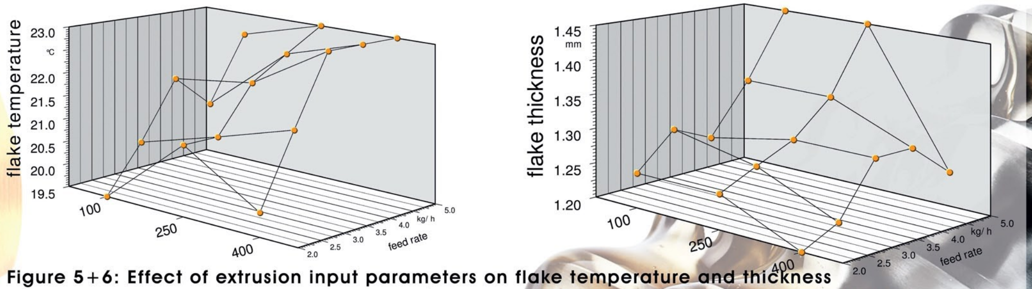

Figure 5+6: Effect of extrusion input parameters on flake temperature and thickness

		ANOVA 1			ANOVA 2		
	melt temperature at die [°C]	flake thickness [mm]	flake temperature [°C]	Powder temperature [°C]	flake thickness [mm]	flake temperature [°C]	Powder temperature [°C]
RMS	5,3546	-	0,46336	0,60043	-	0,86047	-
RMS/Ym	0,033	-	0,021	0,022	-	0,04	-
R^2	0,837	-	0,853	0,646	-	0,415	-
R^2adj	0,763	-	0,819	0,529	-	0,376	-
Q^2	0,58	-	0,712	0,222	-	0,249	-
W^2	0,347	-	1	0,237	-	-	-
DF	11 (9)	-	13 (11)	12 (10)	-	15	-

Above table shows a reasonable correlation of melt temperature to feed rate and screw speed. This was used as argument to consider melt temperature as independent variable for the ANOVA 2. In ANOVA 1 feed rate (FR) and screw speed (SRR) were considered as independent parameters. It can be shown that there is no effect of FR and SRR on the flake thickness. But a good correlation of FR and SRR to the flake temperature after passing the chill roll was found with a standard error of prediction of 2%. Similar an impact of FR and SRR on the powder temperature after milling can be found, but there is less statistical significance as expected.

In ANOVA 2 the authors found no correlation at all between the melt temperature feeding the chill roll and the flake thickness nor the powder temperature after milling. A certain correlation between the melt temperature and the flake temperature can be seen but with a poor statistic significance. The outlet melt temperature range was 40 K and results in flake temperature were only 4 K. Further trials showed that with a higher chill roll cooling water temperature > 20 °C resulted insufficient cooling of the melt.

Concluding it can be said that the chill roll is a downstream device which allows reliable consistent cooling of an extruded melt independent from the compounding process in the extruder.