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## Sample Pages

# Co-Rotating Twin-Screw Extruders: Applications

Klemens Kohlgrüber

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## Preface

The twin-screw extruder is of great importance in various industrial sectors, such as in the plastics, food, and pharmaceuticals industries. The editor published a book on this subject in late 2007 as both English- and German-language editions, the former of which was called simply "Co-Rotating Twin-Screw Extruders". In the meantime a considerably extended and updated 2nd German edition of the book *(Der gleichläufige Doppelschneckenextruder)* was published in 2016. The preface of this German edition translated into English is appended below. About half of this German edition, with a focus on the fundamentals of co-rotating twin-screw extruders, was published in English as "Co-Rotating Twin-Screw Extruders: Fundamentals" in 2019. This current book corresponds to the second half of the German edition, focusing on the applications and functional zones of these extruders. In particular, the following focal points are described:

- Solid transport and melting
- Degassing of polymer melts
- Scale-up and scale-down
- Extruder technology, series, housing variants, materials
- Compounding in practice, color masterbatches
- Reactive extrusion, food extrusion, pharmaceutical applications

The editor would like to thank all the section authors, especially for their English translations. My thanks also go to Mr. Thomas König, who has clarified technical terms and also carried out an overall review. Dr. Smith from Carl Hanser Verlag has again made a considerable contribution to the success of this English edition and has given the editor exceptional support!

Klemens Kohlgrüber, September 2020

#### Preface to the Second German Edition

The 50<sup>th</sup> anniversary of the "twin-screw compounder (ZSK)" was the occasion for the first edition of this book. Therefore, only authors of the companies Bayer (licensor, Chapter 1) and Werner & Pfleiderer (today Coperion, licensee) were involved.

The elaboration of the first edition took place under considerable time pressure because, after the first idea for this book, it should appear on the occasion of the Plastics and Rubber Fair "K 2007".

For the present edition it was my intention as editor to incorporate especially the following improvements and extensions:

- The participation of different companies and universities.
- A greater involvement of technical topics.
- Naturally the consideration of the further developments that have been made in the meantime (concerning screw geometries, calculation approaches, applications, ...).
- The basics of the extruder technique and the process descriptions by means of models should be described in more detail.
- Especially application-oriented practical examples should be incorporated to a larger extent.
- The contributions should be better coordinated.

This has succeeded now in many points of the present second edition. The reader may decide himself on the qualitative improvements. The extent has grown because of the number of contributions and by the more detailed depiction of the basics. The book should now be readable for apprentices in technical professions and simultaneously represent a benefit for experts due to the described applications. Some chapters are partly overlapping; this has been done intentionally. Due to different authors with different explanations regarding the same facts, some topics will become clearer. When coordinating the contributions I have tried to ensure that largely the same denominations and formula symbols have been used. The description of a topic and the interpretation of findings have been the focus of the respective author. In particular cases, a fact can be seen differently by different authors, for example the evaluation regarding usefulness of models (for more details please see Section 1.4). For this reason I refrained from the original intention to write a summary for each contribution. This could lead to an assessment being "counterproductive" in the sense of cooperation.

I would like to take this opportunity to offer heartfelt thanks to all authors for their contributions! I thank Mr. Lechner for the coordination of the contributions of Coperion.

My thanks go to all those who contributed with their comments on improvements and detailed definitions. Furthermore I would like to thank my daughter Kristina for the review of my contributions.

Here my special thanks are due to Ms. Wittmann of the publisher Hanser! She always accompanied the "book project" from the preparation phase until the end and gave valuable contributions for designing the book.

Klemens Kohlgrüber, May 2016

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# Contents

Pre	face		V
The	Auth	iors	VII
The	Editor		VII
The	Coout	hong	vm
me	Coaut	nors	VIII
1	Func	tional Zones in the Extruder	1
1.1	Trans	sport of Solids into and in the Extruder, Feed Limits	1
	1.1.1	Characteristic Values and Calculation Possibilities	2
	1.1.2	Feed Limitations	8
		1.1.2.1 Granulates	8
		1.1.2.2 Powder	9
		1.1.2.3 Flakes	11
		1.1.2.4 Low-Melting Components	11
1.2	Melti	ng of Thermoplastics	12
	1.2.1	Tasks of the Melting Zone	12
	1.2.2	Screw Elements and Screw Configuration	13
	1.2.3	Measuring Techniques	14
	1.2.4	Essential Steps of Melting	16
	1.2.5	Calculation Models	18
1.3	Mixin	ng and Dispersion	23
	1.3.1	Overview, Principles, and Experiments	23
		1.3.1.1 Distributive Mixing – Mixing in Laminar Flow	23
		1.3.1.2 Dispersive Mixing	29
		1.3.1.3 Determining the Mixing Quality	36
		1.3.1.4 Symbols Used in Section 1.3.1	38
	1.3.2	Three-Dimensional Calculations of Mixing and	
		Residence Time Behavior	40
		1.3.2.1 Summary	48

1.4	Devol	atilization of Polymer Melts	49
	1.4.1	Phase Interfaces and Surface Renewal	49
		1.4.1.1 Liquid Distribution and Degree of Filling	49
		1.4.1.2 Devolatilization Times	62
	1.4.2	Concentration in the Devolatilization Zone	70
		1.4.2.1 Influence of Dimensionless Groups	70
		1.4.2.2 Bubble-Free Liquids	71
		1.4.2.3 Influence of Surface Expansion by Bubbles	76
	1.4.3	Design of the Devolatilization Zone	76
	1.4.4	Numerical Simulation of Film Degassing	79
2	Scale	up and Scale down	97
2	Scale		07
2.1	Introc	luction and Basis Rules for Thermally Sensitive Products	8/
	2.1.1	Dissimilarity	88
	2.1.2	Comparison of Production Machines	88
	2.1.3	Scale-down and ways of Design	89
		2.1.3.1 Product Temperature	90
		2.1.3.1.1 Product Cooling via the Housing Wall	91
		2.1.3.1.2 Temperature Change by a Pressure	02
		21313 Temperature Increase by Power Input	03
		21314 Thermal Product Degradation	94
		21315 Temperature Increase and Internal Friction	96
		21316 Relevance of the Shear Rate	97
		21317 Basis Rules for Scale-up/down	99
		2.1.3.1.8 Basis Equations for the Examples	99
	2.1.4	Summary/Prospects	104
2.2	Scale	up and Scale-down by Model Laws	106
	2.2.1	Basic Problem	106
	2.2.2	Simple Scaling Approach	107
	2.2.3	Model-Based Scaling Approach	108
		2.2.3.1 Model Theory	108
		2.2.3.2 Model Exponents	118
		2.2.3.2.1 Lengths Exponent	118
		2.2.3.2.2 Screw Speed Exponent	119
		2.2.3.2.3 Channel Depths Exponent	120
		2.2.3.2.4 Pitch Exponent	120
		2.2.3.2.5 Relationship between Channel Depths	
		Exponent, Selected Boundary Condition,	
		and Resulting Throughput	120
		2.2.3.3 Heat Flows via the Barrel	122
	2.2.4	Experimental Results	123

2.3	Scale-	-up and S	Scale-dowr	with Characteristic Numbers	126
	2.3.1	Charact	eristic Nu	mbers of the Whole Machine	127
		2.3.1.1	Dimensio	nless Throughput	127
		2.3.1.2	Specific I	Energy Input	128
	2.3.2	Geomet	ric Scale I	ransfer	128
		2.3.2.1	Geometri	cally Similar Machines	128
		2.3.2.2	Extruder	Speed and Torque	129
		2.3.2.3	Scale Tra	nsfer with Different Geometries	130
			2.3.2.3.1	Partially Filled Zones	130
			2.3.2.3.2	Pressure Buildup	133
		2.3.2.4	Dimensio	nal Analysis for Real Product Behavior	134
			2.3.2.4.1	Influence of Non-Newtonian Behavior	
				of the Liquid	134
			2.3.2.4.2	Temperature Distribution in the Fluid	135
			2.3.2.4.3	Influence of Varving Temperature on	
				Viscosity	136
		2.3.2.5	Simple E	xample of a Scale-up	137
			1	1 1	
3	Mach	nine Tec	hnology		139
3.1	ZSK S	Series an	d Applicat	ions	139
	3.1.1	Develop	ment up t	o High Torques, Volumes, and Rotations	139
	3.1.2	Torque-	and Volui	ne-Limited Throughputs	143
	3.1.3	Exampl	es of Appl	ications for the Plastics Industry	145
		3.1.3.1	High Tor	que for Glass Fiber Reinforcement	
			of Plastic	- S	145
		3.1.3.2	High Tore	que for Film Extrusion of Non-Dried PET	
			or PLA.	-	147
		3.1.3.3	High Tor	que with Previously Volume-Limited	
			Applicati	ons	147
		3.1.3.4	Processir	g of Temperature- and Shear-Sensitive Products	149
			3.1.3.4.1	Compounding and Pelletizing of PVC-P and	
				PVC-U	149
			3.1.3.4.2	Compounding of Thermoplastic Elastomers	150
	3.1.4	Exampl	es of Appl	ications for the Chemical Industry	152
		3.1.4.1	Adhesive	and Sealing Materials	152
			3.1.4.1.1	Continuous Manufacture of	
				Adhesive Materials	152
			3.1.4.1.2	Continuous Manufacture of Sealing Materials	154
		3.1.4.2	Chemical	Reactions in Twin-Screw Extruders	155
			3.1.4.2.1	Manufacture of Thermoplastic	
				Polyurethane (TPU)	155
			3.1.4.2.2	Peroxidic Degradation of Polypropylenes	156

3.2	Barre	l Units	156
	3.2.1	Introduction	156
	3.2.2	Design Types	157
		3.2.2.1 Tie Rod Version for ZSK 18 - 54	157
		3.2.2.2 Flange Version for ZSK 58 - 320	158
		3.2.2.3 Clamp Version for ZSK 350 - 420	158
	3.2.3	Variants	159
		3.2.3.1 Closed Screw Barrel	159
		3.2.3.2 Closed Screw Barrel with Bore	159
		3.2.3.3 Open Screw Barrel	160
		3.2.3.4 Combination Screw Barrel	160
		3.2.3.5 Special Forms	160
	3.2.4	Wear/Corrosion Protection	161
		3.2.4.1 Solid Barrels: Nitrided or Through-Hardened	161
		3.2.4.2 Barrel with Liner (Oval Bushing)	161
		3.2.4.3 Directly Coated Screw Barrels	162
	3.2.5	Heating of Screw Barrels	162
		3.2.5.1 Heating Cartridges	162
		3.2.5.2 Heater Shells, Heater Plates	163
	3.2.6	Cooling and Tempering	163
		3.2.6.1 One Cycle	163
		3.2.6.2 Two Cycles	164
3.3	Increa	asing the Twin-Screw Extruder's Availability Using Targeted	
	Mater	ial Selection for Components that Come into Contact with	
	Produ	lct	164
	3.3.1	Introduction	164
	3.3.2	Wear Phenomena in Twin-Screw Extruders in Practice	165
		3.3.2.1 Abrasive Wear	166
		3.3.2.2 Adhesive Wear	169
		3.3.2.3 Corrosion	171
	3.3.3	Measurement and Assessment of Wear Parameters	173
		3.3.3.1 Measuring Resistance to Abrasive Wear	174
		3.3.3.2 Measurement of Adhesive Wear	175
		3.3.3.3 Measuring Corrosion	176
	3.3.4	Design Forms and Materials for Extruder Housings and	
		Screw Elements	177
		3.3.4.1 Housing Design Forms	177
		3.3.4.2 Screw Element Design Forms	178
		3.3.4.3 Material Design of Extruder Housings and Liners	181
		3.3.4.4 Material Design of Screw Set Elements	185
	3.3.5	Outlook	187

3.4	Dynai	mic Struc	ctural Analysis of Twin-Screw Extruders and	
	Single	e-Screw I	Discharge Extruders	188
	3.4.1	Structur	ral Model Description	188
	3.4.2	Vibratio	on Analysis on a ZSK	189
	3.4.3	Optimiz	ing Single-Shaft Extruders	195
	3.4.4	Structur	ral Vibration Engineering Design	198
	3.4.5	Summa	ry	203
3.5	Measu	urement	Technology and Process-Integrated Quality Assurance	204
	3.5.1	Metrolo	gical Basics	204
	3.5.2	Measur	ing Pressure and Temperature	205
		3.5.2.1	Temperature	205
		3.5.2.2	Pressure	207
	3.5.3	Rheolog	cical Measurement Technology	210
		3.5.3.1	Laboratory Rheometers	210
		3.5.3.2	Process Rheometers	211
	3.5.4	Color M	easurement	213
	3.5.5	Custom	ized Systems	213
		3.5.5.1	Ultrasonic Measurement Technology	214
		3.5.5.2	Model Predictive Control and Virtual Sensors	214
Л	Appli	ications	of Co. Potating Twin-Screw Extruders	217
-				217
4.1	Comp	ounding	In Practice	217
	4.1.1		Tommus Limitation	21/
		4.1.1.1	Volume Limitation	218
		4.1.1.2	Volume Limitation	218
		4.1.1.3	Further Limitations	218
	410	4.1.1.4 Dromivi		219
	4.1.2	Molt Do	llig	220
	4.1.3	Melt De	gassing	221
		4.1.3.1	Tachnical Design	221
	111	4.1.3.2 Strond I	Technical Design	222
	4.1.4	Drogogg	Control	224
	4.1.5	A 1 5 1	Drogoga Monitoring	220
		4.1.5.1	Caution Trank	220
	116	4.1.3.2		220
	4.1.0		Scraw Design	220 226
		+.1.0.1 // 1.6.2	Wear	220
	117	+.1.0.2 Scalo_11		227
	4.1./		The Ideal Case	227
		+.1./.1	Doality	∠∠/ 220
		+.1./.Z	Noamy	220

		4.1.7.3	Special Features of New Developments	229
		4.1.7.4	Conclusion	229
	4.1.8	Simulat	ion	229
4.2	Color	Masterb	atches	230
	4.2.1	Basic P	rocess Idea	231
	4.2.2	Materia	ls	232
		4.2.2.1	Pigments	233
			4.2.2.1.1 Color Index and Particle Sizes: Pigments at	
			First Glance	235
			4.2.2.1.2 Qualitative Description of the Dispersion	
			Quality in a Masterbatch	236
			4.2.2.1.3 Dispersion Properties of Organic Pigments	237
			4.2.2.1.4 Correlation between Dispersion Properties	
			and Process Parameters	238
		4.2.2.2	Complex Tasks	239
			4.2.2.2.1 Effect Pigments	239
			4.2.2.2.2 Organic Pigments with Different Dispersion	
			Characteristics	240
		4.2.2.3	Selection of the Polymer	240
		4.2.2.4	Additives and Dispersing Agents	241
	4.2.3	Mixing		242
		4.2.3.1	Gravity Mixer	242
		4.2.3.2	Low-Speed Stationary or Mobile (Container) Mixer	242
		4.2.3.3	High-Speed Stationary or Mobile (Container) Mixer	243
		4.2.3.4	Application Example: Production of Blends for	
			Masterbatch in the Hot Process for Staple Fiber and	0.40
	4.0.4	D :	Film Quality	243
	4.2.4	Dosing		244
	4.2.5	Extrude		244
		4.2.5.1	Premix	245
		4.2.5.2	Spiil-Feed	240
		4.2.3.3	Downstream Units	247
	126	4.2.3.4	Determination	240
	4.2.0		Color Mossurement	240
		4.2.0.1	Filter Prossure Test	249
		4.2.0.2	Agglomerates and Cel Particles	250
4.0	Duan -	T.Z.U.J	TDV by Dynamia Vulganigation on Ca Datation	201
4.3	Prepa	ration of	IPV by Dynamic Vuicanization on Co-Rotating	250
	1 WIII-	Classifi	aution of TDE	252
	4.3.1	Droporo	tion of TDV Based on EDDM/DD	252
	4.3.2	riepara		Z0Z

		4.3.2.1	Basic Raw Materials for TPV (EPDM/PP)	253
		4.3.2.2	Curing Agents	254
		4.3.2.3	Manufacturing Process for TPV (EPDM/PP)	254
		4.3.2.4	The Challenge of Dwell Time	256
		4.3.2.5	Properties of TPV (EPDM/PP)	258
	4.3.3	TPV Bas	sed on Renewable Raw Materials ("Bio-TPV")	258
		4.3.3.1	Basic Raw Materials for Bio-TPV	258
		4.3.3.2	Production Process for Bio-TPV	259
		4.3.3.3	Properties of Bio-TPV	260
4.4	Devol	atilizatio	n of Polymer Melts Using Co-Rotating	
	Twin-	Screw Ex	xtruders	263
	4.4.1	Devolati	ilization Tasks	263
	4.4.2	Design	of Devolatilizing Extruders	265
		4.4.2.1	Material Feeding and Flash Devolatilization	267
		4.4.2.2	Staggered Vacuums	269
		4.4.2.3	Fill Level	270
		4.4.2.4	Residual Devolatilization and Use of Stripping Agents	271
		4.4.2.5	Design of Extruder and Devolatilization Sections	276
	4.4.3	Scale-up	o of Devolatilization Extruders	279
	4.4.4	Process	Examples	281
		4.4.4.1	Devolatilization of Solvents from LLDPE Melt Solutions	281
		4.4.4.2	Devolatilizing Solvents from Synthetic Rubber	
			(Styrene-Butadiene Compounds)	282
		4.4.4.3	Devolatilizing Vinyl Acetate from LDPE/EVA Copolymer	282
		4.4.4.4	Devolatilization of POM	283
		4.4.4.5	Devolatilization of PC	284
		4.4.4.6	Devolatilization of PMMA	284
		4.4.4.7	Devolatilization of PES and PSU	285
		4.4.4.8	Devolatilization of ABS	287
		4.4.4.9	Devolatilization of Non-Dried PET	287
	4.4.5	Summa	ry	289
4.5	React	ive Extru	sion	290
	4.5.1	Introdu	ction	290
	4.5.2	Influence	ce of Parameters Using Selected Application Examples	291
		4.5.2.1	Activated Anionic Polymerization of Lactams	293
		4.5.2.2	Polymerization of Acrylates	294
		4.5.2.3	Ring-Opening Polymerization of $\epsilon$ -Caprolactone	296
	4.5.3	Econom	ically Relevant Example: Thermoplastic Polyurethane	297
	4.5.4	Modelin	ıg	299
	4.5.5	Scale-up	)	301
4.6	Food	Extrusio	1	304

	4.6.1	Extrusion of Breakfast Cereals 30		
		4.6.1.1 Raw Materials and Mixing	309	
		4.6.1.2 Preconditioning and Extrusion	313	
		4.6.1.3 Short-Time Tempering and Flaking	318	
		4.6.1.4 Roasting, Spraying, and Drying	321	
	4.6.2	Products	323	
	4.6.3	Food Safety in Food Extrusion	325	
	4.6.4	Summary	328	
	4.6.5	List of Abbreviations	329	
4.7	Extru	sion of Pharmaceutical Masses	331	
	4.7.1	Introduction	331	
	4.7.2	Fundamentals of Melt Extrusion	331	
	4.7.3	Machine Design	332	
	4.7.4	System Layout	334	
	4.7.5	Containment Requirements	339	
	4.7.6	Summary and Outlook	339	
Ind	<b>ex</b>		341	

# Functional Zones in the Extruder

## 1.1 Transport of Solids into and in the Extruder, Feed Limits

Reiner Rudolf, Carsten Conzen

During compounding, mainly solids, and in rare cases liquids or melts, are fed into the feed hopper located at the beginning of the twin-screw extruder. Furthermore, after the melting zone, additional solids can be fed into the extruder via side feeding units and liquids and gases can be injected via injection nozzles.

Twin screw extruders can be operated either starve fed or flood fed.

When run starve fed, less product is fed into the twin-screw extruder via the feed hopper than the screws can convey. Therefore, the feed zone is not completely filled with product.

Advantages include, for example, better recipe accuracy when dosing several components separately and higher flexibility due to the variable energy input caused by the variable throughput/speed ratio (filling degree) [1].

In flood fed operation, the extruder is operated with a fully filled feed hopper. This means that the screws convey as much product as their conveying capacity.

This section is limited to the description of the processes in the solids conveying zone at the beginning of the twin-screw extruder in starved feed operation, as co-rotating twin-screw extruders, unlike single-screw extruders, are generally operated in a starved feed condition.

As shown in Table 1.1, the solids to be dosed can be differentiated according to their particle shape and their melting behavior.

2

Particle shape	Melting behavior		
	Non-melting	Low melting *)	Melting
Pellet	Filler-masterbatches		Polymers
Powder	Filler, pigments	Waxes	Polymers
Flakes		Waxes	Polymers
Fiber	Glass, carbon		

 Table 1.1
 Classification of Solids Fed into the Twin-Screw Extruder and Typical Examples

\*) melting point lower, ca. 100 °C

Usually, solids with different particle shapes and melting behavior are fed simultaneously into the twin screw extruder, e.g. melting polymer granulates together with non-melting filler powder and low-melting wax.

#### 1.1.1 Characteristic Values and Calculation Possibilities

In order to determine the required speed range for a required throughput when operating in starved feed mode or to determine the maximum possible throughput at a given screw speed, it is necessary to know the solids conveying capacity. The intake capacity depends on the free cross-section between the screw elements and the barrel,  $A_{\text{free}}$ , the screw pitch *T*, the bulk density  $\rho_S$ , the conveying efficiency  $\varphi$ , and the screw speed *n*, and can be determined with Equation (1.1) [1].

$$M_F = A_{free} \cdot T \cdot \rho_S \cdot \varphi \cdot n \tag{1.1}$$

However, this approach only applies to simple bulk solids without feed limitation with linear throughput/screw speed behavior (cf. Figure 1.3).

In co-rotating twin-screw extruders with axially open screw channels, the conveying efficiency in Equation (1.1) depends on the friction conditions of the solid with the inner cylinder surface and the screw surface as well as the inner friction of the solid. To achieve the highest possible conveying efficiency, the circumferential force between the cylinder surface and the solid must be as high as possible and the circumferential force between the solid and the screw as low as possible. This can be illustrated using a rotating threaded rod (screw) with a threaded bushing (solid) freely movable on it. The threaded bushing rotates without forward movement on the threaded rod, since the maximum friction force in the thread (screwsolid) is much larger than the maximum air friction on the outer threaded bushing (solid-cylinder). If the bushing is fixed, the possible force for fixing the bushing (solid-cylinder) is considerably greater than the frictional force in the thread (solid-screw), and the bushing is moved forward at the maximum possible axial speed. Thus, at pure axial flow the maximum conveying capacity in a screw system is determined by the bulk density, the free cross-sectional area, the screw pitch, and the screw speed.

$$\dot{M}_F = A_{free} \cdot T \cdot \rho_S \cdot n \tag{1.2}$$

The conveying efficiency can now be determined at a given throughput, screw speed, and bulk density of the polymer as follows:

$$\varphi = \frac{\dot{M}}{A_{free} \cdot T \cdot \rho_{s} \cdot n} \tag{1.3}$$

and is between 0 and 1 [1].

In co-rotating twin-screw extruders, the deflection of the solid through the 8-shaped contour of the cylinder exerts a resistance and thus an increased circumferential force between the solid and the cylinder. This reduces the tendency for the solid to slip off the cylinder wall and results in an increased axial movement of the solid. Tests have shown that with the usual powder, fine grained, and granular granulate fillings the conveying efficiency varies only slightly with the given screw geometry. This can be traced back to the big influence of the suppression of rotation. As can be seen in Figure 1.1, the conveying efficiency decreases with increasing screw pitch. According to Equation (1.1), the optimum pitch in the feed zone is defined by the product of the intake pitch and the conveying efficiency. Therefore, Figure 1.1 also shows the effective intake pitch as the product of the intake pitch and the conveying efficiency [1].



**Figure 1.1** Conveying efficiency of co-rotating two- and three-lobed twin screw elements (effective intake pitch = conveying efficiency × intake pitch) [2]

6

At Covestro (formerly Bayer Technology Services), the solids conveying behavior was investigated experimentally in a co-rotating, tightly intermeshing twin-screw extruder with 58 mm screw diameter and plexiglass housings, with movies being taken from above, below, and from both sides. The selected screw had an initial pitch of 1.4 D with subsequent pitch reduction to 0.7 D. The investigated process conditions were simulated using DEM to determine the accuracy of the simulation. As can be seen from Figure 1.4 to Figure 1.7, experiment and simulation agree very well for all cases.

The different conveying angles of cylindrical and spherical granules are also reproduced very well, as Figure 1.8 shows.

Both experiment and simulation show that dosing fluctuations in the solids conveying zone cannot be compensated and, thus, have an effect at least up to the plasticizing zone (cf. Figure 1.9).

Due to the very good reproduction of reality, it is now possible to carry out screw design and process optimizations of the solids conveying zone of co-rotating twinscrew extruders for granular bulk materials by means of DEM simulation.



Figure 1.4 Comparison DEM simulation (top) with experiment (bottom) - top view



Figure 1.5 Comparison DEM simulation (top) with experiment (bottom) - view from below



Figure 1.6 Comparison DEM simulation (top) with experiment (bottom) - left screw





In the position of the screws shown in Figure 1.35 the part of the sub-stream 3 represented by the cross-sectional area  $A_{32}$  no longer has a free surface. The free surface  $S_{31}^{PB}$  of the cross-sectional area  $A_{31}$  is larger than that of the fully developed liquid pool of the sub-streams 1 and 2. This is due to the greater radial distance of the surface of the screw profile from the inner barrel surface at this point compared to the fully developed liquid pool at this filling level. The free surfaces  $S_{21}^{SA}$  and  $S_{21}^{GO}$  have further decreased compared to Figure 1.33 and Figure 1.34. In contrast to Figure 1.34 on shaft 2 in screw channel 1 on the inside of the barrel surface and on the liquid layer of the screw element the new free surfaces  $S_{12}^{GO}$  and  $S_{12}^{SA}$  have been created. The point  $P_{32}^{PB}$  is no longer part of the sub-stream 3 and is now positioned on the free surface  $S_{12}^{SA}$ .



**Figure 1.36** Liquid distribution in the cross-section of a twin-screw machine with double-flighted screw elements; degree of filling  $\epsilon = 80\%$ ; rotational angle  $\theta_{10} = \theta_p + \pi / i_g$ 

Figure 1.36 presents the liquid distribution in the position  $\theta_{10} = \theta_p + \pi / i_g$ . Compared to Figure 1.33, this corresponds to a rotation of  $\Delta \theta = \pi / i_g$ . The sub-stream 1 on shaft 1 hits the liquid layer in screw channel 1 on shaft 2 at point  $P_{12}^{SP}$ . This coincides phase-shifted with the liquid distribution shown in Figure 1.33. This process is repeated from one sub-stream to another depending on the number of threads  $i_g$  phase-shifted by  $\pi / i_g$ . The change from shaft to shaft takes place for each sub-stream by a rotation of the shafts of  $\Delta \theta = \pi (2 - 1 / i_g)$ . After a rotation of  $\Delta \theta = 2\pi (2 - 1 / i_g)$  the sub-stream is again on the starting shaft but in the other screw channel. After a rotation of  $\Delta \theta = 2\pi (2i_g - 1)$  the liquid pool is again on the same shaft in the same channel. With a double-flighted screw of  $i_g = 2$  this identical situation to the start position is reached after three turns.

The free surfaces represent phase interfaces. They are necessary for the diffusive mass transport from the polymer melt into the gas phase. Concentration boundary layers are developed on these surfaces. For both the free surfaces of the liquid pools and for the liquid layers on the screw elements and the barrel that are temporarily covered by the liquid pool, it is assumed that the concentration boundary layer is periodically mixed with the liquid pool. For complete mixing, the mean concentration of a liquid element reaching the phase interface is equal to the mean liquid concentration in the flow cross-section. Each of these periodically renewed elements thus fulfills the conditions of the model for the diffusive mass transport in polymers as described in Section 3.3 of *Co-Rotating Twin-Screw Extruders: Funda-mentals.* Size and residence time of the liquid elements at the phase boundary must be known to determine the change in the mean concentration of the diffusing species in the devolatilization zone.

Both the surface of the liquid layer on the screw elements and the free surfaces of the liquid pools are helical surfaces. In a general form, these surfaces are defined with the independent parameters  $\varphi, \psi$  by the local vector  $\vec{r}(\varphi, \psi)$  [1]:

$$\vec{r}(\varphi,\psi) = r(\varphi)\cos(\varphi+\psi)\vec{e}_x + r(\varphi)\sin(\varphi+\psi)\vec{e}_y + [h_g/(2\pi)]\psi\vec{e}_z$$
(1.24)

 $h_g$  indicates the thread height (pitch) of the screw. It corresponds to the axial length of a screw element for one winding ( $\psi = 2\pi$ ) along the screw surface. For  $r(\varphi)$ the functions for describing the shape of the liquid pool and the surface of the liquid layer on the screw elements have to be inserted. The used profiles are displayed in Figure 1.33 to Figure 1.36 The shape of the liquid layer on the screw surface results from the geometry of the screw elements, including the layer thickness. The shape of the free surface of the liquid pools is described with an empirical function that approximates the visually observed bead-like shape of the liquid pool.

With the partial derivatives

$$\vec{r}_{\varphi} = \partial \vec{r} / \partial \varphi \text{ and } \vec{r}_{\psi} = \partial \vec{r} / \partial \psi$$
 (1.25)

of the local vector  $\vec{r}(\varphi,\psi)$  follows for the surface element *dS* the relationship:

$$dS = \left| \vec{r}_{\varphi} \times \vec{r}_{\psi} \right| d\varphi d\psi \tag{1.26}$$

By numeric integration in the limits of  $\varphi$  and  $\psi$  one gets the surface *S*:

$$S = \iint \left| \vec{r}_{\varphi} \times \vec{r}_{\psi} \right| d\varphi d\psi \tag{1.27}$$

While the cross-sectional areas *A* for a screw profile are independent of the thread height  $h_g$  the surfaces *S* must be calculated as a function of  $h_g$ .

In order to obtain quantifiable information on the size of the phase interface, the relationship between filling degree and conveying behavior must be considered. Partial filling is always achieved with pressure less conveying if the condition for the volumetric flow number  $\dot{V}^*$ 

$$\dot{V}^* < \dot{V}_{max}^* \tag{1.28}$$

# Scale-up and Scale-down

## 2.1 Introduction and Basis Rules for Thermally Sensitive Products

Klemens Kohlgrüber

The scale-up of a process is an important step and will be found in several sections of this book. The focus is on the following question: how is a production machine to be designed so that the tests carried out with a smaller machine can be transferred to a production machine? The desired throughput with a necessary product quality is often in focus. The price of a large extruder usually correlates with the diameter of the machine. Therefore, the aim is usually that the production machine is as small as possible.

For the size of a machine, rough estimates with the following equation are often executed at the scale-up.

$$\frac{\dot{V}_Y}{\dot{V}_X} = \left(\frac{D_Y}{D_X}\right)^X \tag{2.1}$$

 $\dot{V}_X$  is the throughput of the test machine with diameter  $D_X$  and machine "Y" is the production machine. The throughput ratio  $\frac{\dot{V}_Y}{\dot{V}_X}$  is given based on the definition of the task. The diameter of the production machine  $D_Y$  at the given diameter of a test machine  $D_X$  depends strongly on the so-called scale-up exponent x: the smaller x is, the larger becomes  $D_Y$ . This is shown in a final example.

Empirical values are often given for the scale-up exponent *x*. For processes scaling predominantly with the product volume, it is close to 3; for surface-dominated processes (for example for the evaporation of volatile components) it will generally be lower. The ratio of area/volume is 1/D and gives an exponent 2. For heat transfer it can take the very unfavorable value 1.

In this book, Equation (2.1) is considered in several places, for example in Sections 2.2, 4.4, and 4.5.

Even without the consideration of specific processes, different exponents can be discussed if important basic principles are taken into account. In the following, some examples are given which include, amongst others, the dimensionless throughput, the geometric similarity, and the shear rate at the tip.

#### 2.1.1 Dissimilarity

It is clear that not all functional zones of an extruder produce the same scale-up exponent. Therefore, a scale-up usually involves compromises. A scale-up can also be performed "dissimilarly" to the test machine so that the desired throughput with the necessary product quality is achieved. For example, the opening cross-section of an extruder housing behaves in relation to the volume in the extruder as 1/diameter. In the case of powder dosing, a feed limit can easily occur on a production scale. On a production scale, the feed opening can then be chosen to be geometrically dissimilar compared to the test machine; for example, the feed opening may be relatively larger (L/D larger) or the dosing may take place via two openings. If the material has a very low bulk density, the feed limit on a production scale can be so severe that the economic use of an extruder is not possible. In this case, special large-volume, high-viscosity reactors may be an alternative. This example makes it clear that in many cases it makes sense first to determine a suitable production machine and then to consider a scale-down for a suitable test machine.

#### 2.1.2 Comparison of Production Machines

If the product development took place, for example, in a small type of device or machine, then quite different types of devices and machines could be considered for a large production machine. The "process requirement profile" must be compared with the "machine capability profile". Under the requirement profile, procedural aspects such as viscosity ranges, residence times, temperature control, mixing and dispersing, etc., need to be understood. In addition to the process aspect of feasibility on a production scale, many points play a role. Some points that should be considered when comparing production machines:

- Economic feasibility (includes many points; besides investment, also operating costs: personnel, energy, wear, availability, etc.)
- Process stability (degree of automation, complexity, constant product quality, etc.)

For the next step Potente and Preuß introduced formal relationships to compare the two machines and processes. Only the required relationships and ratios to explain the following method are presented here.

For the speed *v*:

$$v \sim Dn_0 \tag{2.49}$$

For channel width *b*:

$$b \sim D$$
 (2.50)

$$\frac{b_1}{b_0} = \left(\frac{D_1}{D_0}\right) \tag{2.51}$$

For the channel depth *h*:

$$h \sim D^{\psi}$$
 (2.52)

$$\frac{h_1}{h_0} = \left(\frac{D_1}{D_0}\right)^{\psi} \tag{2.53}$$

where  $\psi$  is the channel depth exponent.

For the screw speed

$$n_0 \sim D^{-\chi} \tag{2.54}$$

$$\frac{n_{0,1}}{n_{0,0}} = \left(\frac{D_1}{D_0}\right)^{-\chi}$$
(2.55)

where  $\chi$  is the screw speed exponent.

Since the material properties and the pitch angle are assumed to be constant, the throughputs of the machines can also be set in relation to each other:

$$\frac{\dot{M}_{1}}{\dot{M}_{0}} = \frac{\dot{V}_{1}}{\dot{V}_{0}} \frac{\rho_{1}}{\rho_{0}}$$

$$\Rightarrow \frac{\dot{M}_{1}}{\dot{M}_{0}} = \left(\frac{D_{1}}{D_{0}}\right)^{\psi} \left(\frac{D_{1}}{D_{0}}\right) \left(\frac{D_{1}}{D_{0}}\right) \left(\frac{D_{1}}{D_{0}}\right)^{-\chi}$$

$$\Leftrightarrow \frac{\dot{M}_{1}}{\dot{M}_{0}} = \left(\frac{D_{1}}{D_{0}}\right)^{2+\psi-\chi}$$
(2.56)

where  $\rho$  represents the melt density.

The channel depth exponent  $\psi$  in Equation (2.56) is fixed due to the fixed geometries of model and target machine. The speed exponent  $\chi$  must be determined based on the chosen boundary conditions. This will be discussed in Sections 2.2.3.2.2 and 2.2.3.2.5.

The consideration of the power inputs of both machines requires a consideration of the melt temperatures on both machines. Different solutions result for the individual conditions of the model and target machines in dependency on assuming the following:

- A constant or a variable melt temperature at the die outlet of both extruders
- A constant or variable pitch angle for both extruders

For example, based on the assumptions of a constant die outlet temperature of the melt and a constant pitch angle, the following solution results

$$\frac{P_1}{P_0} = \left(\frac{D_1}{D_0}\right)^{2+\psi-\chi}$$
(2.57)

For other assumptions regarding temperature and pitch angle, other solutions result, which are not discussed here. In this way, further model laws can be derived.

The model laws based on the assumptions of a constant melt temperature at the screw tip and a constant pitch angle are presented in Table 2.2.

Parameter		Model law
Throughput	$\frac{\dot{M}_1}{\dot{M}_0}$	$\left(\frac{D_1}{D_0}\right)^{2+\psi-\chi}$
Screw speed	$\frac{n_{0,1}}{n_{0,0}}$	$\left(\frac{D_1}{D_0}\right)^{-\chi}$
Power	$\frac{P_1}{P_0}$	$\left(\frac{D_1}{D_0}\right)^{2+\psi-\chi}$
Torque	$\frac{M_{D,1}}{M_{D,0}}$	$\left(\frac{D_1}{D_0}\right)^{2+\psi}$
Pressure	$\frac{p_1}{p_0}$	$\left(\frac{D_1}{D_0}\right)^0$
Temperature	$\frac{T_{1,1} - T_{0,1}}{T_{1,0} - T_{0,0}}$	$\left(\frac{D_1}{D_0}\right)^0$

**Table 2.2** Model Laws for the Case of Constant Mass Temperatures at the Screw Tip andConstant Pitch Angle

#### 3.1.4.1.2 Continuous Manufacture of Sealing Materials

Sealing compounds are commonly based on butyl rubber, polyisopropylene, polyurethane, or silicone rubber. Reactive sealing compounds can be crosslinked at room temperature (RTV) or at higher temperatures (HTV). Silicone sealing compounds are chemically hardening single-component systems in which the crosslinking of the substrate is initiated using moisture from the air. As a result, the reaction products are separated. After rapid skin formation on the surface, the crosslinking within the compound continues until hardening is complete. Areas of use include joint sealants in plumbing, construction, and the automobile industry.

Silicone sealing compounds such as those used, for example, in housing construction for sealing plumbing fixtures, are increasingly being manufactured in twinscrew extruders. Silicone polymer (30–50%), silicone oil (35–40%), crosslinking agents, and catalyst components are dosed into the extruder in fluid form using injection nozzles. In the process, the catalyst that reactively activates the mixture is worked in only at the end. As a rule, fillers such as silica (5–10%) create intake problems due to their very low fill density of approximately 50 g/l; they fluidize very easily and carry a great deal of air into the extruder which must then be removed again. A ZSK Mv's deeply cut flights enable significantly better powder intake and thus a throughput increase of over 35% as well compared to a ZSK Mc with an equal center distance. Discharge occurs during cartridge filling by means of a gear pump with a downstream heat exchanger and buffers directly into the cartridge filling unit. Additionally, masses can still be strained.

Typical throughputs for a ZSK 98 Mv sit at 2.6 t/h. Figure 3.15 shows one example of high dosing effort typical for this application.



Figure 3.15 Example of silicone sealing compounds manufacture

#### 3.1.4.2 Chemical Reactions in Twin-Screw Extruders

The reactive extrusion in ZSK-type twin-screw extruders is used for polymerization in the compound originating from monomers or pre-polymerizates, as well as for modification of polymers using grafting, crosslinking, and degradation. The demands upon a continuous reaction system vary according to the processing task.

#### 3.1.4.2.1 Manufacture of Thermoplastic Polyurethane (TPU)

The ZSK allows for continuous manufacture of an extremely wide product spectrum, from soft polyurethane adhesives to the hardest thermoplastic polyurethane elastomers with Shore hardnesses of D60. These linear, thermoplastic polyurethanes are used as construction materials of varying hardness, as highly elastic coating materials, and for fibers and films. The manufacturing process (see also Figure 3.16) requires stoichiometric dosing of components, polyol (usually premixed with a catalyst), and diisocyanates in the fluid or melted state into the ZSK's feed.

The TPU manufacturing process can be expanded to incorporate further additional materials such as stabilizers, lubricants, dyes, and flame retardants that can be added downstream from the main intake via the side feeder (ZS-B) into the melt flow as required.

Discharge takes place via a gear pump and a screen pack changer directly into underwater pelletizing.



Figure 3.16 Plant for continuous manufacture of linear thermoplastic polyurethane

layer structures. Therefore, it is important to minimize the mechanical stress and shear during the incorporation processes in such a way that particle assemblies are broken up and distributed, but the micro structure of the particles remains intact. Consequently, mixtures of organic pigments and effect pigments represent a special case for dispersion, because the optimum processing conditions of the two product groups can be diametrically opposed.

#### 4.2.2.1.1 Color Index and Particle Sizes: Pigments at First Glance

Organic pigments are assigned to a color index (C.I.) based on their chemical structure. The color index bears the color theme and a consecutive number. Pigments with the same C.I. can yet have different crystal morphologies and particle size distributions ( $\rightarrow$  electron micrographs of C.I. Pigment Red 202 and C.I. Pigment Purple 19: Figure 4.11, Figure 4.12). Therefore, the C.I. alone does not give conclusive information about the dispersion behavior of a pigment. Other pigment properties such as opacity, transparency, or hue can also be considerably different with the same C.I.



Figure 4.11 Different types of C.I. Pigment Red 202 [BASF AG]



Figure 4.12 Different types of C.I. Pigment Purple 19 [BASF AG]

Commercially available powder pigments consist of a mixture of primary crystals, aggregates, and agglomerates. Primary crystals are individual crystals that are clearly described by their crystal structure and morphology. Primary crystals that have grown together at the corners and across common surface areas are called aggregates. They are firmly coalesced and can hardly be separated mechanically. Agglomerates are combinations of primary crystals and aggregates. They constitute the major part of powder pigments and, in the dispersion process, need to be broken down into primary crystals and aggregates as completely as possible.

#### 4.2.2.1.2 Qualitative Description of the Dispersion Quality in a Masterbatch

When is an organic pigment well dispersed? This is generally the case when the color properties and fastnesses specified by the manufacturer have been achieved. The properties are tested in specified pigment concentrations in accordance with industrial guidelines and standards. For this purpose, the masterbatch has to be incorporated into defined polymers for testing. Especially hue, purity, tinting strength, as well as opacity or transparency, if required also heat resistance, light and migration fastness, should be checked by the colorist or the application engineer and fall within the scope of the manufacturer's specifications. Furthermore, no implications of poor dispersion should be visible at all when the masterbatch is compounded for testing at final concentration, such as:

- Streaks, specks, dots
- Low brilliance
- Hue shift
- Changed transparency/opacity
- Impairment of mechanical properties, failure in thin layers such as film tears or fiber breakage

Figure 4.13 below shows a typical particle size distribution of an organic pigment after dispersion. Maximum tinting strength and purity are achieved with a preferably large amount of particles around 0.1  $\mu$ m. Particles in the range of 0.2 to 0.5  $\mu$ m contribute to the opacity of the pigment. Fine particles that would impair the migration fastness are rarely generated by the dispersion process. Microscopically visible particles > 10  $\mu$ m mainly cause the above-mentioned implications of poor dispersion. However, there is no direct correlation between the frequency of microscopically visible particles and the color strength of the masterbatch. This important fact will be taken up again in the considerations below.



**Figure 4.56** Left: natural wheat starch with visible starch granules and protein particles; right: extruded, cooked wheat starch [Bühler AG]

The many reasons behind the success of extrusion cooking are described in more detail in [4]. Here, we give a brief summary of the main ones:

- 1. Combination of the different basic process technology operations
- 2. Process and product flexibility
- 3. Low production and investment costs
- 4. Energy efficiency and sustainability

Cold extrusion for the production of pasta products, for instance, is based on the same physical principles and basic process technology operations as extrusion cooking. Semolina (200 to 500  $\mu$ m) is mixed with water (30 to 33%), kneaded, and formed at a die pressure of approximately 110 bar. This process exposes the dough to mechanical and thermal energy, which, unlike extrusion cooking, causes little protein and starch damage. For this reason, the dough temperatures remain below 50 °C. The aim of cold extrusion is to produce dough with a continuous protein matrix which entraps the starch granules. In extrusion cooking, the process is the exact opposite and it is the starch that serves as a binder, rather than the protein.



#### Figure 4.57

Left: cold extrusion – continuous protein matrix (green) with embedded starch granules (purple) Right: hot extrusion – continuous starch matrix (purple) with embedded protein bodies (green) [Bühler AG]

#### 4.6.1 Extrusion of Breakfast Cereals

Preferences regarding breakfast tend to differ depending on the culture. However, breakfast cereals play a significant part. Studies show that annual consumption in Germany amounts to 1 kg per person per year. Cereals are eaten daily in 15% of households, once a week in 33% of households, and at least once a month in 10% of households. Interestingly, 50% of breakfast products made for children are consumed by adults [5].

Based on the example of a modern plant for extruded breakfast cereals, the following sections describe the process steps required to produce end products from the raw materials.

This type of extruder comprises the following process steps:

- 1. Preparation of raw materials and mixing
- 2. Preconditioning and extrusion
- 3. Tempering and flaking
- 4. Drying and spraying
- 5. End drying and roasting

The process flow chart in Figure 4.58 illustrates this type of system.

## Index

#### Α

abrasive wear 166 additives 232, 254 additives and dispersing agents 241 adhesive and sealing materials 152 adhesive wear 169 adiabatic operation 92 agglomerates 231 agglomerates and gel particles 251 application examples for the plastics industry 145 applications of co-rotating twin-screw extruders 217 Arrhenius approach 95 average concentration for a twodimensional extruder model 83 average shear rate 108 axially open 221

#### В

back venting 280 backward-feeding kneading or conveying elements 298 barrels 156 barrel with liner 161 barrier screw 232 base material 231 BASF 235 basic machine geometry 109 basic materials for screw elements – overview 185 basis rules for scale-up/down 99, 104 batch mixer 310 batch-oriented industry 339 batch process 331 batchwise mixing 242 Bayer 105 bioavailability 339 bio-TPV 258 breakfast cereals 304 Brinkmann number 136 bubble-free liquids, degassing 71 Bühler 308 bulk materials 4

#### С

calculating the melting behavior 18 calendering effect 237 calender rolls 238 catalysts 152 ceramic compounds 152 channel cross sections with different pitch/diameter ratios 111 channel depth exponent 112 channel depths exponent 119 characteristic numbers of the whole machine 127 chemical/food/pharma applications 152 CIELAB system 249 coated screw barrels 162 coating materials - overview 185 coefficient of variation 26, 36 co-extrusion 334

cold agglomeration 231 cold mixing 242 color index 235 coloristics 239 color masterbatches 230 color measurement 213, 249 color representation in the coordinate system 249 columns 106 compaction tendency of pigments 238 comparison of production machines 88 complete similarity 106 compound elements 180 compounding 217 compounding routine 220 concentration 36 container mixer 242 contamination 230 conveying element 28 conveyor dryer 323 cooling and tempering 163 co-polymer 252 corrosion 171 - measurement 176 cost-effectiveness 259 cracking - risk of 182 cross mixing 24 cumulative frequency distribution of maximum shear load 46 curing agent 253

#### D

damping factors 194 Danckwerts 83 degradation 257 degradation reactions 291 degree of disaggregation 30 degree of fill 131 degree of filling 55, 293 degree of segregation 37 DEM 6 design of the devolatilization zone 76 devolatilization - inserts 278 - in the compounding step 265 - of ABS 287 - of non-dried PET 287 - of PC 284 - of PES and PSU 285 - of PMMA 284 - of polymer melts 263 - of POM 283 - of reaction products 264 devolatilization of polymer melts 49 devolatilization times 62 devolatilizing - solvents from synthetic rubber 282 - vinyl acetate from LDPE/EVA 282 devolatilizing extruders - design 265 die head 316 diffusive upwind method 80 diffusivity 78 dimensionless groups for devolatilization 70 diols and diisocyanates 298 direct-coated housings 178 direct expanding breakfast cereals 323 disperse melting 17 dispersing in the melt 96 dispersion 29 dispersion behavior 45, 47 dispersion process of pigments 233 dispersion properties and process parameters 238 dispersion properties of organic pigments 237 dispersion quality 30 dispersion quality in a masterbatch 236 dispersive mixing 29 dissimilarity 109 dissimilarity in scaling 88 dissimilar scaling 104 distributive mixing 23 dough formation by raw materials containing starch 309 droplet dispersion 33

dross formation 229 dust formation 309 dwell time 256, *See also* residence time dynamic structural analysis 188 dynamic vulcanization 252

#### E

effect pigments 234 energy balance in the extruder 109 engine supports 192 enthalpy diagram 93 ESA series 195 evacuating dome 222 evaporation energy 268 exit temperature of the melting zone 21 experimentally based modeling 90 extensional flow 24 extrudates from the food industry 304 extruder housings and screw elements - materials 177 extruder speed and torgue 129 extrusion - of breakfast cereals 307 - of meat substitute products 329 - of pharmaceutical masses 331 extrusion cooking 304 extrusion die 316 extrusion nozzle 295

#### F

feed characteristic 25 feed limit 88 feed limitations 8 feed, starve or flood 1 FEM 80 fiber reinforcement of polymers 145 fill level 270 film extrusion 147 filter pressure test 250 filter pressure value 250 finite element method 80 finite volume method 80 first-order reactions 301 flakes 11 flaking 318 flaking roller mill 319 flash devolatilization 267 flash valve 269 floor coverings 152 Flory-Huggins equation 272 flow, creeping 24 flow patterns in laminar flow 24 fluid bed dryer 319 fluidization 10 food extrusion 304 food safety in food extrusion 325 forward vent ports 276 Fourier number 136 free liquid surfaces 69 frequency inverter 226 frequency responses of vibration displacements 201 friction power in the product 98 functionalization and grafting reactions 290 functional zones 88 functional zones in the extruder 1 fundament excitation 192 FVM 80

#### G

geometrically dissimilar 88 geometrically similar machines 128 Good Engineering Practice 338 graft polymer 252 granular bulk materials 6 granulation in the pharmaceutical sense 331 gravimetric dosing 313 gravity mixer 242

#### Н

haptics 261 Hazard Analysis and Critical Control Points (HACCP) 326 heater plates 163 heater shells 163 heat flows via the barrel 122 heating cartridges 162 heating of screw barrels 162 highly active substances 339 high rotations 139 high torques 139 high volumes 139 HIP 181 homogenization and crosslinking 255 hot and contact adhesives 152 hot mixing 242

#### I

infrared thermometers 207 inhomogeneities 221 initial melting 17 injection molding 334 inner friction 2 inorganic pigments 233 investment costs 220

#### Κ

kneading block 28 - principle of operation 28

#### L

laboratory extruder 15, 92 laboratory rheometers 210 Leistritz 231, 333 length of the devolatilization zone 78 lengths exponent 118 limitations in compounding 218 liner housings 178 liquid distribution and degree of filling 49 LLDPE 281 loading cycle 30

#### Μ

machine configuration 109 machine design for pharmaceutical applications 332 masterbatch in the hot process for staple fiber and film guality 243 material 164 material data 229 material properties 109 material types for carrier housings - most common 183 measurement technology 204 melt degassing 221 melt-fed 276 melting - essential steps 16 melting of thermoplastics 12 melt pump 250 melt temperature 113 minimum load 30 mixing 23, 242 mixing and dispersion 23 mixing, dispersive 29 mixing, homogeneous 23 mixing processes during color change 40 mixing quality 36 mixing time and blending quality 312 modal hammer 191 model-based experiments 90 model-based scaling approach 108 model exponents 118 modeling 299 model law exponents 114 model laws for the case of constant mass temperatures 113, 114 model predictive control 214 modular construction 289 molecular weight distribution 292 molecular weight reduction 297 mono batches 230 monolithic elements 179 monolithic housing 177 multi batches 233

#### Ν

natural frequencies 192 NIR and UV-VIS spectroscopy 337 nitriding layer 182 non-Newtonian behavior, influence 134 numerical diffusion 80 numerical simulation of film degassing 79

#### 0

odor improvement 263 one-dimensional modeling 299 opacity 236 open screw barrel 160 operating costs 293 optimum pitch in the feed zone 3 organic pigments 233 outer screw diameter as reference value 55

#### Ρ

partial flow 27 partial flows 24 partial flow sections in a twin-screw extruder 28 partially filled zones 130 partial pressure reduction 274 particle size distribution 237, 311 particle tracking 42 part models 90 PAT (Process Analytical Technology) 337 pellets 230 peroxidic degradation 156 PET 147 pharmaceutical compounds 152 phenolic resins 254 physically different scaling 104 PIGS diagram, Presence, Introduction, Growth, Survival 327 pitch exponent 120 pitch t 111

#### PLA 147

plastic deformation 14 PM-HIP composite materials 186 PMMA 264 polished stainless steel surfaces 332 polymer base material 240 polymer feed concentration 266 polymerization - of acrylates 294 - of caprolactone 296 - of lactams 293 polyolefins 237 powder 9 powder pigments 236 preconditioning and extrusion 313 premix 231, 244, 245, 334 pressure and temperature measurement 205 pressure and temperature progression 166 pressure measurement 207 pressure transducers 209 process conditions, as similar as possible 106 process control 225 process control concept 328 process-integrated quality assurance 204 process parameters 248 process rheometers 211 product characteristics and dosage form 331 product cooling via the housing wall 91 production scale, task 89 product quality 88, 219 product temperature increase through residence time and shear 97 product temperature in scaling 90 PVC 149, 237

#### ۵

quality determination 248

#### R

Raman spectroscopy 337 reactive extrusion 290 rear vent 267 related degassing angle 65 residence time 97, 131, 221, *See also* dwell time residence time behavior 44 residual contents 281 residual devolatilization 264 residual devolatilization and use of stripping agents 271 resistance thermometers 207 reversion 257 rheological measurement 210 roasting 321

#### S

sample 36 sampling 225 SBR 264 scale-down 87,88 scale-down and ways of design 89 scale-up 87, 227, 301 - devolatilization extruders 279 - reactive extrusion process 302 scale-up and scale-down by model laws 106 scale-up and scale-down with characteristic numbers 126 scale-up exponent 280 scale-up exponent x 87 scale-up/scale-down exponent 107 scale-up, simple example 137 scale-up throughput and screw diameter 103 scaling with different geometries 130 Schenkel 105 screen inserts 247 screw channel flight depth - higher 141 screw configuration - sharp 259

screw configuration for ring-opening polymerization 294 screw elements and screw configuration 13 screw shaft seal 276 screw speed exponent 112, 119 sealing materials 154 semi-crystalline polymers 93 sensors 204 set-up times 247 shear flow 24 shear flow and extensional flow in extruders 34 shear gradient 298 shear load 45 shear rate 34 shear rate in the clearances tip/wall and screw/screw 99 shear rate, relevance 97 side devolatilization 278 side feeder 256 SIGMA 105, 108 silicone rubber 152 simple scaling approach 107 simulation 229 simulation of solids transport 5 single-screw extruder 250 single-shaft machine series 195 smoothed particle hydrodynamics 47. 80 solid particle in a shear flow 30 species transport equation 41 specific drive power 93 specific energy input 93, 128 specific energy required for processing 150 specific heat capacity 93, 94 specific mechanical energy input 317 specks 236 speed and throughput for scale transfer 133 SPH 47,80 split-feed 231, 246 spray ceramic 152 staggered vacuums 269

standard deviation 310 stirred tank reactor 106 strand die head 224, 247 strand pelletizing 336 stress-time factor 30 stripping agent 271 - addition 275 structural model description 188 structural vibration engineering design 198 stuffer 222 superposition of drag and pressure flow 110 surface expansion by bubbles 76 surface renewal 49 surface renewal model 84 surfaces of the liquid pools 54 surface-to-volume ratio 123 synthesis of homopolymers and copolymers 290 system layout for pharmaceutical applications 334

#### Т

temperature- and shear-sensitive products 149 temperature distribution in the fluid 135 temperature during devolatilization 269 temperature increase and internal friction 96 temperature in reactive extrusion 257 temperatures and speeds in pharmaceutical applications 334 temperature sensitivity factor 95 tests, aim 90 thermally sensitive products 92 thermal product degradation 94 thermocouples 206 thermoplastic compounding 217 thermoplastic elastomers 150 thermoplastic polyurethane 297 thermoplastic starches 152 thread height 54, 57

three-dimensional calculations of mixing and residence time behavior 40 throughput for the target machine 118 throughput limitation 217 throughput, proportional to diameter to the power x 87 tilting eigen modes 200 tip plating and basic materials 186 tip surface-layer welded elements 179 torque- and volume-limited throughputs 143 torque displayed in the extruder controller 226 torque-limited and volume-limited processes 217 TPA 252 TPC 252 TPE 151, 252 TPS 252 TPU 155, 252 TPV 252 - manufacturing process 254 TPV based on EPDM/PP 252 tracer particles 46 transparency 236 transport of solids 1

#### U

ultrasonic measurement technology 214 underwater pelletizing 247 universal mixer 220 upstream mixing process 221

#### V

vacuum 221 vacuum system 263 variations of the tip clearances 103 vibration analysis 189 vibration velocities 196 volume flow rate 78 volume-limited applications 147 volumetric flow number 54 vulcameter test curves 257 vulcanization accelerator 260

#### W

warm mixing 242 wear 165, 227 - measurement and assessment 173 wear conditions - different 228 wear/corrosion protection 161 Weber number 31 Weber number for breaking up droplets 32

#### Υ

yellowing of material 219

#### Ζ

zipper formation in pelletizing 219 ZSK - generations 139

- MEGAcompounder 142

- series and sizes 141

ZSK series and applications 139