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Research paper

Comparison between a twin-screw extruder and a rotary ring die press. Part II: influence of process variables

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Abstract

The influence of different processing steps on pellet quality was investigated: granulation/extrusion and spheronization. Pellets were produced at two levels of water content on two different types of extruders: a twin screw extruder and a rotary ring die press. In order to control the spheronization process each extrudate was rounded in two spheronizers using two radial velocities, respectively. Pellet shape and size were selected to describe the pellet quality. Under constant spheronization conditions the extrudates behaved dissimilar on the two spheronizers. This could be attributed to the geometry of the friction plates. The spheronizer with the rougher surface applied more mechanical energy to the extrudate and wet pellets which reduced the water content necessary for the formation of good pellets. Eliminating the influence of the spheronization process, high differences were observed in the quality of the extrudates produced by the two extruders. This confirmed the results from the first part of this study. Due to the crystallite-gel-model the different extruder types apply different mechanical stress on the extrudate which affect the network structure of the microcrystalline cellulose gel. The twin-screw extruder produced a more delicate network with a lower water movement. This led to a shift of the optimal moisture content towards higher values. Compared with spheronization, process changes in the granulation/extrusion process were more critical. © 1998 Elsevier Science B.V.

Keywords: Extrusion/spheronization; Twin-screw extruder; Ring die press; Friction plate geometry; Spheronizer speed; Crystallite-gel-model

1. Introduction

In the first part of the study the influence of formulation variables on two types of extruders-a twin-screw extruder and a rotary ring die press-was investigated [1]. Excipients with different water solubility and particle size were processed with a varying moisture content. Their effect on pellet shape, pellet size and power consumption was observed.

For every formulation on both extruder types an optimal moisture content could be determined at which pellets with an optimum aspect ratio (length/width-ratio, AR) were prepared. New characteristic parameters were defined that were derived from the optimal moisture content concerning the corresponding equivalent diameter (D_{eq}) of the pellets and the power consumption of the extruders. The trials revealed fundamental differences in the required moisture content between the two extruder types. These differences were essentially explained by the crystallite-gel-model [2]. But the influence of the spheronizer types and the spheronizing conditions that were not kept constant during the experiments was uncertain.

In the present second part of the paper a trial is described that investigates additionally the influence of the spheronizing conditions: spheronizer type and radial velocity of the

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friction plate. The experimental design with a close control of the spheronization process allows an unbiased comparison between the two extruder types.

2. Materials and methods

2.1. Materials

Pellets were prepared from a binary mixture of 70% lactose-monohydrate (Capsulac 60, Meggle, Wasserburg, Germany) (% w/w) and 30% microcrystalline cellulose (Avicel PH 101, FMC, Cork, Ireland) which were granulated with demineralized water. All materials were used as received.

2.2. Equipment

2.2.1. Extruder

Extrusion was performed on two extruders: a twin-screw extruder (Berstorff ZE 25 × 18 D, Berstorff, Hannover, Germany) and a rotary ring die press (PP-127, Schlüter Maschinenfabrik KG, Neustadt a. Rbge.). They have been used as described in the first part of the paper with one exception. A power-consumption-control-circuit was established for the twin-screw extruder as described previously [3,4]. Extruder and liquid dosage pump were connected to a data acquisition system. The total power consumption was recorded and compared with a preadjusted value. If the actual power consumption exceeded the pre-set value, the pump rate was increased and vice versa until steady state extrusion conditions were achieved.

2.2.2. Spheronizer

Spheronization was performed on two spheronizers: a Nica S320 (Nica AG, Mölndal, Sweden) with a friction plate diameter of 320 mm and a Schlüter RM-300 (Schlüter Maschinenfabrik KG, Neustadt a. Rbge., Germany) with a diameter of 300 mm.

2.3. Methods

2.3.1. Extrusion

For pellets produced on the twin-screw extruder 10 kg of dry powders were blended in a turbula blender (Type T 10 B, Bachofen KG, Basel, Switzerland) for 15 min and poured directly into the powder feeder. Powder feed rate was adjusted to 25 g min^{-1} and screws were rotating at a speed of 60 min^{-1}. The addition of granulation liquid by the water pump depended on the power-consumption-control. After power consumption constantly reached the pre-set levels, 2200 g extrudate per batch was collected.

For pellets produced on the ring die press for each batch 2750 g of dry powders were blended in a planetary mixer (A200, Hobart, Offenburg, Germany) for 5 min on level 1 (96 min^{-1}). Different amounts of granulation liquid were added within 20–40 s and the powders were granulated for further 5 min on level 2 (176 min^{-1}). The granule was filled in the feeder and extrusion was started immediately. The ring die press was set to standard conditions (rotational speed: 250 min^{-1}, roll gap width: 0.33 mm). Net power consumption was determined as mentioned in the first part of the paper. Steady state conditions of the process were monitored by the recorded power consumption. After reaching an equilibrium 2200 g of extrudate was collected.

Total water content of the granules was determined for three samples per batch. It was calculated by the loss of drying at 105°C for 24 h on dry base (m/m).

2.3.2. Spheronization and drying

Extrudate, 500 g, per batch was spheronized on both spheronizers for 5 min at two levels of radial velocity, respectively: 13.4 or 10.1 ms^{-1}. With respect to the different friction plate diameters spheronization was performed on the Nica S320 at 800 and 600 min^{-1} or on the Schlüter RM-300 at 853 and 640 min^{-1}, respectively. The wet pellets were dried in two fluid-bed dryers of the same type (TR2, Glatt AG, Binzen, Germany) for 20 min at 50°C inlet temperature.

2.3.3. Characterization of pellets

Pellets were characterized by an image analysis system Leco 2001 (Leco Instruments, St. Joseph, Germany) which has been described previously [5]. The number of analyzed particles depended on their size. For normal sized batches 350–500, in the case of highly agglomerated batches 95–200 particles, were measured. The following parameters were determined: length L (longest of eight measured feret diameters per particle), breadth B (feret diameter at right angles to length) and projected area A. Aspect ratio AR and the equivalent diameter D_{eq} were calculated according to Eq. (1) and Eq. (2).

$$D_{eq} = \sqrt{\frac{4 \times A}{\pi}} \tag{1}$$

$$AR = \frac{L}{B} \tag{2}$$

Pellet quality was mainly rated by aspect ratio related to pellet shape and D_{eq} related to pellet size. Good pellets should have an AR below 1.1 and should not show any agglomeration indicated by a mean D_{eq} below 1400 μm.

SEM-photographs were taken to get an impression of the morphology of the pellets. The pellets were treated with water in order to dissolve lactose. Water, 100 ml, were added to about 200 mg of pellets. The suspension was shaken for 24 h and subsequently washed at least five times with water. The eluted, wet pellets were frozen at −18°C and freeze-dried (Lyovac GT 3, Leybold-Heraeus, D-Köln).

The dried pellets were coated with a gold layer using a sputter (SCD 005, Bal-Tec AG, Fürstentum-Liechtenstein) with 50 mA for 180 s. Subsequently, SEM-photographs were taken with a scanning electron microscope (XL 20, Philips, NL-Eindhoven).

2.3.4. Experimental design

Pellets were produced on both extruders in a good and poor quality using conditions that were derived from earlier experiments. Moreover, the two extrudates should possess similar moisture contents on both extruders. Therefore on the twin-screw extruder the rate values for power consumption were set to 180 and 300 W. On the ring die press the added amount of granulation liquid was calculated to obtain moisture contents of 40 and 55%. The four extrudate batches were divided into four parts of 500 g in order to be spheronized on both spheronizers at two levels of radial velocity.

3. Results and discussion

3.1. Extrudate production

Table 1 shows characteristic parameters for the extrusion process. Comparing the batches with the same quality net power consumption was approximately the same for both types of extruders. This has to be attributed to chance because of the high scatter in power consumption measurement for the ring die press that was already mentioned [1]. On the twin-screw extruder temperature and pressure were higher for the batch with lower moisture content due to the missing lubrication effect of the water. On the ring die press recorded temperatures were of no significance.

Looking at the results of moisture content (Table 1) and image analysis (Table 2) two observations must be noted. At first on the ring die press the batch with 55.9% moisture content led to a distinct agglomeration on both spheronizers and at both radial velocities. The extrudate was too wet for a successful spheronization. These batches were rejected. However, the extrudate from the twin-screw extruder with 59.9% moisture content could be spheronized into pellets.

On the twin-screw extruder the required moisture content for a good pellet production was shifted to higher values. This was predicted from earlier experiments [1] and explained by the different mechanical stress that was applied to the extrudates during granulation and extrusion.

3.2. Pellet quality: influence of the spheronization process

Figs. 1 and 2 and Table 2 show the results for AR and D_{eq} for all analyzed batches. The two extrudate batches with lower moisture content resulted in similar changes in pellet quality in dependence on the spheronizing conditions. Increasing radial velocity decreased AR and D_{eq} as well as their corresponding standard deviations. Values for the AR were in general lower for the Schlüter RM-300 compared with the Nica S320. The differences in size were negligible.

A different behaviour could be observed for the extrudate with 59.9% moisture content. Increasing radial velocity was of minor influence on AR but increased significantly the equivalent diameter ($P < 0.001$ for both spheronizers). Values for the D_{eq} and its standard deviation were generally higher for the Schlüter RM-300 compared with the Nica S320 due to a beginning uncontrolled agglomeration. But even on the S320 a slight agglomeration occurred at the higher radial velocity. AR values were lower for pellets spheronized on the S320 and at lower radial velocity.

The influence of the spheronizer speed is already well known [6–15]. Higher speed values result in pellets with a more spherical shape [7–10,12,14] and higher bulk or tapped density [8,10] or more agglomerated pellets, respectively in dependence of the extrudate properties. For a certain extrudate the shape depends on the radial velocity of the friction plate independent of the spheronizer diameter [12]. Spheronizer load [13–15] and residence time [6–10, 12,14,15] are known as critical parameters, too. Therefore, the levels for radial velocity were the same for the two spheronizers as well as the load and the residence time. Nevertheless differences in pellet quality prepared from the same extrudate under constant spheronizing conditions could be observed. These differences can be explained by the different geometry of the spheronizer friction plates as the most important component [6]. Fig. 3 shows schematical drawings. In both cases the surface textures are cross-hatch patterns and the grooves intersect each other in 90° angles. Looking at the cross-section the friction plates differ in the dimension of the truncated pyramids. These truncated pyramids have a larger base (12.25 mm^2) and a lower height (0.80 mm) on the Nica S320 compared with the Schlüter

Table 1

Data for extrusion process

	Moisture content (%) (w/w)	Power consumption (W)	SD	Net power consumption (W)	Intended pellet quality	Temperature (°C)	SD (°C)	Pressure (MPa)	SD (MPa)
Ring die press 40%	40.1	1477.0	38.6	235	Low	37.6	–	–	–
Ring die press 55%	55.9	1361.0	36.8	119	High	39.5	–	–	–
Twin-screw extruder 300 W	44.5	302.7	7.4	238	Low	40.6	1.1	0.180	0.013
Twin-screw extruder 180 W	59.9	182.6	6.0	118	High	31.8	1.0	0.067	0.006

RM-300 (base, 9.0 mm^2; height, 1.50 mm). This leads to a smoother surface in case of the S320 or a rougher surface of RM-300 friction plate, respectively. Thus, the transfer of mechanical energy into kinetic energy is more effective on the RM-300. Therefore extrudates with a lower moisture content and plasticity like the 40.1 and 44.5% batches are better spheronized on the RM-300. In contrast extrudates with higher moisture content (59.9% batch) tend more towards agglomeration compared with the Nica S320. It can be stated that geometry of the spheronizer friction plate influences the pellet properties regardless of constant spheronizing conditions.

3.3. Pellet quality: influence of the granulation/extrusion process

Apart from differences due to the spheronizer type and velocity the extrusion process has a high impact on pellet quality. A first remarkable hint for an influence of the extruder type is the fact that the extrudate with 55.9% moisture content from the ring die press plainly agglomerated under all spheronizing conditions. This must be related to an overwetted extrudate. In contrast the extrudate with 59.9% moisture content from the twin-screw extruder was sufficiently spheronized under certain conditions. For a more detailed consideration the results for different extrudate batches under constant spheronizing condition must be compared (Figs. 1 and 2). Changes in D_{eq} values were negligible for increasing the moisture content from 40.1 to 44.5% and must be related to the spheronizing conditions for a further increase up to 59.9%. Pellet shape was more clearly influenced by the extruder type. Increasing the moisture content should have led to pellets with initially decreasing AR values because of the increased liquid/solid ratio and plasticity of the extrudate [1]. But in contrast batches with 44.5% moisture content resulted in pellets with higher AR values than batches with 40.1% regardless of the spheronizing conditions. A further increase of moisture content led to a reduction of the AR values or to an overwetting, respectively.

These results can only be explained by the hypothesis that extrudates of different quality were produced in the two granulation/extrusion processes. Due to the crystallite-gel-model particles of microcrystalline cellulose are transferred into smaller units, ultimately single crystallites, during the granulation/extrusion process and a gel is formed [2]. With decreasing size of the elements building the framework more granulation liquid can be immobilized. The movement of granulation liquid in the wetted powder mass is reduced more effectively. Applying more shear in this process results in a more delicate network of the gel: it can be expected that in this case the optimal moisture content is shifted towards higher values.

The extrudate with 59.9% moisture content of the twin screw extruder could be processed into pellets while the 55.9% extrudate of the ring die press was apparently overwetted under constant spheronizing conditions (Nica S320). Due to this the twin-screw extruder produces a more finely divided crystallite-gel compared with the ring die press. Consequently the extrudate with 44.5% from the twin-screw extruder was underwetted while the 40.1% extrudate from the ring die press could be processed into round pellets under constant spheronizing conditions (Schlüter RM-300). Small differences in the moisture content of extrudates prepared by the same process are related to significant changes in pellet properties [1]. Thus, it is remarkable that the difference in optimal moisture content of the extrudates prepared by different granulation/extrusion processes is quite high.

3.4. Pellet quality: influence of the whole process

From the previous sections it became clear that pellet quality is influenced by both the granulation/extrusion process as well as the spheronization process. This can be discussed together using the relation between AR and D_{eq}

Table 2

Results for image analyze and moisture content determination

Batch no.	Extruder	Radial velocity (m s^{-1})	Spheronizer	AR	SD	D_{eq} (μm)	SD	No. of analyzed particles
1	Ring die press 40%	10.1	Nica S320	1.32	0.21	1340	129	425
2			Schlüter RM-300	1.10	0.06	1330	92	419
3		13.4	Nica S320	1.16	0.12	1311	120	438
4			Schlüter RM-300	1.08	0.05	1313	93	414
–	Ring die press 55%	10.1/13.4	S320/RM-300	–	–	–	–	–
5	Twin-screw extruder 300 W	10.1	Nica S320	1.50	0.29	1311	172	468
6			Schlüter RM-300	1.37	0.17	1348	94	387
7		13.4	Nica S320	1.35	0.19	1298	132	422
8			Schlüter RM-300	1.15	0.08	1323	100	352
9	Twin-screw extruder 180 W	10.1	Nica S320	1.09	0.08	1228	122	501
10			Schlüter RM-300	1.13	0.11	1688	222	195
11		13.4	Nica S320	1.10	0.08	1380	164	360
12			Schlüter RM-300	1.13	0.09	2037	211	95

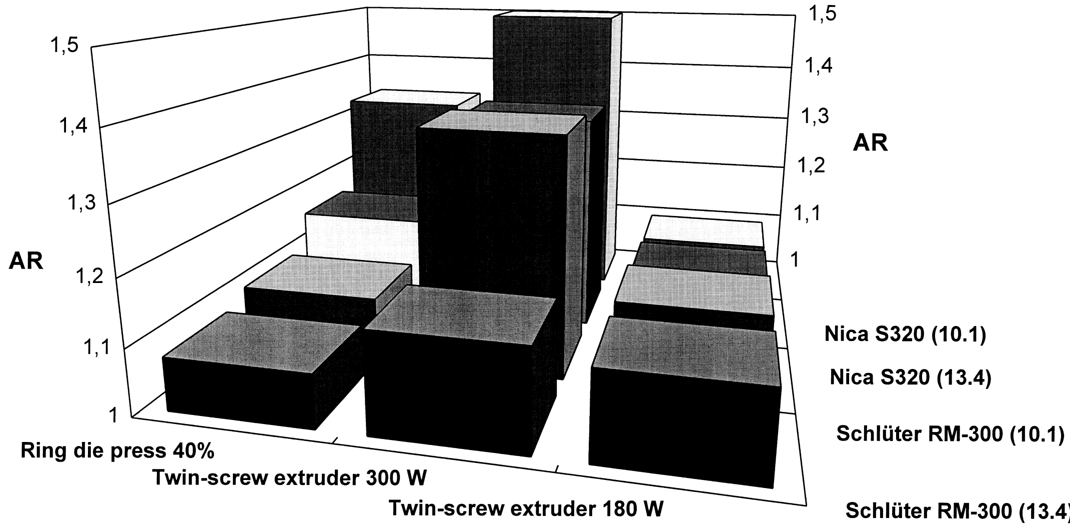

Fig. 1. Relation between extruder type, spheronizer type (radial velocity (m s^{-1})) and AR.

(Fig. 4) which can be visualized by SEM-photographs (Fig. 5). Each pellet batch is numbered according to Table 2. The lactose could be dissolved from the pellets without a disintegration indicating a coherence of the microcrystalline cellulose (Fig. 5). Non-agglomerating, round pellets resulted from certain combinations of all process variables discussed, e.g. twin-screw extruder 180 W, Nica S320 or ring die press 40%, Schlüter RM-300. Starting at a set of optimal conditions changes in process variables can lead to a decrease of pellet quality. As a consequence the optimal moisture content is dependent on the whole process. Mainly it is influenced by the formulation [4] and the extruder type [1]. Although the spheronizing conditions are of minor importance they can be used to improve the results for sub-optimal extrudates. An underwetted extrudate with regard to a certain spheronization process results in lower AR values by increasing the spheronizer velocity (e.g. batch no. 5 → 7, Table 2, Fig. 4) and/or by using the rougher friction plate (e.g. 5 → 8, 5 → 6). On the other hand an agglomeration of a slightly overwetted extrudate can be avoided by the use of a smoother friction plate (e.g. batch no. 12 → 11, Table 2, Fig. 4) and/or a lower velocity (e.g. 12 → 9, 12 → 10).

4. Conclusions

Good pellets in terms of shape and size can only be produced, if the influence variables on all stages of the process are balanced. So successful pelletization requires the control of the whole process. The optimal moisture content for the production of good pellets is dependent on all process

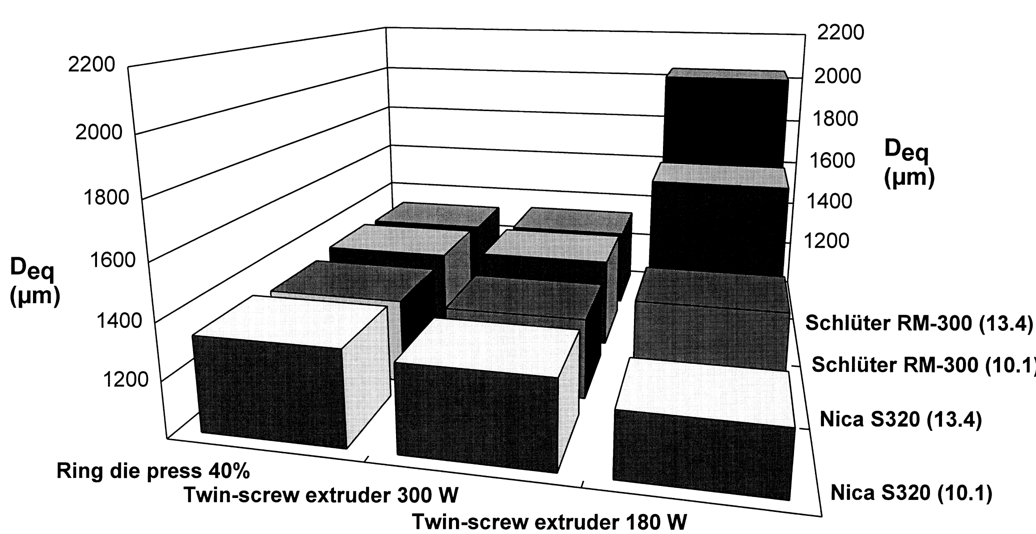

Fig. 2. Relation between extruder type, spheronizer type (radial velocity (m s^{-1})) and D_{eq}.

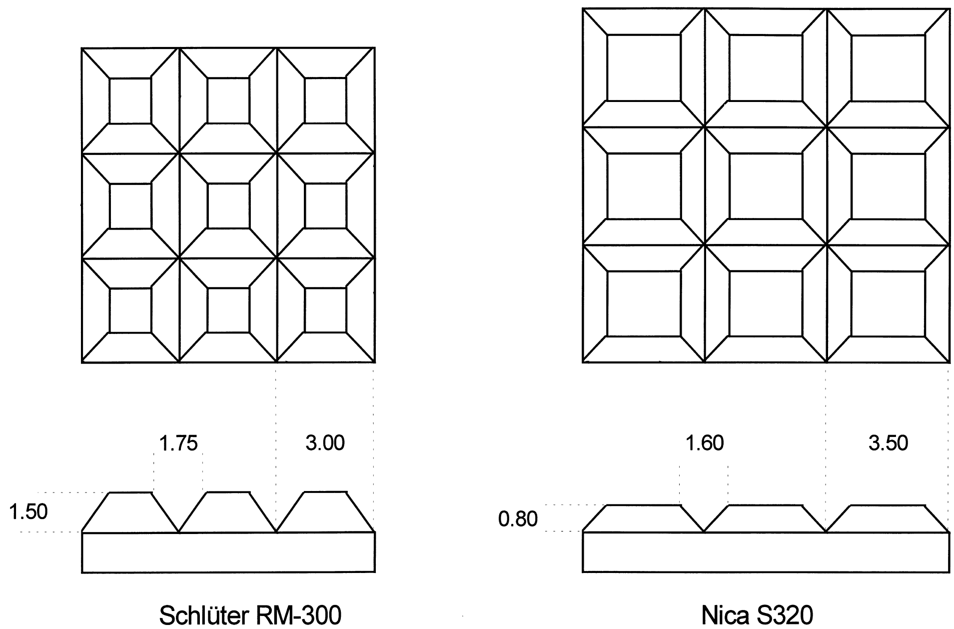

Fig. 3. Schematical drawings of the friction plates of two types of spheronizer: Nica S320 and Schlüter RM-300

stages. Changes in one or more variables must be compensated in many cases by different settings of other variables, e.g. a change in the extruder type could be compensated by a certain adaptation of the moisture content. The granulation/extrusion process has a higher impact on the optimal moisture content and the pellet quality than the spheronization process.

After eliminating the influence of all formulation and process variables except the extruder type the experiments confirmed the results of the first part of this paper. The two extruders produce extrudates of different optimal moisture contents: extrudates from the twin-screw extruder obey a higher optimal moisture content. This can be explained by the formation of a more delicate network of the crystallite-gel in the twin-screw extruder used.

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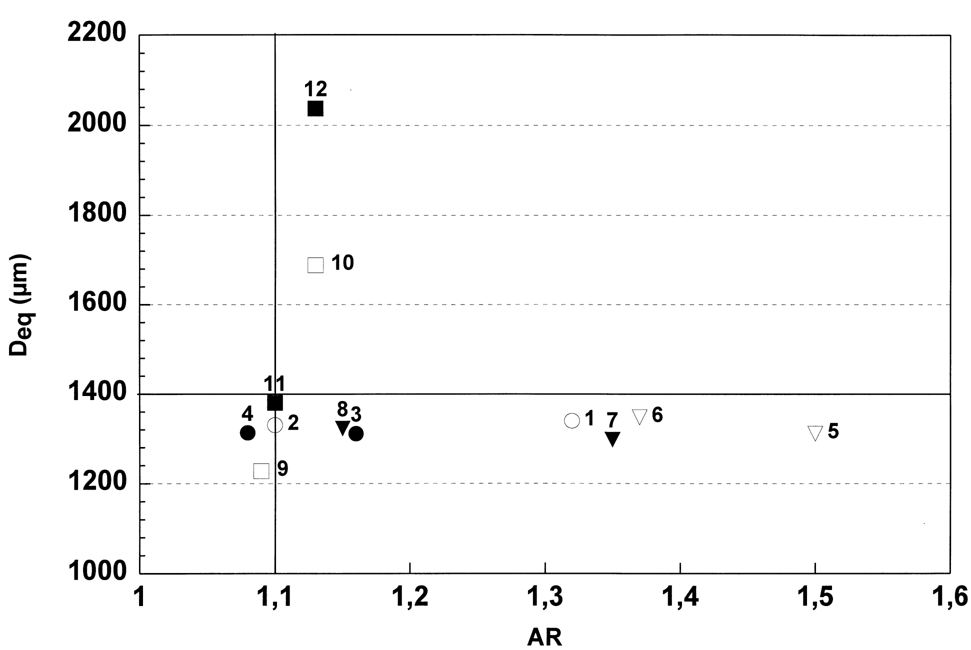

Fig. 4. Relation between AR and D_{eq} in dependence of extrudate, spheronizer type and spheronizer speed. Numbers refer to batch no. (Table 2). ○●, Ring die press 40%; ▼△, twin-screw extruder 300 W; □■, twin-screw extruder 180 W; open symbols, radial velocity = 10.1 m s^{-1}; closed symbols, radial velocity = 13.4 m s^{-1}.

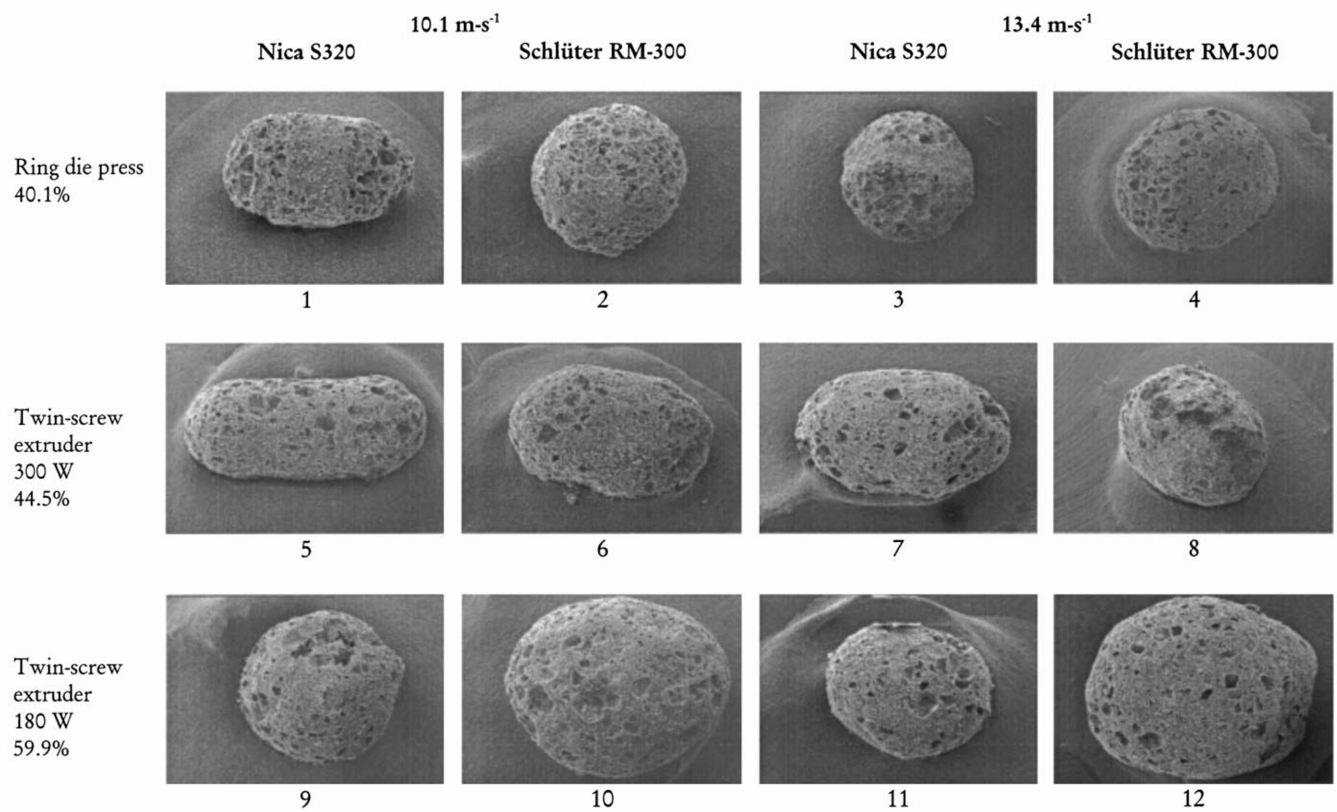

Fig. 5. SEM-photographs of one pellet per batch. Numbers refer to batch no. (Table 2).

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