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Spray Drying of Rice Starch –Aspects in Application

Wolfgang J. Bergthaller[1], Saiyavit Varavinit[2] and Rungtiwa Wongsagonsup[2]
[1] Institute for Cereal, Potato, and Starch Technology, Federal Research Centre for Food and Nutrition, Detmold, Germany
[2] Department of Biotechnology, Faculty of Science, Mahidol University, Bangkok, Thailand

Abstract

Spherical agglomerates of starch isolated from different rice types (regular indica, waxy indica, and regular japonica) were produced by spray drying following a three-factor, three-level central composite design. The process variables selected for this investigation were alternative ways of rice starch extraction (by an alkaline or an enzyme supported process), the concentration of suspended starch (15 to 30 % in dry basis) and the rotation rate of the wheel atomizer of a small scale drying unit (20.000 to 26.000 min^{-1}). Characteristics selected as typical for spherical starch agglomerates were the median particle size of the logarithmic frequency curve and the aerated bulk density. With regard to tablet preparation angle of repose, compressibility, cohesion, and angle of spatula of starch agglomerates were determined to finally establish the flowability index as substantial measure for flowing of these agglomerates.

Statistical evaluation of the presented results revealed that starches from the investigated rice types affected particle size formation in different manner. With regular indica and regular japonica rice starch the particle size can be regulated according to the calculated linear model by starch concentration and atomizer rotation rate, as well. For waxy indica rice starch a model reduced to starch concentration was found. Agglomerate size grows accordingly with increasing suspension concentration but decreasing rotation rate. The way of extraction did not affect agglomerate formation. In starch granule characteristics, however, evident but not yet causally confirmed differences could be detected only in protein content and median particle size.

Within the characteristics used in general for calculation of the flowing index cohesion data varied in many cases substantially and were not further used. For the angle of repose a highly significant regression model was found with regular indica rice starch, but not with waxy indica and regular japonica starch. The angle of spatula was not significantly affected in any way. The analysis of compressibility data produced a situation similar to median particle size. These results as well as the fact that the flowability index is additionally calculated on the basis of further quantities (uniformity, cohesion) give to understand that the flowability index is an inadequate quantity to describe flow behaviour within this study.

For aerated bulk density the starch concentration was again the decisive factor in case of waxy indica and regular japonica starch. And another time the starch of regular indica produced a deviating behaviour. Extraction and rotation rate were effective, too.

To evaluate finally the relevance of quantities established for functionality of tablets (crushing strength, friability, and disintegration time), which were produced with the studied rice starch agglomerates, a correlation analysis was made with particle size and bulk density. Its results did neither reveal a consistent position for the used rice types nor for the agglomerate parameters. Looking finally on starch quality existing differences in protein residues together with granule size could help in finding explanations for the observed phenomena.

Introduction

Rice starch is well known as being of small size. Its size (approximately 5 μm) and angular form are responsible for poor flowing ability at ambient moisture content. However, sufficient flowing ability is an important factor in many forms of application of granular components, in particular for dosage of small quantities. Langan [1] indicates that flowability can be significantly enhanced by lowering moisture content below common equilibrium levels of 12 to 15% or with addition of tricalcium phosphate. While a marked reduction of moisture content and its maintenance are costly, the addition of inorganic salts may not be allowed when using such components in pharmaceutical applications.

A well known procedure to improve flowing ability of small size particles of organic materials while processed consists in the use of various agglomeration techniques to receive stable and well treated products [2]. In the context of potential drying processes listed in literature concerning agglomeration spray drying is a suitable procedure to receive suitable agglomerates [2,3]. The success of spray drying is connected with formation of a spray of droplets that represents a high surface to mass ratio. An ideal spray contains small individual droplets of equal size. A homogenous spray will be the result and heat and mass transfer as well as drying times will then be the same for all droplets. At industrial conditions however, the droplet size is controlled by the type of atomizer used and by the atomization variables applied. With wheel atomizers particle size distributions are highly homogenous and depend on wheel speed and feed rate [3].

Since liquids of particle suspensions intended for drying contain at most solutes these can form solid-state bridges that stick particles together. Drying conditions can therefore significantly affect agglomerate stability. In general, high drying rates increase stability, under unfavourable conditions; however, it may also be diminished. Besides the described material bridges *van der Waals* and form-locking forces [2] may be involved as potential principles in formation of agglomerates from starch suspensions.

The formation of spherical starch agglomerates has been described first in connection with spray drying of rice starch (Figure 1) [4]. The agglomerates were produced as an alternative material suited as direct compression filler for production of pharmaceutical tablets. Flowability and compressibility were important properties in this respect. In 1994 also Zhao and Whistler [5] presented microphotographs of a spherically formed commercial amaranth starch, also named popcorn balls. The amaranth starch representing the commercial sample was, however, of minor purity. The formation of spheres from the typical, polygonal amaranth starch granules was explained in particular by the high protein and lipid content of the sample. Also impure rice starch was said to form the same spherical balls. Obviously, Zhao and Whistler [5] were not aware of the possibility to form spherical agglomerates with rice starch of unknown quality simply by spray drying. Later on, spherically formed starch agglomerates were shown occasionally when acid–modified starches of *Lintner* type were produced on the basis of various starches (tapioca starch, waxy or non-waxy rice starch) [6,7]. Additionally, composite particles were developed on the basis of rice starch together with microcrystalline cellulose (MC). Depending on the ratio of the components and the type of MC the form of the agglomerates as well as powder characteristics could be varied from spherical to irregular smooth forms (8).

As pointed out previously spherical starch agglomerates were produced so far with different starch types, in particular, with rice starch of varied pre-treatment. However, no information could be found by which way and to which extent particle size of starch aggregates together with selected powder properties can be affected in spray drying. With respect to rice types available in Thailand three rice types used for isolation, the isolation processes and selected conditions of spray drying; especially the wheel speed and the starch concentration in the feed were studied. As powder characteristics the degree of angle of repose, aerated bulk density, packed bulk density, percent compressibility, percent cohesion, and degree of angle of spatula are used for example. Following the suggestions of Carr [9] the parameters angle of repose, compressibility, uniformity, cohesion and angle of spatula are frequently converted into individual index values to calculate, finally, the flowability index as a numerical characteristic for powder flow. Cohesion is evaluated only when flowability does not come up to normal [8].

Material and Methods

Rice

Polished regular indica and japonica rice were kindly donated by Rithmers Reismühle GmbH (Bremen, Germany). Polished waxy indica rice was purchased in a local Thai shop in Germany. It was shipped and produced by Universal Rice Co., Ltd. (Samutsakorn, Thailand).

Rice starch Isolation

For isolation of starch from regular indica, regular japonica and waxy indica rice the alkaline and enzymatic procedures described recently [10] were applied. Isolation was done on 8 kg rice samples, each. In order to receive in total sufficient quantities of rice starch for spray-drying studies each isolation process was repeated in subsequent experiments.

Proximate Analysis

Quantitative determinations of moisture, lipids (fat), minerals (ash) and fibre content were determined in triplicate by applying standard procedures described by AOAC (methods 925.09, 920.39, 923.03, and 962.09) [11]. The nitrogen content was determined in duplicate in applying the Dumas combustion principle. For calculation of crude protein a factor of 5.95 was used [12]. The total starch content was determined polarimetrically [13] and starch damage was investigated after enzyme digestion by using commercial test kits described in the Megazyme test procedure (Megazyme International Ireland Ltd., Wicklow, Ireland)[14]. The amylose content was determined by the iodine affinity method [15].

Experimental Design

In order to achieve differences in particle size of starch agglomerates the rotation speed of the spray wheel ("rotation rate") and the concentration of the starch suspension ("concentration") were varied. Since impurities of starch were discussed as effecting the formation of "starch balls" [5] also potential effects of the isolation procedures (alkaline vs. enzymatic, "extraction") were tested, although starches originating from both processes did not reveal significant differences in proximate analysis in a previous study [10]. Following a three-factor, three-level central composite design [16] the effects of process variables on particle size distribution of agglomerates as well as characteristics of flowing ability were investigated. The process variables selected were: type of starch isolation (enzymatic, 50% mixtures, alkaline), slurry concentration (15, 22.5 and 30% dry substance) and rotation speed of atomization wheel (20,000, 23,000 and 26,000 min^{-1}). The levels of each variable were coded as -1, 0, +1, +0.5 and -0.5 for statistical analysis. The levels of the variables slurry concentration and rotation speed were established on the basis of previous unpublished experiments. Dependent variables included the median particle size (D50), the degree of the angle of repose, the aerated and packed bulk density, the compressibility and the degree of the angle of spatula. The experimental design consisted of 16 experimental runs which included two replicates at the centre point (Table 1).

S*pray Drying Experiments*

In preparing slurries for spray drying starch samples of approximately 400 g dry substance were suspended in de-ionized water in composition and concentration according to the experimental design (Table 1). After 0.5 h stirring the slurry was cleared from hard not completely disintegrated pieces of starch. Sieving through a 56 µm sieve of a horizontal vibration sifter (Schallfix Type L2426, Rhewum, Remscheid-Lüttringhausen, Germany) provided a homogenous suspension.

Immediately after sieving the starch suspensions were spray dried in direct stream in a small scale drying unit, Type Minor Production (NIRO A/S, Soeborg, Denmark) equipped with a wheel atomizer, Type FS-1, and having a water evaporation capacity of 13 $kg \cdot h^{-1}$. Rotation speed levels of the atomizer were adjusted according to the experimental design (Table 1). The drying air temperature was set to 190°C at the drying air entrance and to 90°C at the drying air outlet in all experiments. The feed rate was automatically controlled by the system. Each sample of collected starch was cooled to ambient temperature, filled in a polyethylene bag and sealed, and finally stored at room temperature until investigation.

Particle Size Determination

Particle size distribution of isolated starch samples were measured as described by Puchongkavarin et al [7]. The distribution of respective agglomerates was analysed in liquid dispersion with denatured 96% ethanol as the liquid phase by using a laser light scattering instrument, type Mastersizer S (Malvern Instruments Ltd., Malvern, UK). The applied measuring range was 0.01-

2000 µm, with a range lens of 300 RF mm and a beam length of 2.40 mm. All measurements were made in duplicate. With regard to the observed asymmetry of the logarithmic distributions (Figure 2) the median particle size (D50) [µm] of rice starches and rice starch agglomerates was used to characterize the samples.

Flowing Characteristics

For identification of powder flow properties measurements were made with a powder characteristics tester (Model PT-N, Hosokawa Micron Corporation, Osaka, Japan) [8]. The tested characteristics produced data for the angle of repose [°], compressibility [%], cohesion [%] and angle of spatula [°]. Angle of repose, compressibility, cohesion and angle of spatula data were converted into index values according to Carr [9] and summarized to result in flowability indices (Data not shown). Furthermore, data for aerated bulk density [$kg \cdot L^{-1}$] and packed bulk density [$kg \cdot L^{-1}$] were measured with each sample.

Tablet Preparation and Evaluation

On the basis of the prepared samples of spray dried rice starch tablets were prepared in a single punch rotary tablet machine (Type N3B, Narongkarnchange, Bangkok, Thailand) by a direct compression method [7,20]. The tablet machine was equipped with resistance strain gauges for monitoring the compression force. With increasing compression force (≥ 4 kN) capping occurred frequently in tablet pressing and limited determination of tablet properties to the 2 kN compression experiments.

In evaluation of tablet characteristics hardness was determined as crushing strength [N] in an electric hardness tester (Model 4M, Dr. Schleuninger Co., Solothurn, Switzerland), percent friability by a Roche type friabilator (Narongkarnchange, Bangkok, Thailand) and the disintegration time [min] by a USP disintegration apparatus (Model AC 24, Hanson Research Corp., California, U.S.A.) [21].

Results and Discussion

Proximate Composition

The chemical composition of the rice samples (regular and waxy indica rice, regular japonica rice) used for starch isolation is presented in Table 2. Pronounced, but under an agronomical point of view not surprising differences can be seen between samples of polished regular indica and japonica rice in the content of total starch and the protein content. Waxy indica rice was lower in starch content. Interestingly, the waxy indica rice has had a three times higher fibre content.

Recovery and Composition of Isolated Starch

Except the enzymatic isolation from waxy indica rice recovery of rice starch reached in level and in distance results as previously reported on indica rice basis [9] for both, the alkaline and the enzymatic process. Moreover, in recovery the alkaline process surpassed with every rice type the enzymatic one. Regular japonica rice that contained the highest level of starch allowed the best extraction. Recovery from waxy indica rice and regular indica rice followed in an opposite order. But surprisingly, with waxy indica rice the enzymatically-assisted extraction of starch could not reach by far the level of the alkaline procedure. The higher content in crude fibre of the waxy rice type cannot be used presumably as indication for this phenomenon, but on the other hand no other characteristic can help to explain the deviating behaviour, yet.

The levels of residual protein content in the extracted starches revealed differences obviously effected by the rice type, but not that much by the extraction procedure. Lipid and mineral content were to a certain content similar to recently reported figures. The observation that the applied process type (alkaline vs. enzymatic) can be assessed in typical differences of damaged starch could be repeated, but on a lower level. The individual higher levels in damaged starch attributed at last to both applied procedures [10] could not be determined this time. Abandoning in a final aggressive milling previous to determination of damaged starch [15] seemed to allow a significantly smaller level of starch damage. However, the process induced differences observed yet for damaged starch could be found again.

The amylose content in isolated starches demonstrated typical differences depending on the rice type, especially when a waxy type was used as substrate. Significant differences induced by the way of processing could not be observed except the observation that in starch extracted from waxy rice by the alkaline procedure amylose could not be detected any more. This coincides with differences in relative crystallinity determined for the waxy rice starches (52.4 % from alkaline processing versus 50.9 % from the enzymatic one).

Determinations in particle size demonstrated dimensions typical for rice starch, in general [7, 17, 18]. With approximately 5.4 µm median particle size waxy indica rice starch was the bigger type while regular indica rice starch had approximately 4.8 µm. Differences caused by the extraction procedure became obvious only with regular japonica rice starch (Table. 3).

Spray drying experiments

With samples of the spray drying experiments the median particle size and the following powder characteristics demonstrating divers aspects of the flowing behaviour of agglomerated particles have been measured in duplicate: degree of angle of repose, aerated bulk density, packed bulk density, percent compressibility, percent cohesion, and degree of angle of spatula. Mean values of the results were compiled by Wongsagonsup et al. [19] and are presented in Table 4. Data determined for cohesion were not included since they proved to be extremely erroneous.

On the basis of derived mean values of the evaluated particle size results and the results of the indicated powder characteristics multiple regressions and analyses of variance were calculated by using the coded experimental design [16]. With all investigated dependent variables the interactions "extraction*concentration", "extraction*rotation rate" and "concentration*rotation rate" were tested as insignificant ($p > 0.05$) and not further regarded in succeeding regression analyses of the decoded experimental design.

Median Particle Size

In following calculations of the decoded and reduced design the extraction procedure revealed as insignificant ($p > 0.05$) with the starches of all three rice types (Table 5). In two cases, in particular, regular indica and regular japonica rice, they allowed then to establish very similar, highly significant ($p < 10^{-6}$) linear models with highly significant ($p < 0.001$) effects of a positive coefficient for the starch concentration used in spray drying and a negative coefficient for the rotation speed of the atomizer. A typical graph describing the situation of median particle size of agglomerated spherical particles found for starches originating from regular indica and regular japonica rice and evaluated by applying the described un-coded experimental design (Table 1) is presented in Figure 3. An increasing concentration of the starch suspension seems to produce the decisive effect in forming the size of droplets by the atomizer. On the other hand, the higher the rotation speed becomes the more get the droplets reduced and the agglomerated rice starch spheres become smaller after vaporizing the water. Although soluble matter was not added that could help to stick together the starch granules, relative stable spherical agglomerates are formed sticking together by form-locking forces and/or *van der Waals* forces [2] or even water bridges. As long as water as a highly polar liquid phase is not used in producing suspensions in following investigations the starch agglomerates do not fall apart to individual granules.

With waxy rice starch a more simple, but also highly significant ($p < 10^{-6}$) model was found, characterised by a linear relationship of the highly significant ($p < 0.001$) effect of the concentration of the starch suspension, only. In Figure 4 the respective relationship including the 95% confidence intervals is demonstrated. A specific proof for the divergent behaviour of waxy indica rice starch could not be found, yet, and has not been described in literature.

Flowing Characteristics

As the first characteristic the angle of repose was evaluated. In the reduced coded design and then in the decoded design only with regular indica rice starch a highly significant ($p < 10^{-6}$) effect of the variables concentration of starch in the suspension and rotation speed of the atomizer could be found (Table 5). The respective coefficients of the significant ($p < 4 \cdot 10^{-6}$) linear model were also highly significant each ($p < 4.10^{-5}$). For waxy indica starch and regular japonica starch significant

($p < 0.05$) models could not be established. The specific behaviour of regular indica rice starch cannot be explained so far.

Compressibility was determined as the second characteristic. The results of the reduced coded and decoded designs allowed to establish highly significant ($p < 0.001$), but no very close linear models. With respect to the negative, highly significant ($p < = 0.001$) and very similar coefficients determined for regular indica, waxy indica and regular japonica rice, as well, the starch concentration in the spray suspension reduces compressibility in an important way, which is an indication for less dens packing of starch granules in the respective agglomerates. However, with regular indica rice starch an additional regression coefficient has to be regarded. Here, the rotation rate of the atomizer plays also a significant ($p < 0.001$) role by increasing the calculated compressibility to a level that corresponds fairly well the other starches at the central point.

The third characteristic determined in the frame work of flow behaviour, the angle of spatula, could not be distinguished as being affected in an important extent by the tested independent variables. The type of extraction as well as starch concentration in the spray and the rotation rate of the atomizer did not affect this aspect of flow behaviour in a significant way ($p > 0.05$).

Having in mind the results of multiple regression analysis of the experimental design described in this study of spray drying of rice starch (Table 5) and additionally the fact that the flowability index according to Carr [9] is calculated on the basis of quantities not studied so far (uniformity, cohesion), also from this point of view the flowability index cannot be regarded as the adequate quantity to describe flow behaviour of spherically agglomerated rice starch.

Aerated Bulk Density

Although aerated bulk density was determined only as an auxiliary quantity in calculation of characteristics of flow behaviour, significant ($p < 0.001$) effects of starch concentration of suspensions used in spray drying could be found in the reduced regression analysis of the uncoded experimental design for waxy indica and regular japonica rice starch. With regular indica rice starch the situation was more complex again. Here, the type of extraction and the rotation rate of the atomizer played an additional and important role, as can be deduced from the corrected multiple correlation coefficient (corr. $R^2 = 0.883$) and a comparison of observed and predicted results (Figure 5).

Effects on Tablet Properties

The results of the determination of crushing strength (as characteristic for tablet hardness), percent friability and disintegration time as important functional properties of tablets are presented in Table 6. In order to reveal relations between the tested tablet properties and the median particle size and aerated bulk density of rice starch agglomerates, which were established as relevant in the preceding investigation of spray drying, correlation coefficients were calculated and compiled in Table 7. In contrast to earlier ideas only in few cases a relationship proved to be significant and only with crushing strength as a measure for tablet hardness and for regular japonica starch the relation was closer. With respect to results presented previously by Puchongkavarin et al. [7], according to which crystallinity was the decisive factor for tablet functionality, these results may not be surprising. But, agglomerate size and also bulk density were not regarded for investigation so far.

Conclusions

Rice starch agglomerates recovered from spray drying have been used as excipients in production of pharmaceutical tablets. Especially their improved flowability was of interest. From previous investigations it was obvious that spray dried starch aggregates show a logarithmic frequency curve, but process parameters affecting this pattern were not reported in literature. The results gained on the basis of a central composite design using the way of starch extraction, the concentration of starch suspensions and the wheel rotation rate of the atomizer produced in part significant regression equations with coefficients of varying quality in their contribution to the prediction of the selected characteristic, e.g. median particle size, aerated bulk density or measures of flowing behaviour. While the way of starch extraction was of minor importance, the role of rice type found its expression in the differentiation of calculated regression coefficients and their significant or even insignificant contribution. The amount of residual proteins on the starch isolates, potentially in form of surface proteins, could give an indication in explaining the determined differences. Regarding flow behaviour the frequently observed insignificant effect of the used process parameters on measures that provide an important contribution in calculating the flowability index was reason to do further investigations without flow characteristics. However, the following evaluation of significant relations of reasonable tablet properties (crushing strength as a measure of hardness, friability, and disintegration time) with agglomerate properties (particle size or bulk density) by calculation of correlation coefficients could not provide a clear description of the investigated parameters. Previous investigations about the importance of crystallinity for formation of functional tablet properties could give an explanation for the findings presented here.

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Figure 1 Spherical rice starch agglomerates produced by spray drying mentioned 1988 and presented for the first time by Varavinit & Mitrevej [4]

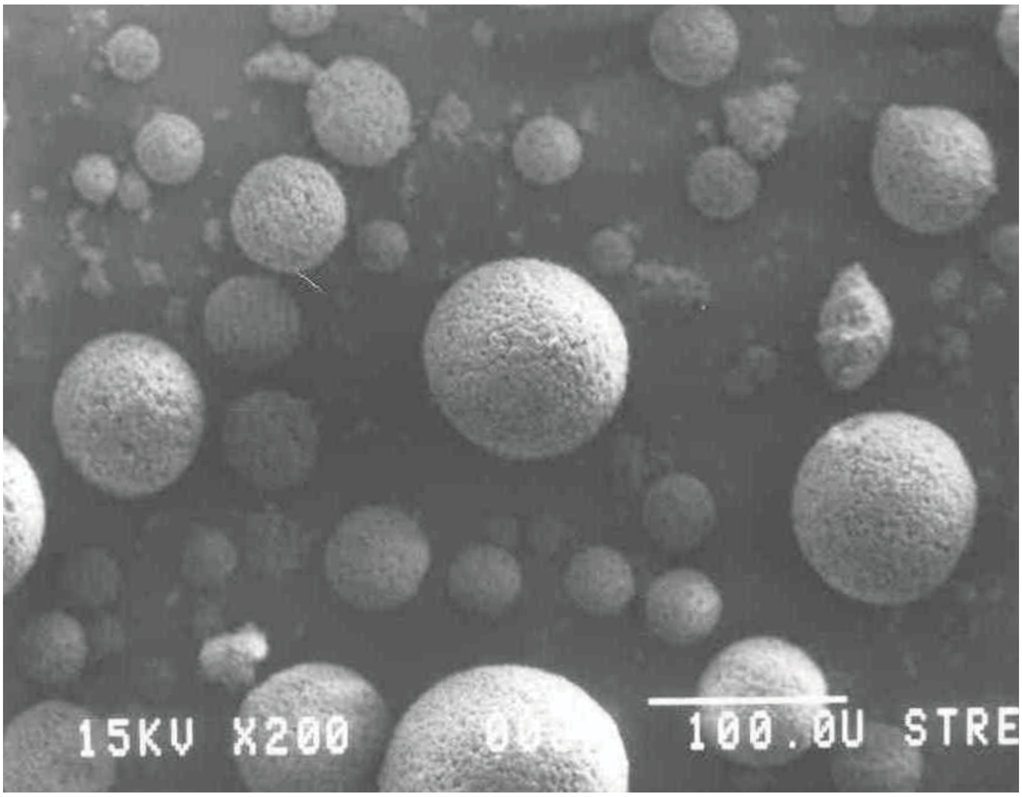

Table 1 Experimental design used in spray drying studies

Run no.	Independent variables					
	Coded values			Real values		
	x_1	x_2	x_3	Starch ratio [%] / Isolation type*	Concentration [% db. w/w]	Rotation speed [min^{-1}]
1	1	1	1	100 E	30	26,000
2	1	1	-1	100 E	30	20,000
3	1	-1	1	100 E	15	26,000
4	1	-1	-1	100 E	15	20,000
5	-1	1	1	100 A	30	26,000
6	-1	1	-1	100 A	30	20,000
7	-1	-1	1	100 A	15	26,000
8	-1	-1	-1	100 A	15	20,000
9	0	0	0	50 E/50 A	22.5	23,000
10	0	0	0	50 E/50 A	22.5	23,000
11	0.5	0	0	75 E/25 A	22.5	23,000
12	-0.5	0	0	25 E/75 A	22.5	23,000
13	0	0.5	0	50 E/50 A	26.25	23,000
14	0	-0.5	0	50 E/50 A	18.75	23,000
15	0	0	0.5	50 E/50 A	22.5	24,500
16	0	0	-0..5	50 E/50 A	22.5	21,500

* E: Enzyme-based isolation
A: Alkaline-based isolation

Figure 2 Comparison of frequency curves of native and agglomerated rice starch

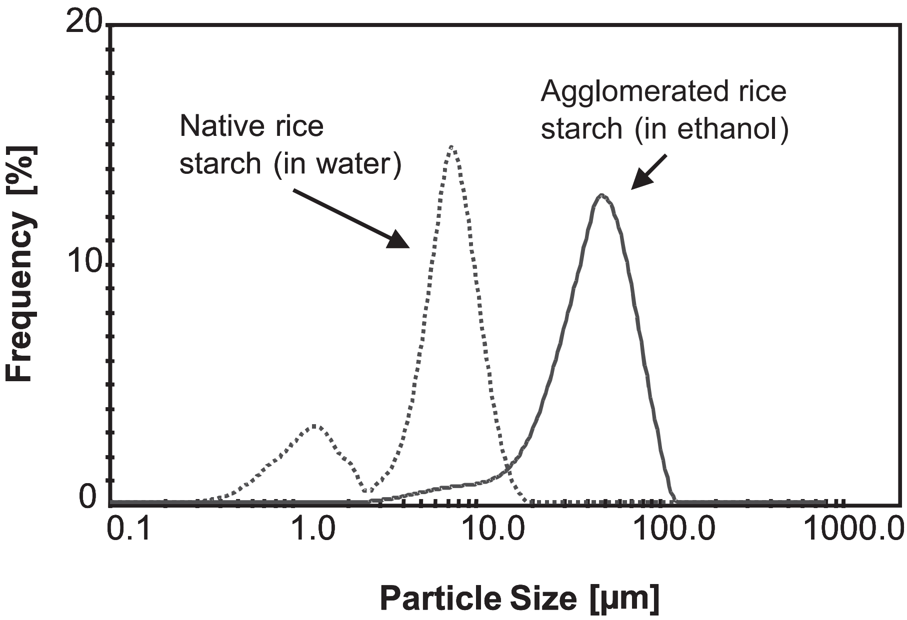

Table 2 Chemical composition of rice samples used for starch isolation

Sample	Protein content [% db.] N x 5.95	Lipid content [% db.]	Mineral content [% db.]	Fibre content [% db.]	Total Starch Content [% db.]
Regular indica rice	9.52±0.03[a]	0.26±0.02[ab]	0.44±0.01[a]	0.06±0.01[c]	89.6 ± 0.26[b]
Waxy indica rice	8.58±0.19[b]	0.21±0.01[b]	0.30±0.00[c]	0.23±0.00[a]	89.0 ± 0.02[c]
Regular japonica rice	7.11±0.01[c]	0.27±0.00[a]	0.37±0.00[b]	0.08±0.00[b]	91.9 ± 0.13[a]

Table 3 Recovery, chemical composition* and particle size* of isolated rice starches

Sample	Recovery [% db.]	Protein content [% db.] N x 5.95	Lipid content [% db.]	Mineral content [% db.]	Amylose Content [% db.]	Damaged Starch [% db.]	Median of Particle Size (mm)
Regular indica RS							
- Alkaline	77.31	0.60 ± 0.00^{b}	0.10 ± 0.01^{b}	0.21 ± 0.01^{a}	26.1 ± 0.32^{a}	2.28 ± 0.02^{c}	4.74 ± 0.00^{a}
- Enzymatic	74.29	0.72 ± 0.01^{a}	0.02 ± 0.01^{a}	0.09 ± 0.00^{c}	25.7 ± 0.14^{a}	1.45 ± 0.00^{f}	4.83 ± 0.00^{a}
Waxy indica RS							
- Alkaline	78.04	0.10 ± 0.00^{e}	0.08 ± 0.00^{b}	0.06 ± 0.00^{d}	0.0 ± 0.00^{c}	2.34 ± 0.03^{b}	5.31 ± 0.01^{c}
- Enzymatic	58.69	0.25 ± 0.01^{d}	0.06 ± 0.03^{b}	0.03 ± 0.01^{e}	0.6 ± 0.01^{c}	1.64 ± 0.03^{e}	5.38 ± 0.02^{c}
Regular japonica RS							
- Alkaline	80.00	0.27 ± 0.00^{c}	0.06 ± 0.00^{b}	0.15 ± 0.01^{b}	16.4 ± 0.39^{b}	2.85 ± 0.01^{a}	4.98 ± 0.10^{b}
- Enzymatic	77.31	0.26 ± 0.00^{c}	0.06 ± 0.03^{b}	0.08 ± 0.00^{c}	16.4 ± 0.31^{b}	1.76 ± 0.03^{d}	5.37 ± 0.00^{c}

Table 4 Mean values of dependent variables compiled according to rice type

Rice Type	Regular Indica				
Run No.	Dependent Variables				
	Median Particle Size [µm]	Angle of Repose [°]	Aerated Bulk Density [kg·L^{-1}]	Compressibility [%]	Angle of Spatula [°]
1	39.27	46.80	0.414	29.45	61.85
2	46.06	34.90	0.439	26.25	59.70
3	23.99	49.50	0.366	36.15	59.45
4	29.28	45.25	0.392	31.75	57.45
5	41.79	43.65	0.463	24.85	55.25
6	45.63	37.00	0.492	20.00	54.25
7	24.33	52.30	0.391	35.55	55.10
8	29.98	44.70	0.419	31.45	66.20
9	34.27	46.25	0.427	27.90	63.50
10	34.38	40.85	0.424	28.10	62.30
11	32.09	45.60	0.424	27.20	63.70
12	34.29	45.70	0.441	27.00	61.40
13	39.04	40.10	0.450	24.90	54.00
14	30.25	45.90	0.426	28.65	61.85
15	31.82	46.80	0.439	26.15	57.55
16	33.83	42.95	0.436	25.80	57.20

Rice Type	Waxy Indica Rice				
Run No.	Dependent Variables				
	Median Particle Size [µm]	Angle of Repose [°]	Aerated Bulk Density [kg·L^{-1}]	Compressibility [%]	Angle of Spatula [°]
1	43.00	47.35	0.443	23.65	61.85
2	41.49	46.90	0.444	19.80	59.70
3	23.31	56.15	0.370	32.00	59.45
4	25.20	54.90	0.371	31.90	57.45
5	39.70	52.40	0.440	23.50	55.25
6	41.91	49.65	0.439	21.90	54.25
7	24.34	49.80	0.373	30.30	55.10
8	27.41	51.15	0.383	30.70	66.20
9	30.00	53.95	0.394	29.95	63.50
10	31.87	54.35	0.401	29.55	62.30
11	32.47	43.50	0.359	31.50	63.70
12	33.90	47.60	0.412	27.95	61.40
13	33.90	45.60	0.415	26.50	54.00
14	28.18	46.80	0.383	33.00	61.85
15	29.92	53.15	0.400	30.05	57.55
16	31.00	51.90	0.396	32.00	57.20

Rice Type	Regular Japonica Rice				
Run No.	Dependent Variables				
	Median Particle Size [µm]	Angle of Repose [°]	Aerated Bulk Density [kg·L^{-1}]	Compressibility [%]	Angle of Spatula [°]
1	40.66	44.70	0.482	19.85	57.25
2	44.72	40.75	0.478	20.95	56.90
3	25.16	46.50	0.415	29.55	62.75
4	30.93	47.05	0.414	29.15	58.40
5	43.15	43.40	0.493	22.60	62.30
6	46.84	43.10	0.512	16.95	55.55
7	25.31	52.85	0.408	32.05	59.35
8	30.99	47.35	0.447	26.40	56.45
9	34.12	45.00	0.449	26.75	61.60
10	35.70	44.15	0.451	23.75	63.30
11	34.37	48.50	0.457	23.70	61.30
12	33.02	46.25	0.480	22.80	64.40
13	39.67	51.15	0.466	23.90	64.85
14	32.09	54.05	0.457	24.05	60.65
15	33.77	53.95	0.453	31.50	63.60
16	35.79	50.80	0.482	22.50	66.95

Table 5 Results of multiple regression analysis for median particle size, angle of repose, aerated bulk density, compressibility and angle of spatula with spray dried rice starch by variation of extraction type, concentration of starch in suspension and rotation rate of the atomizer with regular indica, waxy indica and regular japonica rice

Dependent Variable	Regular Indica				Waxy Indica				Regular Japonica			
	Independent Variable	Coefficient	R^2 (corr.)	p - Vaue	Independent Variable	Coefficient	R^2 (corr.)	p - Vaue	Independent Variable	Coefficient	R^2 (corr.)	p - Vaue
Median Particle Size	Constant	30.2030	0.968	$< 10^{-6}$	Constant	8.1017	0.921	$< 10^{-6}$	Constant	30.0899	0.962	$< 10^{-6}$
	Extraction	n.s.			Extraction	n.s.			Extraction	n.s.		
	Concentration	1.0912			Concentration	1.0776			Concentration	1.0484		
	Rotation rate	-0.0009			Rotation rate	n.s.			Rotation rate	-0.0008		
Angle of Repose	Constant	26.5097	0.830	$< 4 \cdot 10^{-6}$	Constant	n.s.			Constant	n.s.		
	Extraction	n.s.			Extraction				Extraction			
	Concentration	-0.5067			Concentration				Concentration			
	Rotation rate	0.0013			Rotation rate				Rotation rate			
Aerated Bulk Density	Constant	0.4346	0.883	$< 2 \cdot 10^{-6}$	Constant	0.3005	0.782	$< 3 \cdot 10^{-6}$	Constant	0.3583	0.732	$< 1.5 \cdot 10^{-6}$
	Extraction	-0.0190			Extraction	n.s.			Extraction	n.s.		
	Concentration	0.0039			Concentration	0.0045			Concentration	0.0045		
	Rotation rate	-0.000004			Rotation rate	n.s.			Rotation rate	n.s.		
Com-pressibility	Constant	25.8969	0.718	$< 1.1 \cdot 10^{-4}$	Constant	42.2612	0.705	$< 2.9 \cdot 10^{-5}$	Constant	37.7928	0.578	$< 3.83 \cdot 10^{-4}$
	Extraction	n.s.			Extraction	n.s.			Extraction	n.s.		
	Concentration	-0.5682			Concentration	-0.6165			Concentration	-0.5784		
	Rotation rate	0.00066			Rotation rate	n.s.			Rotation rate	n.s.		
Angle of Spatula	Constant	n.s.			Constant	n.s.			Constant	n.s.		
	Extraction				Extraction				Extraction			
	Concentration				Concentration				Concentration			
	Rotation rate				Rotation rate				Rotation rate			

n.s.: not significant

Figure 3 Regression plot describing the contribution of starch concentration (x) and atomizer rotation speed (y) on median particle size (D50) of starch agglomerates (regular indica rice)

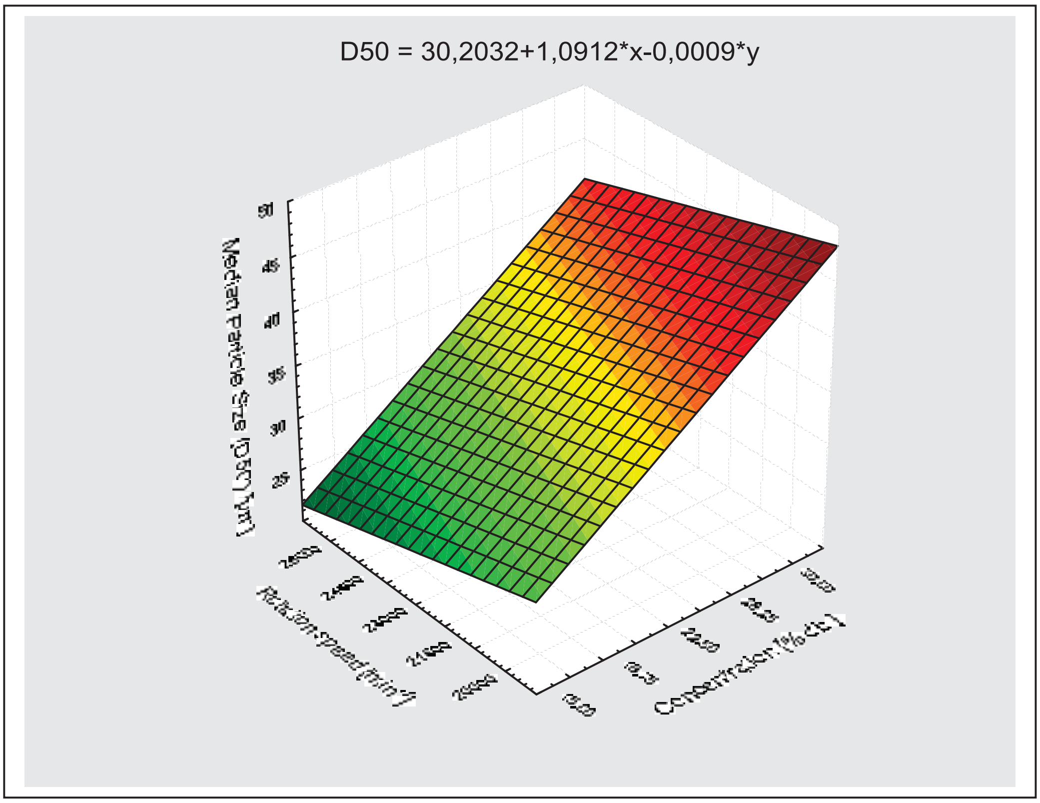

Figure 4 Regression plot presenting the effect of starch concentration on median particle size of agglomerated starch with waxy indica rice

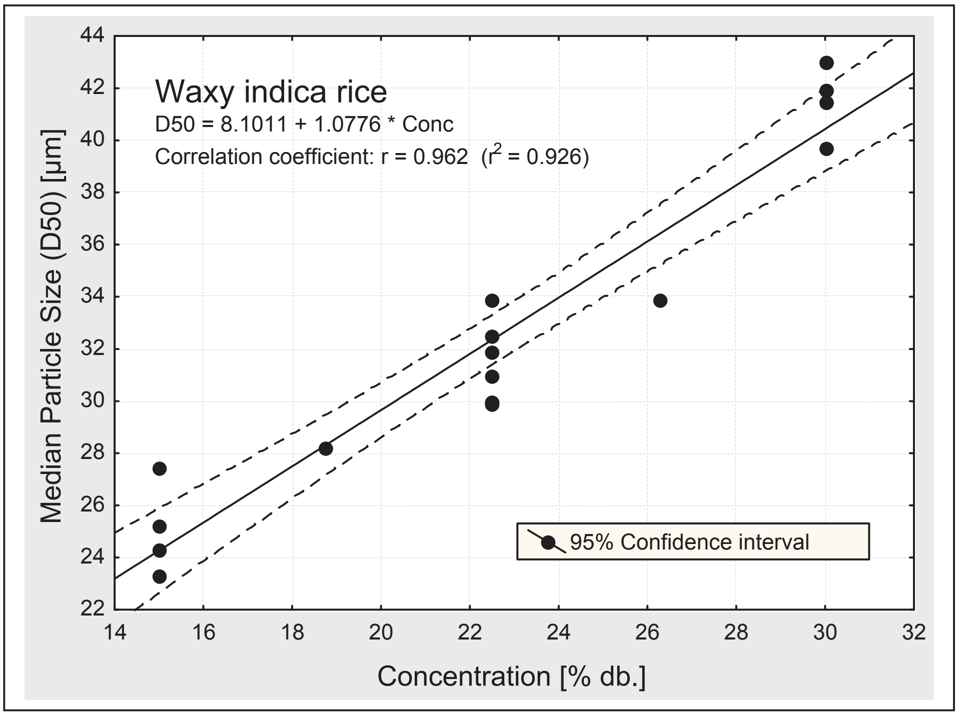

Figure 5 Relation of observed and predicted results of aerated bulk density of agglomerates of spray dried regular indica rice starch

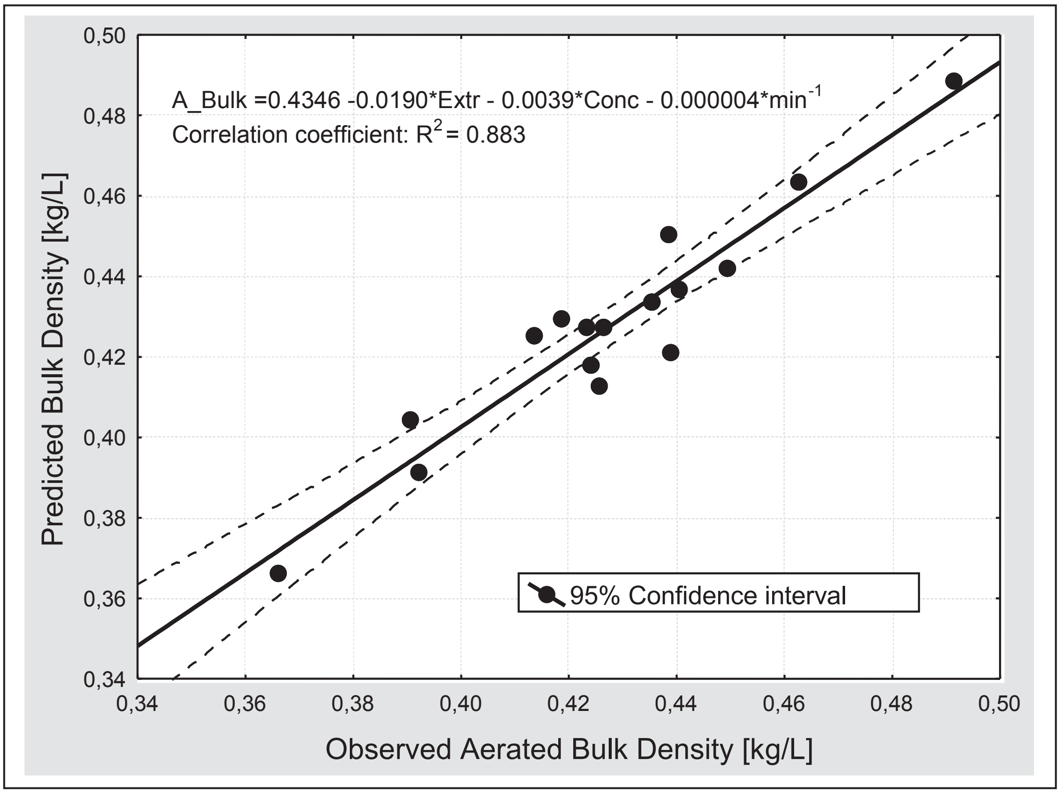

Table 6 Results of the evaluation of tablet properties produced on basis of rice starch agglomerates

Rice Type	Regular Indica			Waxy Indica			Regular Japonica		
Run no.	Crushing strength [N]	Friability [%]	Disintegration time [min^{-1}]	Crushing strength [N]	Friability [%]	Disintegration time [min^{-1}]	Crushing strength [N]	Friability [%]	Disintegration time [min^{-1}]
1	39.23	1.58	2.63	48.05	1.39	2.65	27.46	3.64	2.040
2	38.25	1.22	2.55	40.21	2.31	2.47	23.54	0.46	2.170
3	45.11	1.03	1.88	61.78	1.03	3.05	46.09	1.66	2.310
4	49.03	0.82	2.5	61.78	1.02	2.94	43.15	1.74	2.230
5	57.86	0.93	2.94	50.02	0.83	3.32	12.75	9.04	2.320
6	18.63	3.15	3.14	46.09	0.99	3.55	14.71	7.46	2.230
7	43.15	1.68	2.62	70.61	1.59	3.4	36.29	2.33	2.470
8	41.19	1.27	2.31	60.8	1.24	4.13	36.29	1.81	2.430
9	25.5	2.57	2.62	46.09	1.38	3.22	30.40	2.59	2.150
10	31.38	1.56	2.91	53.94	0.93	2.69	28.44	2.75	1.980
11	34.32	1.52	2.97	50.02	0.61	2.83	35.31	2.03	2.280
12	31.38	2.14	2.51	48.05	0.97	2.68	29.42	2.69	2.360
13	25.5	2.45	2.85	40.21	1.55	3.06	36.29	2.00	2.380
14	36.29	1.85	2.74	44.13	1.07	2.63	32.36	1.88	2.170
15	39.22	1.6	2.93	49.04	1.16	2.77	34.32	2.15	2.730
16	38.25	1.68	2.11	42.17	1.62	4.1	32.36	2.48	3.210

Table 7 Comparison of correlation coefficients of tablet and rice starch agglomerate properties

	Median Particle Size (D50)			Aerated Bulk Density		
Tablet Characteristic	Regular Indica	Waxy Indica	Regular Japonica	Regular Indica	Waxy Indica	Regular Japonica
Crushing Strength	0.318	0.638**	0.823***	0.447	0.574*	0.833***
Percent Friability	0.345	0.177	0.537*	0.576*	0.337	0.581*
Disintegration Time	0.498*	0.209	0.178	0.655*	0.143	0.045

* $p < 0.05$ ($t_{n-2} = 0.497$)
** $p < 0.01$ ($t_{n-2} = 0.623$)
*** $p < 0.001$ ($t_{n-2} = 0.742$)