

Yield of Starch and By-Products in the Processing of Different Varieties of Wrinkled Peas on a Pilot Scale

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ABSTRACT

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A recently developed pilot-scale process for the extraction of starch and by-products from wrinkled peas was used to determine the attainable yield and purity of starch and by-products from different varieties of these peas. It was shown that the starch yield mainly depends on the removal of the starch from the fibers. From the pilot scale experiments, it could be concluded that, for an industrial process, the optimum values of the total starch yield of wrinkled peas and the purity of the starch in relation to the starch content of the starch fraction would be ~80 and 98%,

respectively. The best possible value that can be achieved for mechanical separation of the protein from the starch is in the range of 1.5% protein content of the starch fraction. The protein content can be adjusted to values $\leq 0.5\%$ by alkali extraction of the starch fraction. Similar positive results cannot be achieved for the other fractions. In addition, the water flow balance in the pilot plant demonstrated that the process water flows can only be limited to an economically feasible size when applying membrane filter technology.

The extraction of starch from wrinkled peas has become the subject of special technical interest due to the high amylose content of pea starch. Unfortunately, wrinkled peas have a low starch content so that the yield of amylose per hectare for wrinkled peas is on the same scale as that for maize, wheat, and potatoes. The starch content of some German varieties can be as high as 30% with the amylose content of the starch being ~70%. Protein and fiber, two of the other main pea constituents each account for ~30% of the mass. Peas will only be a viable raw material for starch extraction if it is possible to obtain a high yield of starch and by-products with a high degree of purity. Meuser et al (1995) demonstrated in laboratory tests that extraction of starch was possible with the necessary yield and purity. The objective of the current research was to develop a pilot-scale process that gives starch yield and purity similar to that achieved in the laboratory process.

MATERIAL AND METHODS

Description of the Pilot Plant

The pilot plant was constructed so as to incorporate the same processing stages as the successful laboratory-scale process. The three main separation stages of the process are steeping of the peas, hulling the steeped peas, and disintegration of the protein adhering to the starch granules using a high-pressure homogenizer (high-pressure disintegration) (Westfalia Separator AG 1981). The pilot-plant flow shown in Fig. 1 is not continuous but is operated as a discrete batch system.

The pilot plant consisted of a rubber roller huller (Bühler-Variostuhl, CZWVE 2031), a corrugated roller crusher (Bühler-Variostuhl, CZWE 2031), a toothed-disc mill (Fryma, MZ-100), two vibrating screens (Sweco, 600 LS 24 S, 450 LS 18 S), a wine-press (Quelle), a scroll press (Vetter, P-Spezial), a decanter centrifuge (Westfalia, CA 150), a high-pressure homogenizer (APV-Gaulin, Lab 60), a hydrocyclone unit, (custom-built; cyclones Dorr-Oliver, DOXIE 5), a nozzle centrifuge (Westfalia, NA 7), a basket centrifuge (Krauss Maffei, HZ 25), a membrane filtration unit (Fluid System Division, tube module M 225), various Mohn

pumps (Netsch, 4 NE 20, 2 NE 20) and a dryer-mill (Altenburger Maschinenfabrik, Ultra-Rotor II). The machines were operated as their use required and connected by pumps and pipes. Wrinkled peas (100 kg) were processed over 12 hr in the pilot plant.

The pilot trials were made using wrinkled peas of the Markana, Salout, and Sprinter varieties (Table I). Two different experimental set-ups were selected for each variety, the difference between them being the addition of a liquid cellulase preparation (Röhm, Rohalase 7069) in a concentration of 0.1% to the steeping water used in one of the set-ups. The quantity of process water used was limited to 1,000 L/100 kg of peas.

During operation of the pilot plant, 100 kg of wrinkled peas was steeped in a 600-L tank containing 300 L of tap water at 20°C for 18 hr, after which the steeping water was discarded. The steeped peas were roughly hulled using the rubber roller huller. The distance between the rolls was set at 2.7 mm for the Markana and Sprinter varieties and at 3 mm for the Salout variety. It had previously been established that these settings would bring about the best possible hulling results (Meuser and Pahne 1993). The roll speeds were set at 200 and 38 rpm, respectively, for all trials and the throughput of peas was 280 kg/hr. The roughly hulled peas, some of which had already disintegrated into their two

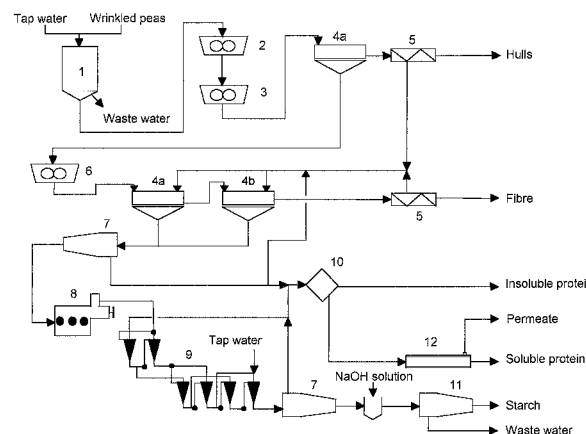


Fig. 1. Flow diagram of the pilot unit used for the processing of wrinkled peas. Equipment legend: 1) steeping tank; 2) rubber roller huller; 3) corrugated roller crusher; 4) Vibrating screens (a,b); 5) scroll press; 6) toothed disc mill; 7,11) decanter centrifuge; 8) triple-piston high-pressure homogenizer; 9) hydrocyclone unit; 10) nozzle separator; 11) membrane filtration unit.

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cotyledons and the hulls at the rough-hulling stage, were then coarsely crushed using the Variostuhl which had been converted to a corrugated roller crusher with a gap width of 1 mm, 3.2 corrugations/cm, roller speeds of 200 and 38 rpm, respectively, and a throughput of 80 kg/hr. In doing so, the cotyledons were crushed into such small fragments between the corrugated rollers that it was possible to separate them almost entirely from the hulls in the next processing stage by wet-screening, using a screen fitted with a screening surface with an aperture size of 5 mm. The hulls had remained coarse-fibered when the cotyledons were crushed so that it was relatively easy to remove them by screening.

Fresh water was used when screening the crushed material. The hulls removed were collected and dehydrated using the grape press, which had been fitted with a cotton screening cloth with an aperture size of 1 mm, until a solids content of ~30% was obtained, after which they were mill-dried. The water pressed out was subsequently used to wash the cotyledon material.

The undersize material, which was largely free from hulls and contained most of the cotyledon material in the peas, was first collected in a tank. It was then ground using the toothed-disc mill (width of gap: 0.365 mm; tool: ordinary linked teeth). This resulted in a pulpy material in which the cell structure of the cotyledons had disintegrated to such an extent that it was possible to separate most of the starch and the protein, which was insoluble under the process conditions, from the fibers forming the walls of the cells comprising the cotyledons by screening.

The two vibrating screens were set up next to each other to separate the ground material. The first screen was fitted with two screening surfaces arranged one above the other. The upper screening surface had an aperture size of 500 μm and the lower one an aperture size of 63 μm . The oversize material retained on the upper screening surface of the first screen was discharged onto the second screen which was fitted with a screening surface with an aperture size of 63 μm . Screening was done by washing the material with recycled process water sprayed onto the screening surfaces by means of spray nozzles. The quantity of washing water sprayed onto the first screen was set at 700 L/hr and that sprayed onto the second at 300 L/hr.

The fiber fraction was continuously dehydrated using the scroll press until a solids content of ~30% was obtained and subsequently mill-dried. The undersize material, which predominantly comprised only starch, insoluble protein particles, starch and protein particles, and the protein dissolved in the aqueous phase of the total material flow, as well as other soluble constituents of the peas was subjected to continuous preliminary concentration in the decanter centrifuge. The underflow of the decanter centrifuge, in which the insoluble material thus reached a concentration of 50%, was stored in a tank until needed. The overflow, with a content of soluble and insoluble materials of 3%, including insoluble protein, was pumped into a buffer tank provided for the process water.

The process water taken from the tank was in accordance with the specifications on limiting the quantity of washing water. This had been fixed by determining the maximum flow of water through the screens which was dependent on the quantity of material supplied to the screening surface per unit time. The process water was thus recycled when screening the disintegrated cotyledons and when the undersize material produced by the screens underwent preliminary concentration.

The concentrate from the underflow of the decanter centrifuge was suspended by adding process water. The suspension was adjusted to a concentration of 20% dry matter content and pumped through the valve of the triple piston high-pressure homogenizer at a throughput of 60 L/hr and a pressure of 100 MPa, any insoluble protein still adhering to the starch granules largely being separated from them in the form of protein particles. The suspension treated by high-pressure homogenization was collected in a mixing vat and subsequently pumped through the hydrocyclone unit at a throughput of 400 L/hr in countercurrent using fresh water to

separate the protein from the starch. The pressure drop in each cyclone had been set at the value most suitable for the respective cyclone stage in preliminary tests. The underflow of the hydrocyclone unit was concentrated to a solids content of 50% using the decanter centrifuge.

The overflows of the hydrocyclone unit and the decanter centrifuge were added to the overflow collected by decanting the under-size material obtained on screening the fibers and passed over the nozzle-type separator. The insoluble protein was discharged through the nozzles of the separator, resulting in a pulp with a solids content of 10% which was dried in the mill-dryer plant using the add-back process.

Aliquot parts of the centrifuge overflow, which contained a large proportion of the substances removed from the peas, were ultrafiltered in order to extract the dissolved protein. The permeate was discarded, apart from the small quantity required to determine the composition of its constituents. Aliquot parts of the retentate were either freeze- or spray-dried. In addition to this, the centrifuge overflow was heated in an experiment to extract the coagulable protein and acidified using sulfuric acid (pH 4.8) in a further experiment conducted for the same purpose.

The underflow of the decanter centrifuge was suspended with 0.1% caustic soda solution in the ratio 1:2 in a mixing vat for 30 min and then concentrated again in the decanter centrifuge. The concentrate was suspended with fresh water in the ratio 1:2 and neutralized with hydrochloric acid. The suspension was then dehydrated in the basket centrifuge until a solids content of ~50% was obtained. The damp starch cake was dried in the mill-dryer.

Statistical Analysis of Experiments and Chemical Analysis of the Wrinkled Peas and Their Intermediate and Final Products

The order in which the trials were conducted was chosen at random. The trials were duplicated experiments. In one of the two experiments for each variety, a liquid cellulase preparation was added. During a 12-hr process run of the pilot plant with each pea variety, several samples were taken in each processing stage. Error attributable to instabilities of the process were excluded by merging the samples before analysis. Aliquots then were characterized according to their chemical composition.

TABLE I
Analytical Characterization of the Wrinkled Pea Varieties
Processed in the Pilot Unit

| Variety | Starch ^a | Protein ^a | TDF ^a | Total ^a |
|----------|---------------------|----------------------|------------------|--------------------|
| Markana | 31.0 | 27.2 | 28.2 | 86.4 |
| Salout | 28.9 | 30.5 | 24.9 | 84.3 |
| Sprinter | 34.1 | 29.3 | 27.3 | 90.7 |

^a Values are %, db. Protein ($N \times 6.25$). TDF = total dietary fiber. Total = starch + protein + TDF.

TABLE II
Statistical Data for the Operation of the Pilot Unit

| Processing Stage | Measured Value | No. of Trials (n) | Mean Value (\bar{x}) ^a |
|------------------------------|--|-------------------|---------------------------------------|
| Steeping | Water absorption (g/100 g of peas) | 10 | 153.00 \pm 8.2 |
| Hulling | Percent of hulled peas | 14 | 84.70 \pm 4.7 |
| | Protein content (% db; $N \times 6.25$) | 10 | 3.10 \pm 0.2 |
| Disintegration and screening | Percent oversize material (>63 μm) | 5 | 34.30 \pm 1.5 |
| | Starch content of fiber ^b (% db) | 7 | 21.90 \pm 1.1 |
| Dewatering | Solids content of press cake ^b (% db) | 4 | 63.20 \pm 3.5 |
| Separation | Yield of insoluble protein (% db) | 5 | 30.20 \pm 1.0 |
| Raffination | Protein content of refined starch (% db, $N \times 6.25$) | 5 | 0.71 \pm 0.03 |

^a Mean value \pm standard deviation.

^b Fiber fraction including hulls.

Before the pilot-scale experiments, the reproducibility of the experimental set-up was tested by running multiple trials for each particular processing stage with different pea varieties and subjecting the analytical data of the corresponding samples to a statistical analysis. Mean values and standard deviations of the data were calculated. For the various processing stages, at least four trials were made. The results of the preliminary study are shown in Table II. The figures for the standard deviation of each processing stage were taken into account for the conclusions to be drawn from the results of the pilot-scale experiments with respect to the reproducibility of the process run.

Both the wrinkled peas and their intermediate and final products were characterized analytically to determine the purity of the products and to calculate the distribution of the constituents of the peas in the products. To this end, the water content was determined either by drying the damp material as specified in method EN ISO 1666 (DIN 1994) or by titration using method EN ISO 5381 (DIN 1994). The latter was used whenever the water content of freeze-dried products was to be determined. This applied to all residues resulting from freeze-drying steeping and process water as well as the retentates and permeates resulting from its ultrafiltration. The concentration of dissolved substances in these fluids was determined by freeze-drying a weighed portion of the fluids, taking into consideration the residual water content of the freeze-dried residues.

For titration using method EN ISO 5381, the sample (~100 mg) was weighed out in a 50-mL screw-cap jar (Schott, GL 32) and subsequently suspended in 20 mL of methanol. The jar was closed and placed in an oven heated to 80°C for 30 min to extract the water, after which it was cooled to 20°C in a waterbath. Titration used 5 mL of the suspension which was pipetted into the titration vessel of an automatic titrator (Metrohm, 702 SM Titrino).

TABLE III
Yield of the Fractions Extracted from the Wrinkled Pea Varieties Processed in the Laboratory and in the Pilot Unit

| Fraction | Dry Matter Yield ^a | | | | | | |
|------------------------------|-------------------------------|------------|-----------------|--------|------|----------|------|
| | Lab Unit | Pilot Unit | | | | | |
| | | Markana | | Salout | | Sprinter | |
| Sprinter | A ^b | B | A | B | A | B | |
| Starch | 31 | 22.2 | 20.8 | 24.1 | 23.2 | 23.3 | 22.2 |
| Insoluble protein | 12 | 7.3 | 10.4 | 10.5 | 11.4 | 11.9 | 8.0 |
| Soluble protein ^c | 24 | 21.9 | nr ^d | 22.0 | nr | 20.6 | nr |
| Hulls | 6 | 3.4 | 3.2 | 7.2 | 6.25 | 5.6 | 4.4 |
| Fiber | 27 | 36.7 | 41.2 | 31.4 | 30.6 | 34.9 | 34.9 |
| Total | 100 | 91.5 | ... | 95.2 | ... | 96.3 | ... |

^a Calculated as percent of the dry matter of the wrinkled peas.

^b A = without and B = with addition of the cellulase preparation.

^c Protein contained in the retentate.

^d Not recovered because ultrafiltration with cellulose-acetate membranes could not be applied to the cellulase activity containing process water.

TABLE IV
Analytical Characterization of the Fiber Extracted from the Wrinkled Pea Varieties Processed in the Pilot Unit

| Variety | Treatment ^a | Starch ^b | Protein ^b | TDF ^b | Total ^b |
|----------|------------------------|---------------------|----------------------|------------------|--------------------|
| Markana | A | 24.0 | 20.3 | 52.8 | 97.1 |
| | B | 24.2 | 18.2 | 54.4 | 96.8 |
| Salout | A | 17.0 | 25.9 | 54.2 | 97.1 |
| | B | 15.4 | 26.2 | 56.0 | 97.6 |
| Sprinter | A | 23.6 | 18.7 | 55.1 | 97.4 |
| | B | 20.4 | 24.7 | 52.3 | 97.4 |

^a A = without and B = with addition of the cellulase preparation.

^b Values are %, db of the fiber. Protein (N × 6.25). TDF = total dietary fiber. Total = starch + protein + TDF.

The protein and ash contents of the sample were determined in accordance with methods EN ISO 3188 (DIN 1994) and EN ISO 3593 (DIN 1994). The factor N × 6.25 was used to convert the nitrogen content of the specimen to obtain the protein content.

Determination of the starch content was based on ICC standard method 128 (ICC 1986). However, it is not possible to quantify the wrinkled pea starch by this method because part of it is not available for enzymic hydrolysis, so the method was extended to include a stage involving chemical analysis. This consisted of determining the residue of the hydrolysate retained on the filter after filtration by drying the filter. The residue was dissolved in 10 mL of 0.1N caustic soda solution. Any starch that had not been digested enzymically, and which was assumed to consist almost entirely of amylose, was dissolved in the caustic soda solution. The amylose in the solution was determined by amperometric titration with iodine in accordance with the method described by Richter et al (1968). The sum of the starch determined by means of the hydrolysate and the amylose determined in the residue was recorded as the starch content of the samples.

Although this method of determining the starch content has a certain built-in error, this must be tolerated in respect of the statements to be made here, above all because the method enables a more exact statement of the actual starch content of wrinkled peas to be made than any other. This is due to the fact that the starch is specifically determined in two analytical stages, in the first of which >90% of its mass is established by means of the glucose formed.

The starch content of the starch fraction extracted from the wrinkled peas as determined by this method was the value used to make statements on the purity of the starch which also contained protein, minerals (ash), and fibers, depending on the degree of refinement achieved.

The amylose content of the extracted starch was determined by amperometric titration using the method referred to above (Richter et al 1968). In view of the high degree of purity of the starch and the analytical error in determining both the starch and the amylose, the decision was made not to calculate the amylose content on the basis of the actual starch content.

The AOAC method of determining the total dietary fiber (Prosky et al 1985) was used to establish the content of fibrous material in the peas, which consisted essentially of the hulls and the cell walls of the cotyledons.

The values obtained for the water, starch, protein, and mineral contents of the samples were used to calculate the purity of the products and their respective yields for each variety of wrinkled pea used. In addition to this, the water flow balance of the pilot plant was calculated by determining the volumes of the material flows and the concentrations of solid material in them. The starch yield was determined by multiplying the mass yield of starch by its degree of purity, which had been divided by 100, and then dividing it by the starch content of the peas. The figure thus obtained was multiplied by 100 to give the starch yield as a percentage.

TABLE V
Yield and Purity of Starch Extracted from the Wrinkled Pea Varieties Processed in the Laboratory and in the Pilot Unit

| Variety | Treatment ^a | Starch | |
|----------|------------------------|--------------------|---------------------|
| | | Yield ^b | Purity ^c |
| Sprinter | Laboratory | 89.0 | 98.0 |
| Markana | A | 68.7 | 96.0 |
| | B | 65.1 | 97.0 |
| Salout | A | 79.2 | 95.1 |
| | B | 77.8 | 97.0 |
| Sprinter | A | 65.1 | 95.5 |
| | B | 63.1 | 97.0 |

^a A = without and B = with addition of the cellulase preparation.

^b Calculated as percent of the dry matter of the wrinkled peas.

^c Calculated as percent of the dry matter of the starch.

RESULTS AND DISCUSSION

There were no great differences in the relative compositions of the wrinkled peas used in the pilot trials (Table I), despite the fact that they differed markedly in shape, size, and color. Starch, protein and total dietary fiber each accounted for ~30% of the total mass of the peas. The relative difference between the highest and lowest starch contents (Sprinter and Salout, respectively) was 18%. The corresponding differences in the protein and total dietary fiber contents were somewhat smaller (12 and 13%, respectively). The relationships between the starch and protein contents did not reveal any intrinsic interdependence. Thus, for example, peas of the Sprinter variety had a relatively high protein content of 29.3% in spite of having a starch content of 34.1%, which was higher than that of any other variety. The relationships between protein and total dietary fiber did, however, suggest that their respective contents in wrinkled peas are in inverse proportion to each other. The three main constituents accounted for between 84.3 and 90.7% of the total mass of the peas. The remainder (that added to the other figures to give a total of 100%) consisted mainly of organic material which was free of nitrogen and largely soluble in water.

A comparison of the mass yields achieved in the laboratory-scale process, with unlimited use of water, and in the pilot process, in which the quantity of water used was limited, shows that less starch had been separated from the fibers under the conditions prevailing in the pilot process than had been the case in the laboratory-scale process (Table III). The relationship between the mass yields of insoluble and dissolved protein in the peas being compared and their total mass remained approximately the same in both processes. By contrast, the comparison shows that the mass yield of fibers was considerably higher. The determination of the starch content in the fibers demonstrated that separation of the fibers using a limited quantity of water had been unsatisfactory (Table IV). This statement would not have been very different even if the composition of the material that was inevitably lost in the pilot trials could have been taken into account. The percentage by mass of the starch remaining in the fibers was so high that it would only have improved the results for the mass yield of starch by the proportion of starch contained in the material that had been lost.

The starch yield of the peas, in relation to their starch content, ranged from 63.1 to 79.2% in the pilot trials, with and without adding cellulase to the steeping water (Table V). It was thus considerably lower than the yield of 89% achieved in the laboratory-scale process. Apart from the possibility of increasing the starch yield by reducing the losses occurring during the course of the process, which can be achieved with ease in an industrial process, any increase in the starch yield will mainly depend on better removal of the starch from the fibers.

This statement applies to each of the three varieties of pea under investigation for which the results revealed scarcely any differences as far as processibility was concerned. The higher starch

yield for the Salout variety can easily be explained by the lower starch content as compared with the other varieties.

The values obtained for the dry matter yield and the starch yield as well as the purity of the starch (Tables III and V) suggest a certain cellulase activity of the enzyme preparation as the values exhibit the same trend. As the different values obtained for the mechanical dehydration of the fibers (Table VI) reveal a similar, but considerably more significant trend, a noticeable hydrolytic effect on the constituents of the cell walls of the cotyledons cannot be ruled out at least, thus providing an explanation of the differences observed both in the starch yield and purity and in the dehydration of the fibers. However, as far as interpreting the test results is concerned, it was far more important to establish that the trials, which were done in a randomly selected order, had produced repeatable results that were practically identical for each pea variety.

The starch remaining in the hulls accounted for <2% of the total starch content (Tables VI and VII). From this it follows that, after taking into account the percentage by mass of the hulls, which is ~6% for wrinkled peas, the crushed cotyledons had been separated almost entirely from the hulls by screening, without too many hull constituents ending up in the undersize material at the same time. It is for this reason that it would also be possible to apply the screening process used to separate the hulls from the cotyledons on an industrial scale even though it would seem more appropriate to use hydrocyclones to separate the solids. It was not possible to do this in the pilot plant as the material flows passing through the hydrocyclone suitably dimensioned for the separation process were too large.

Separating the hulls from the cotyledons to the greatest extent possible is desirable for two reasons. One is that the quantity of material on the screening surface is reduced when the fibers are subsequently separated from the starch and protein. The other pertains to the possible use of the products. In this regard, it is important to draw attention to the other physical properties of the cotyledon fibers such as their capacity to bind water which is much greater than that of the hulls enclosing them.

In each trial, the fibers bound between 16.3 and 32.1% of the total starch (Table VI). The fibers contained between 17.0 and 24.2% starch (Table IV). The starch content of the fibers is relatively low compared with similar products, such as potato or cassava pulp (Meuser et al 1987). It can be derived from this that an excellent result with regard to the degree to which the starch could be removed had already been achieved by means of the grinding process used and that it would not have been possible to improve it noticeably without causing any negative consequences for the purity of the starch. Further grinding of the cells of the cotyledons with the toothed-disc mill would, above all, have resulted in a larger proportion of fibers ending up in the undersize material. This would have impaired the effectiveness of the centrifugal separation processes subsequently used to remove and refine the starch.

The sum of the masses determined analytically in the extracted starch, obtained by adding together the starch, protein, and ash

TABLE VI
Influence of the Wrinkled Pea Varieties on the Starch Content and the Water Binding Capacity of the Fiber Fraction

| Variety | Treatment ^a | Loss in Starch Yield ^b | | Water Bound to Fiber ^c | |
|----------|------------------------|-----------------------------------|-------|-----------------------------------|--------------------|
| | | Hulls | Fiber | Before ^d | After ^d |
| Markana | A | 0.4 | 28.4 | 987 | 269 |
| | B | 0.4 | 32.1 | 1,104 | 207 |
| Salout | A | 2.0 | 18.5 | 1,166 | 438 |
| | B | 1.5 | 16.3 | 793 | 283 |
| Sprinter | A | 1.6 | 24.2 | 920 | 372 |
| | B | 1.3 | 25.3 | 880 | 264 |

^a A = without and B = with addition of the cellulase preparation.

^b Calculated as percent of the starch content of the wrinkled peas.

^c Calculated as kg of water/100 kg of dry matter.

^d Before and after mechanical treatment.

TABLE VII
Analytical Characterization of the Hulls Extracted from the Wrinkled Pea Varieties Processed in the Pilot Unit

| Variety | Treatment ^a | Starch ^b | Protein ^b | TDF ^b | Total ^b |
|----------|------------------------|---------------------|----------------------|------------------|--------------------|
| Markana | A | 3.4 | 5.1 | 88.9 | 97.4 |
| | B | 3.4 | 7.1 | 89.2 | 99.7 |
| Salout | A | 8.2 | 11.5 | 76.6 | 96.3 |
| | B | 7.5 | 8.8 | 80.7 | 97.0 |
| Sprinter | A | 11.2 | 9.6 | 77.1 | 97.9 |
| | B | 10.6 | 9.7 | 79.6 | 99.9 |

^a A = without and B = with addition of the cellulase preparation

^b Values are %, db of the hulls. Protein (N × 6.25). TDF = total dietary fiber. Total = starch + protein + TDF.

TABLE VIII
Analytical Characterization of the Starch Extracted from the Wrinkled Pea Varieties Processed in the Pilot Unit

| Variety | Treatment ^a | Starch ^b | Amylose ^b | Protein ^b | Ash ^b | Total ^b |
|----------|------------------------|---------------------|----------------------|----------------------|------------------|--------------------|
| Markana | A | 96.0 | 68.6 | 1.1 | 0.515 | 97.5 |
| | B | 97.0 | 71.6 | 1.0 | 0.484 | 98.5 |
| Salout | A | 95.1 | 65.6 | 0.8 | 0.207 | 96.1 |
| | B | 97.0 | 67.5 | 0.6 | 0.235 | 97.8 |
| Sprinter | A | 95.5 | 67.1 | 0.7 | 0.401 | 96.6 |
| | B | 97.0 | 68.4 | 1.2 | 0.454 | 98.6 |

^a A = without and B = with addition of the cellulase preparation.

^b Values are %, db of the starch. Protein (N × 6.25). TDF = total dietary fiber. Total = starch + protein + TDF.

TABLE IX
Analytical Characterization of the Insoluble Protein Extracted from the Wrinkled Pea Varieties Processed in the Pilot Unit

| Variety | Treatment ^a | Protein ^b | Starch ^b |
|----------|------------------------|----------------------|---------------------|
| Markana | A | 75.6 | 7.5 |
| | B | 81.7 | 3.0 |
| Salout | A | 82.9 | 2.2 |
| | B | 82.7 | 0.6 |
| Sprinter | A | 74.8 | 8.1 |
| | B | 80.5 | 6.6 |

^a A = without and B = with addition of the cellulase preparation.

^b Values are %, db of the insoluble protein. Protein (N × 6.25).

contents, and for which a mean value of 97.5% has been calculated, demonstrates that the starch contained only a small proportion of fibers and other substances (Table VIII). It will not be possible to reduce the percentage of fibers any further in an industrial process either as, even in the wet-milling process, the cell walls of the cotyledons are broken into such small fragments that they pass through the screen and cannot subsequently be separated from the starch. This risk is, of course, much greater in all dry-milling processes used to process the raw material (Gabriel-Blanke et al 1990).

The purity of the starch for which on average a value of 96.2% was calculated by the method used to determine the starch content of the starch fraction (Table V), was, however, only achieved after subjecting the starch refined in the hydrocyclone unit to the alkali extraction stage used in the process. Before this, the protein content of the starch was ~1.5%, which can be regarded as the best possible value that can be achieved for mechanical separation of the protein from the starch. Further refinement of the starch by mechanical means is not possible as the uneven surfaces and the irregular shape of the starch granules are an obstacle to the mechanical removal and separation of the insoluble protein.

We had already demonstrated both the effectiveness and the limits of using a high-pressure homogenizer to separate protein from starch in our previous work (Meuser et al 1993, 1995). The results obtained for the separation of starch from protein after high-pressure homogenization, shown in Fig. 2 by the differences in the color of the two phases of the sediment, are an impressive demonstration of the effectiveness of this processing stage and of the necessity of applying it to the undersize material obtained when removing the fibers. Although it would also have been possible to use high-pressure homogenization to regrind the milled cotyledons, this would have had no advantages for the subsequent treatment of the ground material produced using the toothed-disk mill. It would instead have been coupled with the disadvantage that the fibers would have been ground too finely. This would have led to an increase in the fiber content of the starch (Pahne and Meuser 1995).

The protein content of the starch, which was on average 0.9% after alkali extraction (Table VIII), can be reduced even further by the same method. To do so, all that is required is to treat the starch with caustic soda solution more intensively than was the case in



Fig. 2. Centrifugally separated wrinkled pea starch from the underflow of the hydrocyclones used for starch refining. Protein layer content = 1.5% dry matter.

these trials. In other trials, we have achieved protein contents of ~0.5%.

A lower protein content is essential for many of the intended industrial applications of these starches, for example their use in the manufacture of high-quality films. However, applications of this nature depend not only on the purity of the starch but above all on its amylose content, which ranged between 65 and 70% for the varieties of wrinkled peas investigated here. There were only small differences in the amylose contents of the extracted starches. It should be noted that recent research has indicated that an amylose content of ~70% is still too low for the film-forming properties of the amylose to be exploited when using these types of starch (W. Burchard, *personal communication*).

A summary of the problems involved in separating the starch from the wet-ground cotyledon material and its extraction and refinement after high-pressure homogenization of the undersize fraction obtained on screening the fibers enables us to conclude that, in an industrial process, the optimum values of the total starch yield of wrinkled peas and the purity of the starch in relation to the starch content of the starch fraction would be ~80 and 98%, respectively. The protein content of the starch, which is of crucial importance for its industrial application, can be adjusted to values ≤0.5% by alkali extraction of the starch fraction.

Similar positive results were not achieved for the other fractions. The distribution of protein in the other fractions proved to be a problem as regards its extraction. On average, it was possible to yield ~10% insoluble protein by centrifugal separation with a solids content of only 10% and an average protein content of 78% from the total mass of the peas used (Tables III and IX). This proportion of the mass was, however, only 25% of the total protein of the peas. The protein had a slimy viscosity and was difficult to dry. Although it was possible to spray-dry it, we preferred mill-drying using the add-back process for economic reasons, it being possible to dry the product just as gently as by spray-drying. The description of the functional and nutritional properties of this fraction is currently the subject of other research work.

A proportion of the total protein similar in size to the insoluble protein fraction remained in the fibers in which it was mainly present as insoluble protein (Table IV). For this reason, it was just as impossible to separate the protein from the fiber fraction as from the starch. In addition to this, part of the dissolved protein remained in the fibers owing to the recycling of the process water and the marked capacity of the fibers to bind water.

On average, it was only possible to obtain a dry matter content of 22.3% (without cellulase) or 28.1% (with cellulase) for the fibers in the trials performed using the scroll press (Table VI). This means that the protein dissolved in the water bound by the fibers also remained part of the dried fiber fraction after the water

TABLE X
Yield of Protein Fractions and Their Protein Content Extracted
by Different Methods from Wrinkled Pea Variety Markana
Processed in the Pilot Unit

| Criterion | Soluble Protein | | | |
|------------------------------------|--------------------------------|--------------------------------------|-------------------------------|-----------------|
| | Insoluble Protein ^a | Isoelectric Coagulation ^a | Heat Coagulation ^a | Ultrafiltration |
| Dry matter (%) | 10.0 | 21.3 | 17.2 | 10.0 |
| Protein ^b | 76.1 | 69.6 | 70.4 | 50.9 |
| Yield ^c | 7.3 | 10.3 | 10.5 | 17.7 |
| Precipitate/retentate ^c | ... | 67.1 | 70.0 | 81.8 |
| Protein yield ^c | 20.1 | 26.2 | 27.4 | 32.0 |

^a Separated by centrifugation.

^b Calculated as % of the dry matter content of the protein fraction.

^c Calculated as % of the pea dry matter.

^d Calculated as % of the protein content of the process water (retentate).

^e Calculated as % of the pea protein content.

had been expressed. The resultant fiber composition (Table IV) meant that the fibers were only suitable for use as animal feed. There are hardly any industrial applications that can be considered for these fibers owing to their high protein content although their high water-binding capacity is of interest. Removing the protein and, where necessary, the starch from the fibers requires additional processing stages. Attention is drawn to this here without discussing the technical possibilities any further.

Extraction of the insoluble substances from the wrinkled peas can thus be regarded as having been resolved technically. The same cannot be said of the extraction of the soluble substances as the processes used for this purpose have proved to be so costly that their application in the extraction of usable products is questionable for economic reasons. Nevertheless, the processes have been used in order to obtain a conclusive solution to the task in hand.

In the pilot trials, the dissolved material had a concentration of 3% dry matter which consisted of 43% protein or other substances containing nitrogen. The results given in Table X show that the percentage of dissolved protein that can be obtained by means of isoelectric and thermal precipitation is equivalent to ~70% of the substances containing nitrogen in the process water. This constitutes an additional protein yield of between 25-30% of the total protein under the trial conditions and in relation to the insoluble protein fraction obtained. The differences between the mass yields obtained in the two precipitation processes were small, as were the differences between the protein contents of the precipitates. These were 67.1 and 70.0% (for isoelectric and thermal precipitation, respectively) and were thus on the same scale as the protein content of the insoluble protein.

It was possible to increase the concentration of dissolved protein in the process water flow by ultrafiltration. The protein content of the dry mass of the retentate thus rose to 50.9%. Spray-drying the retentate resulted in a slightly higher protein yield than precipitation (32.0% compared with ≤27.4%). The proteins in the retentate also retained their native properties to a large extent when spray-dried, whereas the precipitated proteins had lost them entirely.

It should be noted that it will be possible to increase the protein content of the retentate still further by using different membranes. However, this will be coupled with the disadvantage that the permeate will become contaminated with a larger proportion of organic substances, making its further treatment or disposal more difficult. The scale of the industrial process concerned and other boundary conditions, such as the site, will be crucial in deciding which course to take in this matter.

It is already becoming apparent that the use of ultrafiltration to achieve the highest possible recovery of dissolved protein can only be considered if special importance comes to be attached to its functional properties, taking its possible uses in industry into account. Otherwise, limiting the process water flow by hyperfil-

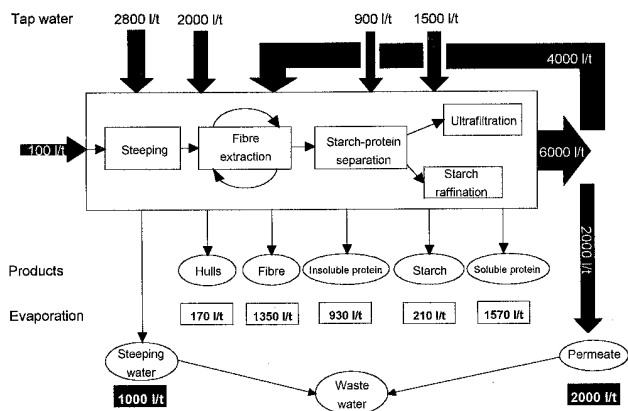


Fig. 3. Balance of the process water streams of the pilot unit calculated on the basis of the processing of one metric ton (t) of wrinkled peas.

tration instead of ultrafiltration will have to be considered because the permeate that cannot be returned to the process water cycle could then easily be disposed of as waste water. This process already constitutes state-of-the-art technology in the extraction of starch from potatoes (Meuser et al 1982).

The water flow balance in the pilot plant, which is shown in Fig. 3 and which has been included in the results calculated for the ultrafiltration plant, demonstrates that the process water flows cannot be limited to a scale that would be economic in an industrial process based on the pilot process that has been developed unless membrane filter technology is used.

In the example chosen here, it has been calculated that using ultrafiltration to recirculate part of the process water in the form of the permeate would have meant evaporating 4,230 L of water per ton of peas to dry the hulls, fibers, insoluble protein, and starch as well as to extract the soluble protein as dry material. In addition to this, ~3,000 L of waste water would have been produced per ton of peas, which can be broken down into 1,000 L of steeping water and 2,000 L of permeate. The waste water contained 2% organic material under the conditions of the pilot trials. This figure would be even higher if the cut-off of the ultrafiltration membrane were altered to allow recovery of dissolved protein with a higher protein content.

The problem of disposing of highly contaminated waste water could, however, be avoided by using hyperfiltration instead of ultrafiltration. It would then be possible to return all the permeate to the process water cycle. Most of the protein in the residue could then be precipitated thermally and the remaining material flow of dissolved substances could be concentrated by evaporation. This method of recovering dissolved substances from the process water in wrinkled pea starch plants would correspond to that used in potato starch plants (Meuser et al 1982).

A calculation of the cost of applying the process, based on the results achieved to date, will be given in another report. It will enable the risk involved in putting the process into practice on an industrial scale to be estimated in relation to the possible uses of the products.

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