Fractionation of pea flour with pilot scale sieving.

I. Physical and chemical characteristics of pea seed fractions

Chiraze Maaroufi\textsuperscript{a,}\textsuperscript{*}, J.-P. Melcion\textsuperscript{b}, F. de Monredon\textsuperscript{b}, B. Giboulot\textsuperscript{b}, D. Guibert\textsuperscript{b}, Marie-Pierre Le Guen\textsuperscript{c}

\textsuperscript{a}Institut National de la Recherche Agronomique (INRA), Laboratoire de Nutrition et d’Alimentation, 16, rue Claude Bernard, 75231 Paris cedex 05, France
\textsuperscript{b}Institut National de la Recherche Agronomique (INRA), BP 71627–44316 Nantes cedex 03, France
\textsuperscript{c}G.I.E. EURETEC II, 12, avenue Georges V, 75008 Paris, France

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Abstract

Facing a lack of methods and criteria to characterize feed particle properties, a grinding–fractionation process was applied to pea seeds to obtain different particle size fractions, physically and chemically defined. Seeds were first ground with a hammermill on a 4 mm screen. The flour obtained was divided into nine granulometric fractions with a sieving classifier using a large range of mesh openings from 2.5 to 0.122 mm. To interpret the results, hulls and kernels were separated with an air classification device and ground on a 0.5 mm screen. Classification efficiency of the granulometric fractions was checked. Each fraction was characterized by chemical composition (crude protein — CP, starch, crude fiber — CF, water-insoluble cell walls — WICW and ash) and also physically by particle size distribution, median diameter ($d_{50}$), a dispersion parameter (logarithmic standard deviation), specific surface area (SPSA), total porous volume (TPV) using a nitrogen adsorption apparatus, bulk density and apparent density (DENap. — helium multipycnometer device). The fractionation process led to well-differentiated fractions. Great differences in chemical composition of the granulometric fractions were noted. The smaller the size, the higher the contents of both CP (from 21.8 and 25.7% DM) and starch (from 47.9 to 62.2% DM), and the lower the cell wall contents (CF from 0.9 to 11.8% DM and WICW from 5.9 to 22.4% DM). Within a range of $d_{50}$ (from 59 to 2720 \textmu m), the SPSA (from 0.102 to 0.418 m$^2$ g$^{-1}$) increased with particle fineness, and seemed to be an interesting criterion to characterize specifically each fraction. The TPV values were low (from 0.1 to 0.8 mm$^3$ g$^{-1}$) and the densities values showed irregular evolution.

* Corresponding author. Tel.: +33-1-44-08-17-55; fax: +33-1-44-08-18-53.

E-mail addresses: maaroufi@inapg.inra.fr (C. Maaroufi), melcion@nantes.inra.fr (J.-P. Melcion)
on the granulometric distribution (bulk density from 530 to 666 g dm⁻³ and DENap. from 1.427 to 1.465 g cm⁻³). A general significant evolution (p<0.05) of chemical composition according to the geometry of the particles (d₅₀ and SPSA) appeared. The chemical and physical characteristics of the size fractions seemed to reflect the different comminution laws followed by the hulls and the kernels, while grinding pea seeds. This phenomenon would have led to a physical separation of the botanical constituents of the pea seed, with coarse fractions in which hulls are accumulated together with a majority of parietal constituents, finer fractions enriched with kernels and cellular constituents and the smallest fraction mainly composed of starch granules.

1. Introduction

The grinding process has often been applied to feeds because of its nutritional and zootechnical impact. This effect is explained by an increase of the ratio surface/volume of the substrate and by the breakdown of cell walls structures, leading to an increased accessibility to cellular contents (Brennan et al., 1981).

As a consequence of the heterogeneous tissue structure (Hansen and Stewart, 1965) of materials of vegetal origin, grinding has been demonstrated to produce individual heterogeneous particles in size and chemical composition (Emanuele and Staples, 1988; Bertrand et al., 1998). Methods and criteria proposed to characterize particle properties are scarce and more or less adapted to allow a good interpretation of the observed results in animal feeding. Physical properties such as particle sizes, surface or porosity, may explain the nutritional behaviour of the feed as well as the chemical properties. So, a more complete physical and chemical analysis has to be carried out on the feed particles to better characterise the feed material and to avoid misinterpretations.

The aim of the study is to check to what extent the classification of pea flour (Pisum sativum L.) has been effective in size and to determine the physical and chemical characteristics of resulting fractions. From the relationships between these characteristics, an attempt was made to improve the definition of a feed flour.

2. Material and methods

Smooth pea seeds (Pisum sativum L., Madria cultivar) were provided by ITCF (Institut Technique des Céréales et des Fourrages, 91350 Boigneville, France). They were harvested in 1993 and stored at room temperature during 1 year before use. The peas had a weight of 204 g for 1000 seeds and contained a low level of trypsin unit inhibitors of 2.3 TUI mg⁻¹ DM.

2.1. Sample preparation

The seeds were first ground with a pilot scale hammermill Forplex A0 type (92106 Boulogne-Billancourt, France) using a circular 4-mm screen. The peripheral speed of the
rotor was 65 m s\(^{-1}\). A 4-mm aperture size screen was chosen to obtain a large range of particle size distribution. Then, the ground material was divided into nine particle size classes in a 100–3000 \(\mu\)m range with a Sweco continuous dry sieving classifier (Parc Industriel, 1400 Nivelles, Belgium) using a range of square mesh openings of 2.5, 1.94, 1.52, 1.04, 0.76, 0.5, 0.23, 0.122 mm. These dimensions were considered as identifiers of the fractions (Table 1). The hulls were removed from the kernels in an aliquot sample of a coarse fraction (F1.94) with a home-made air classification device. Hulls and kernels were ground with an impact grinder Cyclotec (Tecator AB., PO Box 70, 26301 Höganaäs, Sweden) using a 0.5 mm screen for analytical determinations.

2.2. Physical characterization

2.2.1. Particle size

Particle size was determined on a duplicate 100-g sample with a Bühler laboratory siever MLU 300 (Bühler-Miag, 9240 Uzwil, Switzerland) using a set of woven-wire cloth sieves having a diameter of 26 cm (12 sieves maximum). The sieve openings were chosen according to AFNOR specifications NF X11-501 (AFNOR NF X11-501, 1970) which recommend a geometrical progression of screen sizes in a 3150–3180 \(\mu\)m range. The sieving time was 15 min.

The particle size distribution of very fine flours as ground hulls and kernels fractions was determined using laser (Helium-Neon: wavelength 633 nm) diffraction according to AFNOR standard NF X11-666 (AFNOR Standard X11-666, 1984). It was performed with a Malvern Mastersizer IP granulometer (Malvern Instruments S.A, 91893 Orsay cedex, France) which was equipped with a lens of 1000-mm focal length. Diffraction pattern analysis was carried out in air on a stream of dry powder. Determinations were repeated twice.

The size of particles was reported in terms of median particle diameter \(d_{50}\) (in \(\mu\)m) and of logarithmic standard deviation (S.D.) by weight (AFNOR NF X11-635, 1985; AFNOR NF X11-636, 1985). The median size is the midpoint of the distribution. At this point, half of the particle are smaller and half are larger. The logarithmic (or geometric) standard deviation was considered as an expression of the spread of particle size distribution.
With the laser particle size analysis, the repeatability on the $d_{50}$ value of the studied granulometric fractions was assessed by calculating a coefficient of variation on measures tested on the same day with the same person. It was estimated to be 1.3%.

2.2.2. Classification efficiency

The feed flow which is classified on a screen of size $X_i$ is theoretically divided into one coarse resulting flow (coarser than $X_i$) and one fine flow (equal or finer than $X_i$). However actually, a part of the finer flow is contaminating the coarser flow and inversely. The classification curve or Tromp curves (Tromp, 1937) are known to represent the classifier performances. They are calculated for each $X_i$ by means of mechanical sieving of the obtained classified fractions. The features of the classification curves (adapted from Prasher, 1986) which are of interest are: (a) the limits of separation; (b) the cut size (or size modulus), which corresponds to 50% of the feed passing to the coarse stream. It is therefore that size which has equal probability of passing to either coarse of fine streams; (c) the sharpness of cut (or distribution modulus) which refers to the overlap size range between the limits of separation.

2.2.3. Apparent density and bulk density

The apparent density (g cm$^{-3}$) is the density of the material including closed and inaccessible pores (Rouquerol et al., 1994). It is given by the ratio between the sample mass and the volume of the solid material, volume of the internal pores included. The volume of the solid particles was measured with a Quantachrome multipycnometer (5, Aerial Way, Syosset, NY, USA) using helium as a fluid medium. The measurement was repeated three times. The coefficient of variation calculated on the same day with the same person on the pea fractions indicated a repeatability of $\approx 0.8\%$.

The bulk density (g dm$^{-3}$) is the density including pores and interparticle voids. It is defined by the ratio between the mass of the sample which is needed to fill one unitary volume (usually 1 dm$^3$). It was measured using a Nilema-litre (Tripette & Renaud, 75038 Paris, France). The measurement was repeated three times. The coefficient of variation calculated on the same day with the same person on the pea fractions was of $\approx 0.6\%$.

2.2.4. Surface area and total porous volume

The specific surface area of the particles (SPSA in m$^2$ g$^{-1}$) was determined by measuring the adsorption of nitrogen at 77°C according to the B.E.T. principle (AFNOR NF X11-620, 1994) and using a Micromeritics apparatus type Gemini III (Norcross, Georgia, USA). The measurement was repeated twice after out gassing of the samples for 24 h at 60°C. The reproducibility measured on a reference sample of alumina (alumina CRM 169) on different days was $0.104 \pm 0.012$ m$^2$ g$^{-1}$, i.e. a variation coefficient of ca. 11.5%.

The total porous volume (TPV, cm$^3$ g$^{-1}$) was determined by using the same apparatus. It is calculated as $V_a/D$, where $V_a$ is the volume of adsorbed nitrogen and $D$ a conversion ratio liquid/gas of the adsorbate ($D_{\text{nitrogen}}=1.5458 \times 10^{-3}$). The repeatability on the granulometric fractions was however low, as the coefficient of variation calculated on the same day with the same person was approximately of 25%.
Assuming a cylindrical shape of all the pores existing on the external surface of the particles, an average pore radius \( r \) is calculated:

\[
r = 2 \times \frac{TPV}{SPSA}
\]

Assuming a spherical shape of the particles, the surface area was also calculated (SPSA_{calc}) from the particle size distribution data \( (d_{50} \text{ and S.D.}) \) using the following relationship:

\[
SPSA_{calc} = \frac{6}{\text{apparent density} \times d_{50}} \times \exp \left(0.5(\ln \text{S.D.})^2\right)
\]

2.3. Chemical characterization

Moisture, crude protein (CP) and crude fiber (CF) contents were determined according to official methods (AFNOR, 1985). Starch (STA) was measured using the polarimetric procedure (Ewers method). Water-insoluble cell wall (WICW) was determined according to AFNOR V18-111 (1989).

2.4. Statistical analyses

The SAS package (SAS, 1995) was used for statistical calculations. A particle size effect was studied by analysis of variance on the criteria measured in the laboratory (densities and SPSA). Regression analysis was performed to study the relationship between couples of data, and principal component analysis (PCA) has been used for a more general interpretation in a multi-dimensional space (between-classes correlations).

3. Results

3.1. Quality of the fractionation process

The sharpness of cut (Table 1) varied from 1.10 to 1.18. It was higher for the F0.5 and F1.52 fractions and traduced its lower level of ‘granulometric purity’. Concurrently, the values of cut size appeared generally distant from the size of the sieve openings, on which the fractionation was done. A pollution of the coarser fraction by the finer particles from the below fraction could explain this result.

3.2. Physical characteristics

3.2.1. Granulometry

The whole pea flour presented a median particle diameter of 0.669 mm and a logarithmic standard deviation (S.D.) of 1.99. The median particle diameter \( d_{50} \) varied largely among fractions (Table 1). However, some of them (F1.94 to F0.76) presented
values out of the interval delimited by the size aperture of the sieves used to prepare the fractions (e.g. $d_{50}$=0.934 mm for the fraction 1.52–1.04 mm). The logarithmic standard deviation indicated narrow distributions around the $d_{50}$ (1.13–1.30), except for the throughs (3.16). The hulls and the kernels fractions came from a finer grinding than the pea granulometric fractions (0.5 mm versus 4 mm screen). In comparison with the kernels particles, the hulls particles were coarser and less variable around their mean size (Table 2). The particle size distribution (Fig. 1) of the kernels showed a bimodal distribution, whose first peak could mainly be identified as starch granules (size of ca. 40 μm).

It should be noted that the hulls and kernels analysis can improve the interpretation of the results for the different granulometric fractions. However, a direct comparison between these fractions is not valid as they were derived from two levels of grinding.

### 3.2.2. Apparent density and bulk density

The apparent density (DENap.) of the whole pea flour was of 1.432 g cm$^{-3}$ (Table 2). It ranged for the granulometric fractions from 1.427 to 1.465 g cm$^{-3}$. The bulk density (DENbu.) of the fractions ranged from 530 to 666 g dm$^{-3}$ (Table 2). The whole flour presented a higher value (715 g dm$^{-3}$), that can be explained by its high heterogeneity of particle size and form, leading to a probable decrease of the interparticle voids. Significant differences ($p<0.001$, $n=30$, RSD DENap.=$0.0014$, RSD DENbu.=$7.4$) appeared between fractions, but the evolution of these parameters according to the particle size distribution was irregular.
3.2.3. Surface area and total porous volume

The measured surface area values varied from 0.102 to 0.418 m$^2$ g$^{-1}$ (Table 2). They increased as the particle size decreased, with a stronger effect for the two finer fractions F0.23 and F0.122 and particularly for the throughs ($p<0.01$, $n=20$, RSD=0.048). Owing to a finer grinding, hulls and kernels particles presented obviously higher SPSA values (0.409 and 0.533 m$^2$ g$^{-1}$, respectively).

The comparison with the calculated surface area allows an assessment of the distortion coming from the spherical shape hypothesis used in this calculation (Table 2). The closer the ratio ‘SPSA/SPSAcalc.’ (SPSAra.) is to the value 1, the more the particles present a smooth surface and/or spherical form. This is illustrated by the hulls and kernels results, which show that this hypothesis is obviously false for the hulls. For the granulometric fractions, this ratio was getting closer to 1 as the particles became finer, mainly for F0.122 and the throughs. This confirms for these last fractions, their smoother surface.

The total porous volume values ranged between 0.1 and 0.8 mm$^3$ g$^{-1}$ (Table 2). They increased as the particle size decreased. Hulls and kernels particles had higher values (3.2 and 1.1 mm$^3$ g$^{-1}$, respectively). This was probably due to the finer grinding of these fractions. The difference between hulls and kernels could be attributable to the higher hulls SPSA. The porosity evolved in the same way as SPSA. Indeed, the surface provided by particle pores was included in the SPSA evaluation.

3.3. Chemical characteristics

The fractionation led to marked differences in chemical composition of the granulometric fractions (Table 3). The smaller the size of classified particles, the higher the contents of both CP (from 21.8 to 25.7% DM) and starch (from 47.9 to 62.2% DM) and the lower the cell wall contents (CF from 0.9 to 11.8% DM and WICW from 5.9 to
22.4% DM). This evolution was not regular. In particular, the cell wall content went through maximal values for the F1.52 and F1.04 fractions, whereas the cellular constituents contents were minimal. Moreover, a step in the contents evolution appeared for the finer fractions under F1.04, with a marked drop of cell wall content and an increase of the cellular constituents contents. The throughs contained much more starch and less CP than the fraction immediately above (F0.122). Logically, starch and CP were concentrated in kernels, and structural carbohydrates in hulls.

3.4. Cumulative proportions of the different characteristics according to the size of particles

The cumulated percentages on each size class for the various characteristics are presented in Figs. 2 and 3. The curves had nearly the same shape and their slopes differed more or less from each other. This can reflect the differential separation of the various parameters (more or less important quantities for a given size of particle). For the fraction F0.23 (d_{50} of ca. 335 μm), the cumulated proportion of dry matter retained on this sieve was ca. 31%. At this size of particle, 40–50% of the surface area was located in the fine part of the feed, but only ca. 30% of the density (Fig. 2). For the chemical parameters (Fig. 3), the cytoplasmic constituents are separated more rapidly (higher slopes) than the parietal ones (respectively, ca. 30% and <20% found in the fine particles).

3.5. Multivariate analysis

3.5.1. Correlation analysis

3.5.1.1. Correlation between chemical criteria. The chemical carbohydrate parameters STA, CF, WICW were well-correlated, while the CP appeared mainly linked to the CF
alone ($|\rho|>0.7$) (Table 4). When the throughs was removed from the analysis, all correlations became significant (Table 5). WICW and CF (hulls) were opposed to STA and CP (kernels). The link with moisture could be explained by a preferential adsorption of water by cell walls ($\rho$ moisture/WICW=0.7). This phenomenon was underlined by

Fig. 2. Physical parameters (cumulated% — DENbu, bulk density; DENap, apparent density; SPSA, specific surface area; SPSAra, SPSA calculated/SPSA measured; TPV, total porous volume; DM, dry matter).

Fig. 3. Chemical composition (cumulated% — CP, crude protein; WICW, water-insoluble cell walls; DM, dry matter).
Table 4
Correlations between physical and chemical characteristics a

<table>
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<th>Substrate</th>
<th>Crude protein</th>
<th>Crude fiber</th>
<th>Starch</th>
<th>Cell walls</th>
<th>Raw ash</th>
<th>Moisture</th>
<th>Bulk density</th>
<th>Apparent density</th>
<th>SPSAb</th>
<th>SPSAra. c</th>
<th>TPVd</th>
<th>Median diameter</th>
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<tr>
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<td>-0.66</td>
<td>-0.02</td>
<td>-0.70</td>
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a Correlation coefficients/prob.>|IR| under H0: R=0/N=10. The numbers in italics represent the significant correlations at the level of 5% (|IR|: 0.63).
b Measured surface area.
c Measured surface area/calculated surface area.
d Total porous volume.
Table 5
Correlations between physical and chemical characteristics (without throughs)\(^a\)

<table>
<thead>
<tr>
<th></th>
<th>Crude protein</th>
<th>Crude fiber</th>
<th>Starch</th>
<th>Cell walls</th>
<th>Raw ash</th>
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<th>Apparent density</th>
<th>SPSA(^b)</th>
<th>SPSAra.(^c)</th>
<th>TPV(^d)</th>
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</tr>
<tr>
<td>Median diameter</td>
<td>-0.80</td>
<td>0.70</td>
<td>-0.62</td>
<td>0.41</td>
<td>-0.73</td>
<td>0.13</td>
<td>-0.41</td>
<td>-0.11</td>
<td>-0.69</td>
<td>-0.73</td>
<td>-0.85</td>
<td>1.00</td>
</tr>
</tbody>
</table>

\(^a\) Correlation coefficients/prob.>|R| under H0: \(\rho=0/N=10\). The numbers in italics represent the significant correlations at the level of 5% (|R|: 0.67).

\(^b\) Measured surface area.

\(^c\) Measured surface area/calculated surface area.

\(^d\) Total porous volume.
Hooper and Welch (1985), assessing the influence of the fibrous structure of legume forages in the water molecules trap.

3.5.1.2. Correlation between physical criteria. There were less significant correlations between the physical parameters (Table 4). The SPSA or the ratio SPSAcalc./SPSA (SPSAra.) were positively linked to the TPV ($\rho>0.8$) and the SPSA to the apparent density (DENap.) ($\rho=0.7$). The mean particle size ($d_{50}$) was linked significantly with the TPV alone ($\rho=−0.8$).

The throughs almost always presented an extreme position in the relationship between variables. This is due to high values of SPSA, SPSAra., TPV and a very low value of $d_{50}$. With the withdrawal of this fraction from the analysis (Table 5), the density values were not correlated to other variates, but the ‘morphologic’ criteria (SPSA, SPSAra., TPV and $d_{50}$) were significantly and mutually correlated. As we could expect, the SPSA of the much lower $d_{50}$ fractions was higher. The fact that TPV also appeared higher for the small particles could be explained by more surface cracks for these fractions.

Finally, moisture was linked negatively to the SPSA criteria, although the measurement was done on dry samples after the out gassing (Table 4). This could be due to a laboratory artefact: the measured surface (SPSA) could be directly linked to the water withdrawal, because of an effect of exposed surface to exchanges with the air while grinding or fractionating. With the smallest particles (high SPSA), these processes would lead to a reduction of the interstitial spaces that are responsible for the water holding capacity (Robertson and Eastwood, 1981).

3.5.1.3. Correlation between physical and chemical criteria. We notice a general evolution of the chemical composition (CP, STA, CF, WICW) according to the geometry of the particles ($d_{50}$, SPSA, SPSAra.) ($\rho>0.6$, Table 4), except that the $d_{50}$ was not correlated to the WICW and the surface parameters were not correlated to the CP. But, by removing the throughs from the data, the surface parameters and the CP became positively correlated ($\rho>0.6$, Table 5).

3.5.2. Principal component analysis

The two first components (10 variables and 10 observations) accounted for 81% of the total variance (Fig. 4). The first axis was mainly linked on one hand, to the fibre contents of the particles, to their median diameter and to a lesser extent to the moisture content, and on the other hand, to the starch content and the SPSA parameters. The second axis was more associated with a protein enrichment. The apparent density is not well-represented on the correlation circle.

The projection of the granulometric fractions on the components (Fig. 4) indicates a clustering of the samples into three groups:

1. a group of coarse fractions (F2.5, F1.94, F1.52, F1.04), that contain more cell walls and less starch, that have a higher moisture level, an intermediary or even low value for F1.94 of bulk density and a low value of SPSA;
2. a group of intermediate to fine size fractions, enriched with CP and starch, poorer with cell walls, and higher in bulk density;
Fig. 4. Correlation circle for the chemical and physical characteristics of the pea fractions (CP, crude protein; CF, crude fiber; WICW, water-insoluble cell walls; DENbu, bulk density; DENap, apparent density; SPSA, specific surface area; SPSAra, SPSA calculated/SPSA measured; \( d_{50} \), median diameter) and projection of the pea fractions on the components.
3. a group with the throughs alone, higher in SPSA and SPSAra., higher in starch content, but lower in CP content according to the F0.122 fraction, and with a low value of bulk density, close to that of F1.94.

On the individuals projection, the whole flour had a special position. This is due to its high bulk density value, that can be explained by its high heterogeneity in particle size (see Section 3.2.2).

4. Discussion

The applied fractionation process produced nine particle size classes well-differentiated from each other with narrow particle size distributions (except for the throughs), but well spread over a large granulometric range (size of particles between <0.1 mm and ca. 4 mm). The cut size values were often distant from the size of the sieve openings used for fractionation. This could be attributed to the fact that fractions were obtained from a continuous siever (Sweco sieving classifier) while particle size distribution was measured by discontinuous sieving (laboratory siever Bühler MLU 300). Their different characteristics (screen apertures, thickness of the wire forming the screen, mesh number) would have led to these discrepancies. The values of sharpness of cut did not vary too much. The small observed differences can be linked, on the one hand to the carry over of the fine particles with the oversize particles, and on the other hand to an abrasion phenomenon of the particles on the screen (Smigerski, 1984).

Regarding fraction characterization, chemical composition variations were due to a more or less deep and fast separation of different botanical constituents of the pea seed. These differences in composition have to be avoided in close-circuits grinding whose purpose is to spare energy through a proper association of grinding and screening machines. Conversely, in some mineral or food industries (flour milling, semolina industry, dehulling in oil production) fractionation has to be used in order to remove specific components from the raw materials. Friedrich and Hansen (1980) found a starch enrichment in the finer fractions (0.5–1.0 mm) separated by sieving from ground barley, maize and wheat. Air classification is also a fractionation process commonly used in the food industry in order to separate finely ground materials (legume seeds or wheat) meals into a coarser starch-rich fraction (30–40 μm) and a finer protein/cell walls-rich fraction (15–25 μm) (Sosulski, 1983; Wright et al., 1984).

These results with peas are in agreement with those from Galyean et al. (1981) with maize or those obtained earlier by Hansen and Henderson (1966) with barley and maize in animal feeding. Nevertheless, studies on products coming from the fractionation of an initial flour are scarce. Frequently, flours differing in particle size are whole flours. They are achieved using grinders equipped with different screen sizes (Bjorndal et al., 1990; De Boever et al., 1993; Wadhwa et al., 1998) or using separate types of grinder (Lykos and Varga, 1995; Hunt, 1996). The resulting meals are then of poor particle size homogeneity: they consist of a mixture of relatively fine and coarse particles, whose chemical characteristics are similar to those of the initial raw material.

Among the physical characteristics, two groups are distinguished: these are the measures linked to the morphology of the particles \(d_{50}, \text{SPSA, TPV}\) and the measures
linked to the mass (DENap. and DENSE). The evolution of these two groups differs between fractions. The ‘morphology’ parameters increased rapidly in the fine particles, that contained therefore a higher percentage of the total surface area and TPV of the flour, while density values evolved in a more irregular manner. Some physical characteristics could be considered as composition tracers: a high specific surface area is for example an indicator of particles enriched with cytoplasmic constituents. These criteria could be potentially used as a way to better define the characteristics of feedstuffs.

In contrast to our density results, several authors showed that as feed particle size decreases, density increases (Hooper and Welch, 1985; Martz and Belyea, 1986; Siciliano-Jones and Murphy, 1991). The experiments usually involved soaking the particles in solutions of different gravity gradients. The results may differ owing to the hydration capacity of the particles. It is interesting to note that on dry feeding, the apparent density of fractions did not change systematically with particle size. With the same method, De Boever et al. (1993) achieved a similar result with maize silage. So, the treatment ‘grinding–fractionation’ does not lead to fines of greater density. That was shown as well by the non-significant correlation between density and chemical composition. So this criterion did not permit a specific characterization of the different granulometric fractions.

In the same way, it is doubtful that TPV could be regarded as a significant parameter in the characterization of fractions. Even if it appeared highly correlated to the median diameter ($\rho=-0.8$), the need to assimilate all the external cracks on the particles to cylindric pores for its determination, the low values recorded and the high variation between the measures, reduce the interest of TPV in feed characterization. Chesson et al. (1997) reported higher TPV values of ca. 2.7 mm$^3$ g$^{-1}$ for wheat ground through 1 mm screen size. These data are more similar to the more finely ground hulls and kernels (3.2 and 1.1 mm$^3$ g$^{-1}$, respectively) than to the granulometric fractions (0.5 mm grinding screen versus 4 mm grinding screen), which suggests that TPV is linked to the fineness of grinding. However, even if the hulls and kernels fractions were obtained after a finer grinding than the wheat flour, their TPV were of the same order as the wheat value; so we can deduce that the pea flour would be less porous. The calculated mean radius of the pores was from 2 to 6 nm for the different fractions indicating that pea fractions are composed principally of meso-pores (defined as being from 2 to 30 nm).

In a more global approach, the different physical and chemical characteristics of the granulometric fractions seemed to reflect the fact that hulls and kernels follow different comminution laws. So, two populations of particles were coexisting: a population of hull particles accumulated in the coarser fractions (F1.94 to F1.04), and a population of kernels, probably bi-modal and more represented in the medium and fine fractions.

Effectively, the first element suggesting the differential grinding behaviour of hulls and kernels was their different particle size distribution, in spite of being ground on the same screen. The higher elasticity of hulls made them more resistant to the crushing action of the grinder, certainly because of their high content of structural carbohydrates (Brennan et al., 1981). This resistance would explain why the hull particles of the pea were coarser than kernel ones. The ‘grinding–fractionation’ process has led to a physical separation of the constituents of the pea seed in three groups of fractions: a group of coarse fractions
where hulls were accumulated, a group of finer fractions enriched with kernels and the throughs mainly composed by starch granules.

5. Implications

Nine granulometric fractions were produced with a good classification accuracy. They were characterized chemically and physically. The observed results or existing relationships appeared complex as was also the grinding process that led, after fractionation of a pea flour, to granulometric fractions of variable physical and chemical characteristics. According to the constituents of the seed, there were different comminution laws, that could explain these results. The application of different particle characterization methods (linked to shape or mass of the particles), other than simple sieving appeared interesting to define more specifically and completely the feed particles. It is necessary indeed, to take into account different points in order to compare with other research studies:

The type of apparatus (e.g. granulometry by mechanical sieving and granulometry by laser diffraction) can give different results as Siciliano-Jones and Murphy (1991) showed by observing the evolution of two types of density called true and functional specific gravity, when altering the size particles or studying the in vitro fermentation of different feedstuffs.

The criteria of particles qualification, as the definition of their size (e.g. mean particle size or specific surface area); besides Michalet-Doreau and Cerneau (1991) advised the use of criteria like mean particle size which are more precise than one like the size of the grinding screen.

The intrinsic qualities of the substrate (moisture, chemical composition, mechanical properties of the cells...) (Brennan et al., 1981).

The technological process of preparation (type of grinder...), as Richards et al. (1995) underlined when obtaining differences in the in vitro degradation of the cereals starch, with the use of different grinding principles or different grinding parameters.

In a second paper, we will try to set up an original work based on the association of technology and characterization developed in the present part and nutritional studies in order to assess the ability of physical characterization to explain and predict the nutritive value of peas.

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