Sunflower protein enrichment
Methods and potential applications

Marcello Murru* and Concepcion Lera Calvo

Cargill R&D Centre, Vilvoorde, Belgium

Received 10 December 2019 – Accepted 27 February 2020

Abstract – A method to increase the protein content of sunflower meal was developed that uses a combination of milling, sieving and gravity tables to separate fractions with higher and lower protein content. The investigation allowed to compare different mills’ ability to break down the lumps of raw sunflower meal and allow a suitable mechanical separation with sieving and gravity separation. Different settings of the mills were tested with or without material pre-sieving. Sieve mesh sizes were investigated in the range 250 to 500 μm that allowed the production of high protein fine material and a good performance of the gravity table separation. Sunflower meal was successfully enriched in protein to a level similar to low protein soybean meal by utilising the process described in this work. In particular proteins were increased on average by 7% to a level of 43.5%. The yield of the separation can justify industrial applications of this process whereby the high protein material can have a potential use in feed and food formulations.

Keywords: sunflower meal / protein / fibres / gravity separation / sieving / milling

1 Introduction

Sunflower seeds are an important source of oil and proteins especially in Europe where the largest producers are concentrated. The World production of sunflower seeds for 2019/2020 is estimated by the United States Department of Agriculture (USDA) 51.38 million metric tons with ¾ of the production concentrated in Ukraine, Russia and the European Union (Seiler and Gulya, 2016; “World Sunflower Production 2019/2020”, 2019) making it the 3rd ranking oilseed after soybean and rapeseed (MPOB, 2019) in terms of production volumes.

Due to their high oil content sunflower seeds are normally processed in oil extraction to produce high quality oil mostly for food application and a residual solid material that is traditionally
known as sunflower meal. The oil crushing process is typically composed of a preparation step where the seeds are cleaned, dried, often dehulled, thermally treated and an extraction step where they are pressed and in most cases finally extracted with a solvent (Mulder et al., 2012). In the present article we will only consider the solvent extracted meal. 

Despite the relatively low lysine content and metabolizable energy, sunflower meal is regarded as a good protein source by the feed industry due to its high protein availability (Salunkhe, 1992) and the limited number of anti-nutritional factors (Burel et al., 2012). Sunflower meal can be used to effectively replace soybean meal in poultry (Senkoylu and Dale, 1999; Ditta and King, 2017), swine (Thacker and Kirkwood, 1992) and ruminants (Stake et al., 1973).

Rama et al. reported that DM digestibility may be hindered by fibres due to reduced intestinal transit time (Rama Rao et al., 2006) and in general fibres are seen as one of the main obstacle to the high incorporation of sunflower meal in animal feed, especially monogastric.

Over the last decade oil seeds are used more and more in food applications for their nutritional and general health benefits.

Sunflower flour, as the secondary stream of sunflower oil production, can be applied in a broad range of bakery applications, given the specific composition. The sunflower flour is rich in proteins, fibres, minerals, vitamins and is a good source of anti-oxidants (Grasso et al., 2019).

The first bakery application range, which can be considered, is based on low fat hard biscuits to high fat soft biscuits and wafers, using mainly wheat flour as most important ingredient. The traditionally used wheat flour could partially be replaced by the sunflower flour, depending on the formulation, the replacement level could amount to 40%. Replacement levels above 10% will create products which are different from the reference, in terms of hardness, spread, appearance and taste. Interesting new developments can be designed and some further cosmetic adaptions could help to improve the texture and mouth feel.

Typical formulation for biscuits (sweet as well as savoury) contains 10 to 30% oil or fat, around 15 to 25% of sweetener and 30 to 45% of wheat flour, the remaining part is filled by fibre, water and minor components like leavening agent, salt and flavours (Manley, 2001). Up to 50% of the wheat flour can be replaced by sunflower flour, resulting in a different biscuit showing a much larger spread compared to the reference, a slightly increased hardness and a darker appearance. However, one-third replacement of the wheat flour would deliver a biscuit more similar to the reference (Grasso et al., 2019).

Table 1. Percent composition of sunflower seeds components and of fully dehulled protein meal. (Shahidi, 2005).

| Hull   | Kernel | Fully dehulled meal |
|--------|--------|---------------------|
| Oil    | 3      | 67                  |
| Protein| 3      | 21                  |
| Fibre  | 61     | 3                   |
| Ash    | 3      | 3                   |
| N-free extract | 31 | 6 |

Replacement level of wheat flour by sunflower flour in wafer amounts to maximum 15% to deliver a good wafer and amounts to maximum 10% to result in a comparable-to-reference product, and ideally maximum 5% to show no deviations.

The second group of applications for sunflower flour can be that of breakfast cereals and cereal bars, making use of corn flour. Up to 20% replacement of the corn flour by sunflower flour in extruded breakfast cereals can result in less expanded products. However, replacement level of maximum 10% could be considered as an excellent expansion regulator, enhancing the vitamins, minerals and anti-oxidants.

Similar replacement level of 20% corn flour by sunflower flour can be applied in a corn granola bar, resulting in a lower density and hardness of the bar. Further adjustments are required in replacement to correct for hardness, colour and mouth feel.

In both food and feed applications there is a need to enhance the protein content and reduce the mostly insoluble fibres that reduce DM digestibility and result into unacceptable aspect in food due to presence of black specs.

Sunflower seed hulls make up to 30% of the seed weight (Mulder et al., 2012; Hamm et al., 2013) and have a rather limited value due to their poor nutritional properties. Typical compositions of sunflower hulls and kernels are shown in Table 1.

As proteins are the most valuable components in the majority of the applications, a fully dehulled and defatted meal with a theoretical composition indicated in Table 1 would be an ideal production target. However, standard commercial sunflower meals protein content ranges from about 28% to 36% depending on the level of decortication adopted in the crushing preparation. A full composition of commodity sunflower meal grades is shown in Table 2.

Dehulling of oilseeds is typically done when possible in order to improve the protein content of the oilseed meal after oil extraction and to maximise the capacity of the extractors as hulls typically do not contain significant quantities of oil. In sunflower seeds dehulling is also important because of the presence of waxes in the hulls that would be extracted together with the oil during solvent extraction (Denise, 1983; Mulder et al., 2012). However, if less than 15% of the hull remains in the seed pressing efficiency is reduced and percolation may become problematic during solvent extraction (Beal, 1987; Bockisch, 1998) thus compromising the oil yield. (Raß et al., 2008) explain that pressing kernels leads to blockage of the oil drainage pathways in the material due to lower, elastic retractive forces for extraction. Hulls can be used as fuel material though the energy requirement of a standard sunflower crushing plant would be fulfilled with only 25% of the hull removal (Mulder et al., 2012), therefore high dehulling rate would result in large quantities of hulls to be transported off site.

The dehulling described so far in this article is traditionally called head-end or front-end dehulling as it is located in the beginning of the crushing process before pressing and extraction occur (Fetzer, 1983; Mulder et al., 2012).

When head-end dehulling is not practically or economically convenient, tail-end dehulling (following oil extraction) can be used to increase the protein content of the final oilseed meal. Given the difficulty in efficient rapeseed dehulling and
the relatively high oil content of the hulls fraction (McKinnon et al., 1995) head-end dehulling of rapeseed is not often commercially utilised, therefore several studies were conducted on tail-end dehulling of rapeseed meal with varying techniques. Sieving is known to be a simple but effective way of increasing the protein content of oilseeds since smaller particles tend to have higher protein content. Particles with high fibres content are normally lighter and may have a more elongated shape when compared with particles with low fibres.

A list of possible tail-end dehulling methods is shown in Table 3. Several articles were published on the topic and the list is not intended to be exhaustive.

All the techniques listed in Table 3 can be preceded by milling to increase the yield of the protein rich fraction as for example indicated by (Laguna et al., 2018) who target ultra fine particles in order to break down the material before separation.

In this work we will focus on the potential that gravity tables have for the separation of proteins and fibre of sunflower meal. Gravity tables are traditionally used in the mining industry to separate the valuable compounds in the ore but also in waste management and in cereals and oilseeds cleaning to remove sprouted, broken and damaged kernels which are normally concentrated in the least dense fraction (Singhal et al., 1997).

As mentioned by (Kannan et al., 2017) a gravity table consists of an inclined oscillating deck the transports the heavier fraction towards the higher end, while the lighter one is collected at the base. The deck of a gravity table is fitted with a net through which a vertical air flow fluidises the particles present on top of the deck. The oscillation of the deck applies a traction that ensures that the dense product is pushed to the upper end while the lighter particles roll over the layer of heavy product in the opposite direction (Kannan et al., 2017). The same authors explain the importance of the operating parameters of the gravity table, namely the longitudinal and transverse deck inclination, the deck eccentric speed and the particles fluidizing conditions (the air flow rate). A detailed description of the operating principle of the gravity table is provided by (Das, 1986) who highlights that the particles will

| Parameter                  | Unit | As fed | On DM | As fed | On DM |
|----------------------------|------|--------|-------|--------|-------|
| Dry matter                 | %    | 88.9   | 100   | 90.5   | 100   |
| Crude protein              | %    | 27.3   | 30.7  | 36.6   | 40.5  |
| Crude fibre                | %    | 26.3   | 29.6  | 17.9   | 19.7  |
| Crude fat                  | %    | 1.9    | 2.1   | 1.2    | 1.4   |
| Ash                        | %    | 6      | 6.8   | 6.5    | 7.2   |
| Insoluble ash              | %    | 0.5    | 0.6   | 0.1    | 0.1   |
| NDF                        | %    | 41.8   | 47    | 31.5   | 34.9  |
| ADF                        | %    | 30     | 33.8  | 21.1   | 23.3  |
| Lignin                     | %    | 10.2   | 11.4  | 6.8    | 7.5   |
| Water insoluble cell walls | %    | 46.5   | 52.3  | 27.9   | 30.8  |
| Starch                     | %    | 3.4    | 3.8   | 3.5    | 3.9   |
| Total sugars               | %    | 5.3    | 6     | 6.7    | 7.4   |
| Gross energy (kcal)        | kcal/kg | 4120 | 4630  | 4190   | 4630  |
| Gross energy (MJ)          | MJ/kg | 17.2   | 19.4  | 17.5   | 19.4  |

Table 2. Typical composition of commercial dehulled and non-dehulled sunflower meal. (Sauvant et al., 2004).

| Technique                  | References                  | Oilseed          |
|----------------------------|-----------------------------|------------------|
| Sieving/Screening          | (Davin, 1983)               | Rapeseed         |
|                            | (Mostafa and Murru, 2018)   | Rapeseed         |
|                            | (Saito et al., 2007)        | Rapeseed         |
|                            | (Leterme, 2013)             | Sunflower        |
|                            | (McCurdy and March, 1992)   | Rapeseed         |
|                            | (Lević et al., 1992)        | Sunflower        |
| Air classification         | (Laudadio et al., 2013)     | Sunflower        |
|                            | (Ulrich, 2002, 2010)        | Sunflower        |
|                            | (Delrue and Van De Watering, 2008) | Rapeseed        |
|                            | (Laguna et al., 2018)       | Rapeseed/        |
|                            | (Banjac et al., 2013; 2014) | Sunflower        |
| Electrostatic separation   | (Delrue and Van De Watering, 2008) | Rapeseed        |
|                            | (Basset et al., 2016)       | Rapeseed         |
|                            | (Laguna et al., 2018)       | Rapeseed/        |
|                            | (Xing et al., 2018)         | Sunflower        |
|                            | (Kdidi et al., 2019)        | Soybean          |
| Gravity separation         | (Hahn et al., 2014)         | Sunflower/       |
|                            |                             | Rapeseed         |

Table 3. Possible tail-end dehulling techniques and relative literatures References

| Parameter                  | Unit | As fed | On DM | As fed | On DM |
|----------------------------|------|--------|-------|--------|-------|
| Dry matter                 | %    | 88.9   | 100   | 90.5   | 100   |
| Crude protein              | %    | 27.3   | 30.7  | 36.6   | 40.5  |
| Crude fibre                | %    | 26.3   | 29.6  | 17.9   | 19.7  |
| Crude fat                  | %    | 1.9    | 2.1   | 1.2    | 1.4   |
| Ash                        | %    | 6      | 6.8   | 6.5    | 7.2   |
| Insoluble ash              | %    | 0.5    | 0.6   | 0.1    | 0.1   |
| NDF                        | %    | 41.8   | 47    | 31.5   | 34.9  |
| ADF                        | %    | 30     | 33.8  | 21.1   | 23.3  |
| Lignin                     | %    | 10.2   | 11.4  | 6.8    | 7.5   |
| Water insoluble cell walls | %    | 46.5   | 52.3  | 27.9   | 30.8  |
| Starch                     | %    | 3.4    | 3.8   | 3.5    | 3.9   |
| Total sugars               | %    | 5.3    | 6     | 6.7    | 7.4   |
| Gross energy (kcal)        | kcal/kg | 4120 | 4630  | 4190   | 4630  |
| Gross energy (MJ)          | MJ/kg | 17.2   | 19.4  | 17.5   | 19.4  |

Table 4. Composition of raw sunflower meal used in this study.
slide on the deck surface when the magnitude of the required force due to gravity and deck acceleration is greater than the magnitude of the available frictional force. Since all such forces are proportional to the mass of the particles we can assume that density and volume will play a significant role. For this reason it is usually recommended to have a relatively narrow particle size distribution in the feed material to be able to separate the components based on density rather than size.

In this work a combination of milling and sieving was evaluated as first step of the protein enrichment process and preparation for the gravity separation.

2 Materials and methods

2.1 Materials used

The raw sunflower meal (SFM) used for this study was produced in the Cargill Kakhovka (Ukraine) plant by crushing locally produced seeds through front-end dehulling, pressing and solvent extraction. Composition of the raw material is shown in Table 4. The material was collected in the production site after drying and shipped in sealed plastic drums.

The cumulative particle size distribution of the raw material is shown in Figure 1. Agglomerates, occasionally larger than 1 cm are present in the material. The material tends to segregate during transport, therefore it was carefully remixed before running the experiments.

Sunflower meal is often sold in pellets form, however, for this study the product used was in form of lose meal and dried in a standard meal drier.

2.2 Analytical methods

The analytical methods used in this study are shown in Table 5. All analyses reported in the article are expressed on “as is” basis. Sampling was identified as a critical activity as the material has the tendency to segregate resulting in uneven distribution of the hull fraction within the sample. The samples from the experiments were always remixed before sampling and analyses were carried out in duplicates.

Samples for protein analysis were milled with a lab mill Retsch ZM200 fitted with a sieve of 1 mm opening before being processed in the Thermofisher Flash 2000 nitrogen analyser. The Protein/N conversion factor used for sunflower samples was 6.25.

2.3 Experimental equipment

The first part of the plan consisted in milling the material with the 3 different mills. The milled material was sieved with 3 different meshes to explore the possibility of obtaining sunflower meal with at least 40% proteins with a yield that would allow a performance of the gravity table that could result in a final protein content in the region of 43–44% that is close to LowPro soybean meal (Sauvant et al., 2004).

Milling affects heavily the sieving performance and in the layout selected in this study generating too fine particles of hulls and cotyledon would result in low selectivity in the sieving and poor performance in the gravity table (dust would be blown away or fall below the mesh of the deck). Preliminary sieving the meal before milling on a 1.0 mm mesh was attempted to reduce the amount of fine dusts.

The experimental plan was split into 2 phases:
– determine if pre-sieving the raw material is beneficial to improving fines protein content and overall performance of the process in terms of yield and protein content;
– evaluate different mills’ influence on:

\[\text{Raw material PSD}\]

Fig. 1. Cumulative particle size distribution of the raw sunflower meal.

Particle size distribution analyses were carried out by using stacks of sieves that were placed in a tapping and shaking machine.

Table 5. Analytical methods used in this study.

| Analysis       | Method                  | Reference                                      | Accuracy                 |
|----------------|-------------------------|------------------------------------------------|--------------------------|
| Moisture       | Oven at 135°C for 2 h    | SOP Cargill MOI-001 ref method AOAC, 2000, 925.09 | MOI 9–11% Ue = ±0.8%     |
| Crude protein  | Dumas                   | NF V 18–120 (1997) – AOAC 2000, method 968.06    | CP 20% Ue = ±0.7% CP 30% Ue = ±0.9% CP 40% Ue = ±1.2% CP 50% Ue = ±1.5% CP 60% Ue = ±1.8% |
| Crude fiber    | Weende                  | SOP Cargill FIB-005 ref method AOAC, 2000, 978.10 | FIBER 10% Ue = ±0.8% FIBER 20% Ue = ±1.6% FIBER 40% Ue = ±3.3% |

Ue is the expanded uncertainty calculated using a coverage factor = 2; Level of confidence is approximately 95%.
Once the right sieve mesh size was identified for all the mills, running the full process with milling sieving and gravity table was necessary in order to verify the effect of the mill on the overall performance.

The goal was to identify if the process was capable of achieving a protein content in the region $\geq 43\%$ w/w and fibres content $\leq 11\%$ w/w.

Mass balances were performed on the global experiment and at each step of the process in order to verify the reliability of the analytical and experimental data. Fibres quantification in some experiments is not always reliable as mass balances for some experiments did not close by $> 5\%$.

All samples produced in the experiments were collected in polyethylene bags, sealed, labelled and shipped for analysis. Analysts re-homogenised the samples before carrying out the analytical procedure. Figures 2 and 3 show the configuration used in the phase 2 of the experimental work after the phase 1 where pre-sieving.
was evaluated and the mesh size of the fine sieving had been selected.

### 2.4 Milling

Sunflower meal can be rather agglomerated even after desolventisation and drying in agitated equipment. Milling is required in order to disentangle the cotyledon chunks from the hull particles before attempting separation (Laguna et al., 2018). Different mills were evaluated for this purpose:
- hammer mill;
- disc mill;
- roller mill.

Other mills like delumpers were also considered but discarded after recommendation from the manufacturers. Higher intensity mills like pin or ball mills were deemed inappropriate as they would mill the product too finely and therefore generate excessive dust which would reduce the yield and the performance of the gravity tables.

The following laboratory / small pilot mills were used in the experiments:
- Hosokawa Alpine hammer mill–Universal mill 25 MZ (1979) fitted with:
  - 2 mm screen;
  - capacity up to 120 Kg/h;
  - variable speed of the hammers 1500–3000 RPM;
- Buhler laboratory roller mil MLU202:
  - capacity ~10–15 Kg/h;
  - rollers used: 154 mm diameter, 70 mm long with 9.5 corrugations/cm, 4% spiral: 0.1 land and style of grooves = 18;
  - gaps between the rollers: 0.15–1 mm;
- Perten LM3600 Disc Mill with varying distances between rotor and stator:
  - power input ~1.3 kW;
  - disc material: hardened steel;
  - disc diameter 100 mm;
  - capacity for sunflower meal ~15 Kg/h;
  - gap between stator and rotor expressed with numbers ranging from 2 to 8, with lower number indicating smaller gap and therefore finer grinding.

Laboratory scale roller mills can occasionally experience some variations in the gap between the rollers when relatively large agglomerates are fed through the mill and this can be the source of some inaccuracy. Therefore the roller mill could not be fed directly to the roller at the intended gap therefore a pre-milling step was carried out on wider rollers gap setting followed by grinding to the intended gap. Despite this procedure occasional deviations in the rollers opening would be identified.

---

### 2.5 Sieving

The sieving machines used for the experimental work are the following batch sieves:
- Russell Finex 2217300 with the following specifications:
  - vibrating sieve;
  - effective screen area 0.207 m²;
  - motor rotation 1400–2800 RPM;
  - screens used–250 µm, 1 mm, 1.4 mm;
- Sweco S18S with the following specification:
  - vibratory separator;
  - effective screen 0.059 m²;
  - motor 10 kW at 1400 RPM;
  - screens used 300, 400, 500 µm.

### 2.6 Gravity table

The last separation step was carried out on a Cimbria gravity table LGA with a capacity for sunflower meal of about 40 Kg/h based on milled and sieved sunflower meal.

The gravity table had a 100% dynamically counter-balanced eccentric deck system with 1-IEC motor 0.37 kW:
Table 7. Results of preliminary sieving trials (composition on as is basis).

| Mill      | Setting | Pre-sieving | Fines Sieving | Fines | Coarse | Coarse |
|-----------|---------|-------------|---------------|-------|--------|--------|
|           |         |             | yield %       | % protein | % fiber | % moist. | yield % | % protein | % fiber | % moist. |
| Disc      | NO      | 250         | 13.5          | 40.3   | 11.8   | 9.6     | 86.5    | 35.7      | 19.9    | 9.9     |
|           | YES     | 250         | 16.0          | 39.8   | 12.4   | 9.4     | 84.0    | 36.8      | 18.5    | 10.1    |
|           | NO      | 250         | 11.6          | 40.7   | 11.9   | 10.3    | 88.4    | 35.7      | 20.1    | 9.9     |
|           | YES     | 250         | 13.1          | 39.9   | 12.1   | 10.2    | 86.9    | 36.9      | 19.6    | 10.1    |
|           | NO      | 400         | 26.7          | 40.0   | 14.5   | 9.2     | 73.3    | 33.8      | 19.0    | 9.5     |
|           | YES     | 400         | 32.8          | 39.9   | 13.7   | 10.2    | 67.2    | 35.2      | 20.4    | 9.5     |
|           | NO      | 400         | 24.5          | 38.9   | 14.3   | 8.8     | 75.5    | 37.1      | 19.2    | 9.5     |
|           | YES     | 400         | 26.4          | 39.4   | 15.7   | 10.3    | 73.6    | 36.0      | 17.9    | 9.3     |
|           | NO      | 500         | 32.8          | 38.0   | 15.2   | 10.0    | 67.2    | 35.9      | 20.2    | 9.9     |
|           | YES     | 500         | 42.8          | 38.4   | 15.5   | 9.4     | 57.2    | 35.7      | 20.1    | 9.3     |
|           | NO      | 500         | 32.5          | 38.0   | 15.7   | 9.7     | 67.5    | 37.1      | 19.0    | 9.3     |
|           | YES     | 500         | 31.8          | 38.7   | 17.1   | 10.3    | 68.2    | 35.9      | 20.2    | 9.9     |
| 1500 RPM  | NO      | 250         | 19.8          | 39.7   | 13.3   | 8.7     | 80.2    | 36.0      | 20.5    | 8.3     |
|           | YES     | 250         | 17.8          | 40.8   | 13.6   | 9.8     | 82.2    | 36.3      | 22.0    | 9.4     |
| 1500 RPM  | NO      | 400         | 39.1          | 38.3   | 16.4   | 8.7     | 60.9    | 35.5      | 20.1    | 8.6     |
|           | YES     | 400         | NA            | 39.1   | 15.1   | 9.7     | NA      | 36.4      | 20.5    | 9.6     |
| 1500 RPM  | NO      | 500         | 46.6          | 37.6   | 18.4   | 8.4     | 53.4    | 36.6      | 20.0    | 8.4     |
|           | YES     | 500         | 37.1          | 38.5   | 17.0   | 9.2     | 62.9    | 38.4      | 18.8    | 9.0     |
| 0.58 mm   | NO      | 250         | 10.3          | 43.2   | 10.8   | 9.9     | 89.7    | 36.1      | 19.6    | 9.4     |
|           | YES     | 250         | 11.7          | 43.7   | 10.3   | 9.8     | 88.3    | 36.4      | 19.9    | 9.5     |
| 0.58 mm   | NO      | 400         | 26.2          | 43.4   | 13.5   | 9.8     | 73.8    | 35.5      | 21.0    | 9.4     |
|           | YES     | 400         | 25.5          | 43.2   | 13.5   | 9.9     | 74.5    | 36.8      | 20.3    | 9.8     |
| 0.58 mm   | NO      | 500         | 31.3          | 42.5   | 13.5   | 9.9     | 68.7    | 36.0      | 20.8    | 9.9     |
|           | YES     | 500         | 28.3          | 42.4   | 13.2   | 9.8     | 71.7    | 35.2      | 20.0    | 9.6     |

Table 8. Comparison of yield, protein and fibre content of different fine fractions of sieved sunflower meal.

| Fine fraction (mm) | Yield % | Protein % | Fiber % | Moisture % |
|--------------------|---------|-----------|---------|------------|
| 0.36–0.6           | 8.7     | 40.3      | 14.3    | 9.4        |
| 0.18–0.36          | 5.4     | 43.1      | 10.6    | 9.4        |
| <0.18              | 1.2     | 43.6      | 8.3     | 8.4        |

- longitudinal inclination: 0°–2°;
- transverse inclination: 3°–5.5°;
- eccentric drive speed: 50 Hz;
- fan drive speed: 50 Hz;
- max air inlet (setting 10): 37 m³/min;
- table area 0.2 m².

A schematic of the deck of the gravity table LGA is shown in Figure 4. The settings of the gravity table could change depending on the milling and sieve mesh used in the previous steps. Table 6 shows the parameter range for each material coming from the different combinations of milling and sieving. Changes of the process parameters were needed to optimise the separation depending on the feed material used. For each condition reported in this article several runs were carried out on the gravity tables, however only the best one or 2 separation performances are reported in the results section.

Blending of the fines and heavy material to obtain the final product was not carried out. Final product composition was instead calculated from the composition data of fines and heavy from gravity table.

3 Results and discussion

3.1 Pre-sieving (Phase 1)

The impact that pre-sieving sunflower meal ahead of milling has on sieving yield and protein content of fine and coarse fractions is shown in Table 7. The pattern is clearly dependent on the type of mill selected and the results are discussed for the 3 different mills in the following sections. As a reference the raw material (not milled) was sieved with a stack of sieves and the composition of the fine fractions below 0.6 mm was measured. Table 8 shows that by sieving the standard sunflower meal relatively high protein content can be achieved, though with a low yield.
The low yield of the fine fraction is due to the agglomerated nature of the sunflower meal following the processes of pressing and solvent extraction.

3.2 Hammer mill

The hammer mill was found to effectively de-agglomerating the sunflower meal and the milled material had the aspect of a rather uniform powder (see Fig. 5).

As expected, the yield of the fine fraction increases when the mesh size increases. Despite the large increase in fines yield the fibre content shows only a modest increase.

Yield decreases and protein content slightly increases when the material is pre-sieved (Fig. 6). Hammer mill is confirming the hypothesis that there is no selectivity towards milling loose meal particles (whether hulls or cotyledon fragments) and agglomerates. This is consistent with the idea that small hulls are further reduced to smaller size and pass through the fine sieve thus reducing protein content in that fraction.

In conclusion, if hammer mill is to be used in this process, pre-sieving seems to be beneficial since it slightly increases the protein content of the fines and reduces the yield. However, it is arguable if an investment in an extra sieving equipment can be justified for a marginal improvement.

From the results of the current trials and past experience it was decided to use 250 μm sieve for the process including the gravity table as it allows a higher protein content of the fines and provides a large amount of material to be processed on the gravity tables (that is expected to achieve a better separation).

The option of using an even lower mesh size was discarded as this would make it hard for the gravity tables to separate the particles and would increase the amount of dust during the separation.
3.3 Roller mill

As it can be seen in Figure 7 the fine fractions obtained when grinding with the roller mill at 0.58 mm gap show a protein content comfortably above 43% even when the material is sieved with a 400 μm sieve. A peculiarity of the roller mill is that much of the loose hulls fed into the mill remain intact after milling and can be clearly seen in the milled material (Fig. 5). This is clearly reflected in the high protein content of the fines and in their lower yield. Fines yield is also lower because several particles are flattened rather than milled, therefore they do not pass through the fine mesh. This phenomenon is likely to impact negatively if the material is more humid and elastic and the agglomerates end up being flattened instead of being broken resulting in poor separation of cotyledons and hulls.

3.4 Disc mill

The Perten disc mill was preliminary tested with different gaps between rotor and stator and the settings of 4 and 6 were selected on the basis of the particle size distribution (Fig. 8), the protein content of the fine fraction and the potential amount that could be processed in the gravity table. Setting 8 was discarded as it was producing a large amount of particles > 1 mm and visually it was determined that it was not appropriately breaking down the lumps. Setting 2 (not reported in the data) on the other hand was milling the material to a fine dust and was therefore also discarded.

The performance of the disc mill is rather similar to that of the hammer mill and the material also has a similar aspect (Fig. 5). The protein content of the fine fraction is relatively low even though the fines yield is 4–5% lower than that obtained with the hammer mill on the same sieve size. It can be concluded that also the disc mill does not have selectivity in grinding hulls particles or cotyledon particles. Pre-sieving the meal seems to have only minor effects on the protein and the fibres content. However, the yield seems to increase when pre-sieving is carried out ahead of milling (see Figs. 9–11). This finding was surprising, but it was reproducible as it was also observed in the second part of the study (see Tab. 9). A potential explanation is that when only agglomerated particles are fed into the mill, more friction is experienced by the material and therefore more fines are produced. As the difference in protein content between 250 and 400 μm mesh is limited, 250 μm is the recommended mesh size since the fines yield is lower and more material can therefore be processed in the gravity table.

4 Full process: Milling sieving and gravity table (Phase 2)

The consolidated results of the second phase are shown in Table 9. Critical parameters in the evaluation are the overall yield, the protein and the fibre content of the final material.
The target yield for the experiment was 65%, however, this was challenging in some experiments as visually the separation of the gravity table seemed poor. In such experiments separation quality with the gravity table was prioritised versus yield.

Figure 12 shows that there is a clear positive correlation between the protein content of the final product and that of the heavy fraction of the gravity table. This is an indication that most of the contribution to the total protein enrichment of the final product (mix of fines + heavy) is derived from the heavy fraction of the gravity table as it can achieve higher proteins than just sieving and has a higher yield.

In this preliminary study several parameters were changed for each experiment therefore only mild correlations between variables could be achieved. However, the protein content of the final product was on average 43.5 (see Fig. 13) that is in the range of low-pro soybean meal (Sauvant et al., 2004) and results in a protein increase of about 7% from the starting material. Fibre content was on average 12.13 which is about twice that of low-pro soybean meal.

The yield of the final product is on average 66% (Fig. 14) and the combination of the two parameters (yield and protein content) results in the key economic driver for the process described in this article.

Table 9 confirms the small variation of protein and yield in the fine fraction in case of pre-sieving before milling (hammer and disc mill). However, the data from the table also show that pre-sieving does not bring apparent benefits to the overall protein and fibre separation neither in terms of protein content nor in terms of yield of the final product.

For materials milled with the roller mill a mild negative correlation between rollers gap and protein content of final product exists though much of the variability is seen in the range 0.75 to 1 mm (Fig. 15). This is due to the fact that too wide gap results into poor performance of the GT due to improper break down of the lumps hence the small particles still agglomerated do not differ much in density.

The layout used for the phase 2 test on the roller mill is shown in Figure 3. The layout is different from the one of the hammer and disc mill since it was decided to use an intermediate sieve (coarse sieve) between the mill and the fines sieve. As previously mentioned the action of the roller mill allows the hulls to pass through the roller while breaking down the lumps. However, it was visually noticed that a portion of the large hulls tend to be drown towards the heavy fraction thus lowering its protein content. Given the difference in size between the hulls and the remaining ground particles, the meal was sieved with a coarse sieve with 1 or 1.4 mm mesh, depending on the selected gap between the rollers of the mill, in order to remove the large hulls before reaching the gravity table.

This coarse sieving allowed to reduce the load to and improve the separation of the gravity table and produce a coarse by-product with proteins as low as 15% and crude fibres as high as 41%.

Based on the results of the first phase trials, pre-sieving was not performed in the second phase for the roller mill set-up. It should be noted that disc and hammer mill lower protein content of the fines is counterbalanced by the high protein content of the heavy fraction from the gravity table. After lumps are effectively broken down in the milling step, sieving with lower mesh size results in lower fines yield and an increased amount of material and wider
| Test Mill | Setting | Sieving | Fine | Heavy | Light | Coarse | Yield M | Yield L | Yield F | Yield Byprod | Yield Byprod Coarse |
|----------|---------|--------|-----|------|------|-------|--------|--------|--------|-------------|-------------------|
| D1 Disc | NO 60 | 75.9 | 42.8 | 12.3 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| D2 Disc | NO 60 | 69.5 | 43.9 | 12.4 | 9.9 | 14.2 | 49.7 | 25.0 | 30.8 | 9.6 | 44.7 | 12.6 |
| D3 Disc | NO 60 | 74.4 | 43.2 | 11.8 | 9.4 | 16.6 | 54.7 | 22.9 | 33.5 | 9.3 | 34.8 | 22.9 |
| D4 Disc | NO 60 | 64.0 | 45.0 | 12.6 | 9.4 | 14.2 | 49.7 | 25.0 | 30.8 | 9.6 | 44.7 | 12.6 |
| H1 Hammer | NO 300 | 76.3 | 44.6 | 12.4 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| H2 Hammer | NO 300 | 66.3 | 43.2 | 11.8 | 9.4 | 16.6 | 54.7 | 22.9 | 33.5 | 9.3 | 34.8 | 22.9 |
| R1 Roller | NO 300 | 71.0 | 43.6 | 15.0 | 8.6 | 11.2 | 62.0 | 23.9 | 30.7 | 8.7 | 32.7 | 20.9 |
| R2 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R3 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R4 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R5 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R6 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R7 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R8 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R9 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R10 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R11 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R12 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R13 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R14 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R15 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R16 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R17 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R18 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R19 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |
| R20 Roller | NO 300 | 75.0 | 44.6 | 12.1 | 9.9 | 11.5 | 57.1 | 24.4 | 33.2 | 9.6 | 32.7 | 24.4 |

M.Murru and C.L. Calvo: OCL 2020, 27, 17

Page 11 of 14
Table 10. Average yield and composition of product for the three mills used.

| Mill   | Product yield | Average composition product |
|--------|---------------|-----------------------------|
|        | %             | % protein                   | % fiber | % moist. |
| Roller | 64.1          | 43.4                        | 12.3    | 9.7      |
| Hammer | 69.2          | 43.3                        | 11.4    | 9.7      |
| Disc   | 68.7          | 43.7                        | 12.1    | 9.6      |

Fig. 13. Distribution of protein content across all the trials carried out in this study.

Fig. 14. Distribution of final product yields achieved in all the trials carried out in this study (for full process with milling, sieving and gravity tables).
particle size distribution in the feed of the gravity table. Therefore, it would be desirable to find a balance between low fines yield and the narrow particle size distribution needed for the GT.

As shown in Table 10 the average yield and composition obtained with the three mills do not show a clear winner, instead we can conclude that all mills can be used to increase the protein content of sunflower meal to a level similar to that of low-pro soybean meal with a yield much higher than what can be achieved with just sieving.

5 Conclusions

This work provided evidence that the fractionation of sunflower meal carried out with a combination of milling, sieving and gravity tables can achieve a protein content close to that of low-pro soybean meal.

The process is feasible with all the three mills investigated in this work achieving an excess of 65% yield and a protein content in the region of 43%.

Each test comprising of the three steps was optimised at the best of the authors' knowledge, however, more work is needed to reach optimal performance for the process. In particular, the optimisation of the gravity separation should be focussed on the finer grinding explored in this article. It should also be noted that upon scale-up, due to the higher surface available an improvement in the separation efficiency of the gravity tables can be expected.

Commercial applications should also consider the potential use of the by-product of the separation. In fact, while the high protein product can find suitable uses in food and feed, the lower protein, high fibre by-product needs to be further evaluated if its protein content is lower than that of a standard low pro sunflower meal (about 27% w/w).

Large variability in and low correlation between yield and protein content of the heavy fraction of the gravity table suggests that an in-depth gravity table parametric study should be carried out.

References

Banjac V, Čolović R, Vukmirović DM, et al. 2013. Protein enrichment of sunflower meal by air classification. Food Feed Res 40: 77–83.
McKinnon JJ, Mustafa AF, Cohen RDH. 1995. Nutritional evaluation and processing of canola hulls for ruminants. *Can J Anim Sci* 75. Canada: Agricultural Institute Of Canada, 231 p.

Mostafa Y, Murru M. 2018. Oilseed meal. US2018146696(A1).

MPOB. 2019. Oilseeds and protein crops market situation [WWW Document]. Comm Common Organ Agric Mark. https://circabc.europa.eu/sd/a/215a681a-5f50-4a4b-a953-e8fc6336819c/oilseeds-marketsituation.pdf (Accessed 10/25/19).

Mulder W, Sanders J, Carre P, et al. 2012. Chapter 3: Primary processing. In: Kazmi A, ed. Advanced oil crop biorefineries. Royal Society of Chemistry, 110 p.

Rama Rao SV, Raju MVLN, Panda AK, Reddy MR. 2006. Sunflower seed meal as a substitute for soybean meal in commercial broiler chicken diets. *Br Poult Sci* 47: 592–598.

Raß M, Schein C, Matthäus B. 2008. Virgin sunflower oil. *Eur J Lipid Sci Technol* 110: 618–624.

Saito S, Sato T, Fujiwara S, et al. 2007. Method for production of rapeseed meal.

Salunkhe DK. 1992. World oilseeds Chemistry, technology, and utilization. New York: Van Nostrand Reinhold.

Sauvant D, Perez J-M, Tran G. 2004. Tables of composition and nutritional value of feed materials: pigs, poultry, cattle, sheep, goats, rabbits, horses and fish. Wageningen; Paris: Wageningen Academic Publishers; INRA.

Seiler GJ, Gulya TJ. 2016. Sunflower: Overview. *Ref Modul Food Sci.*

Shahidi F. 2005. Sunflower oil. In: Bailey’s Ind Oil Fat Prod, Vol. 1-6, 6th ed.

Singhal RS, Kulkarni PR, Rege DV. 1997. 2.9 Detection of damaged grains in sound grains. In: Handb Indices Food Qual Authent.

Stake PE, Owens MJ, Schingoethe DJ. 1973. Rapeseed, sunflower, and soybean meal supplementation of calf rations. *J Dairy Sci* 56: 783–788.

Thacker PA, Kirkwood RN. 1992. Non-traditional feeds for use in swine production. Taylor & Francis.

Ulrich W. 2002. Method and system for preparing extraction meal from sunflower seeds for animal feed. WO02080699(A2).

Ulrich W. 2010. Waste-free processing extraction meal from sunflower seed, comprises separating the meal into first fraction with high raw protein- and low raw fiber content and second fraction with low raw protein- and high raw fiber content by sieving. DE102009032931(A1).

World Sunflower Production 2019/2020 [WWW Document]. 2019. http://www.worldagriculturalproduction.com/crops/sunflower.aspx.

Xing Q, de Wit M, Kyriakopoulou K, Boom RM, Schutyser MAI. 2018. Protein enrichment of defatted soybean flour by fine milling and electrostatic separation. *Innov Food Sci Emerg Technol* 50: 42–49.

Cite this article as: Murru M, Calvo CL. 2020. Sunflower protein enrichment Methods and potential applications. *OCL* 27: 17.