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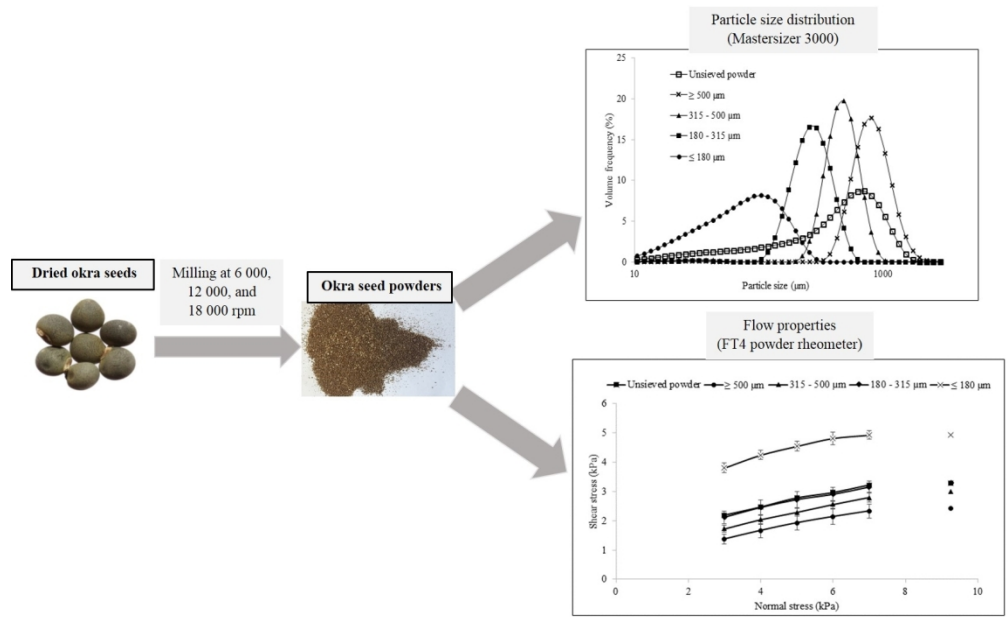
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Effect of milling and sieving processes on the physicochemical properties and flow properties of okra powders

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Graphical abstract

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2
3 1 Effect of milling and sieving processes on the physicochemical properties and flow properties
4 2 of okra powders.

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21
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26
27 15 **Abstract**

28
29 16 The aim of this work was on one hand to investigate the influence of milling speed and mean
30 17 particle size of granulometric classes of okra (*Abelmoschus esculentus* (L.) Moench) seed
31 18 powders on proximal composition (related to nutritional potential). On the other hand, it was
32 19 tried to relate flow properties to powder physicochemical characteristics, namely particle size
33 20 distribution and proximal composition. Then, dry okra seeds were ground with an ultra-
34 21 centrifugal mill at three different rotational speeds: 6 000, 12 000, and 18 000 rpm. Each powder
35 22 was separated into four granulometric classes: $\geq 500 \mu\text{m}$, 315 - 500 μm , 180 - 315 μm , and \leq
36 23 180 μm . Milling speed affected the particle size distribution of dry okra seed powders: on one
37 24 hand, the higher the milling speed, the lower the mean particle size, mainly because of the
38 25 higher the proportion of fine particles ($< 100 \mu\text{m}$); on the other hand, at low milling speed, dry
39 26 okra seed powders were rather monomodal and constituted of large particles, whereas at high
40 27 milling speed they were bimodal and composed of two populations (fine and large particles).
41 28 In granulometric classes, fat and protein contents were significantly higher for fine particles,
42 29 unlike carbohydrate and ash contents that were lower. This differential distribution of
43 30 macronutrients within granulometric classes was enhanced at higher milling speed. Finally, the
44 31 $\leq 180 \mu\text{m}$ granulometric class, corresponding to the smallest mean particle size, had a low
45 32 flowability, confirmed by their high cohesion and compressibility, which can be related to its
46 33 high fat content making it sticky. On the contrary, the $\geq 500 \mu\text{m}$ granulometric class was not
47 34 cohesive, leading to good flow properties.

35 **Key words:** okra seeds; milling; sieving; particle size; FT4 powder rheometer; flowability

36 1. Introduction

37 Okra (*Abelmoschus Esculentus* (L.) Moench) is a vegetable widely grown in tropical and
38 subtropical regions (Torkpo *et al.*, 2009; Jarret *et al.*, 2011). It is called bamia or banya in some
39 parts of the world, particularly in Asia and the Middle East (Uka *et al.*, 2013). This crop is
40 generally considered as drought-resistant and has fewer pests than soybean (Miller *et al.*, 1949;
41 Jarret *et al.*, 2011). Okra plays an important role in the human diet by supplying carbohydrates,
42 protein, fat, minerals, and vitamins that are usually deficient in staple foods (Adelakun *et al.*,
43 2009). It is a vegetable with an enormous economic potential for poor communities because of
44 its sales potential in rural and urban markets (Adelakun *et al.*, 2011). Although the seed pods
45 are most often used (Camciuc *et al.*, 1998; Ndangui *et al.*, 2010), mature seeds are known to
46 have superior nutritional quality. (Oyelade *et al.*, 2003) reported that okra seed is rich in proteins
47 and lipids. A Nigerian research program initiated by Nigeria reported that dry okra seeds were
48 a good source of proteins (45 %) and fat (34 %) (Oyelade *et al.*, 2003; Adelakun *et al.*, 2009),
49 ~~classifying it as a nutrient-rich plant,~~ which could be used for feeding children and fight against
50 malnutrition. ~~The high nutritional potential of okra seeds was also evidenced. In the Republic~~
51 ~~of the Congo, research about okra seeds began in 2010 with Ndangui and Kimbonguila~~ Ndangui
52 *et al.* (2010), who determined the proximate composition of okra seeds (24 % proteins, 24 %
53 fat, 36 % carbohydrates), ~~and were concluded that okra have high nutritional potential. The~~
54 ~~nutritional importance of okra seeds reawakened interest in bringing the crop into commercial~~
55 ~~production.~~ Although extensive information is available about nutritional quality of okra seeds,
56 no study, to our knowledge, focused on the distribution of nutritional compounds within
57 granulometric classes of okra seed powders produced by milling processes. In fact, it can be
58 hypothesized that large particles may be richer in fibers, which are difficult to grind; hence,
59 small particles may be enriched in minerals, fat, and proteins and have a higher nutritional
60 quality, as previously reported for various plant parts (Becker *et al.*, 2016; Zaiter *et al.*, 2016;
61 Deli *et al.*, 2019a). For this purpose, the current study deals with the production of okra seed
62 powders by successive milling and sieving processes, with the aim to determine if some
63 granulometric classes of okra seeds powder could both have a great nutritional quality and good
64 ~~flow properties~~ (Adelakun *et al.*, 2011; Chen *et al.*, 2015), ~~making them suitable for the~~
65 ~~production of food supplements.~~ Milling induces particle size reduction, broadening of particle
66 size distribution, and increases specific surface area, leading to the improvement of material
67 functionalities, especially regarding extraction efficiency and bioavailability of nutritional
68 ~~bioactive~~ compounds (Crayveld *et al.*, 2009; Zhao *et al.*, 2009; Zhang *et al.*, 2012; Zaiter *et al.*,

1
2
3 69 2016). The combination of milling and sieving processes could be interesting to identify
4
5 70 granulometric classes richer in fat and proteins, ~~generally concentrating bioactive compounds~~
6
7 71 (Brewer *et al.*, 2014; Becker *et al.*, 2016; Lucas-González *et al.*, 2017). ~~This would enable~~
8
9 72 ~~optimizing the extraction of bioactive compounds of plants, by reducing solvent consumption~~
10
11 73 ~~and extraction time (Zaiter *et al.*, 2016; Deli *et al.*, 2019a).~~

12 74 ~~Particle size control seems also interesting with regard to powder flowability~~ Nutritional quality
13
14 75 of food powders is not the only criterion for their production at industrial scale, their ability to
15
16 76 be transported, handled, and stored is also crucial for their manufacturing. Powder flowability
17
18 77 is one of the most important powder technofunctionalities regarding these aspects. Thus, this
19
20 78 study intended to evaluate the nutritional quality but also the flowability of granulometric
21
22 79 classes of okra seed powders. It has been shown that particle size distribution is a major
23
24 80 characteristic controlling the flowability of powders having similar proximate composition
25
26 81 (Petit *et al.*, 2017). A decrease in particle size is often associated with an increase in cohesion
27
28 82 because the interparticular forces are increased when the distance between particles is reduced
29
30 83 owing to higher interparticular surface contact area. So, small particles are generally cohesive
31
32 84 and have a poor flowability ~~lower capacity to flow~~. The major influence of particle size on flow
33
34 85 behavior has been recently reported for many powdered foods and food supplements: for
35
36 86 instance, *foufou* and *foutou* powders (instant plantain-based powders) (Gnagne *et al.*, 2017),
37
38 87 *Boscia senegalensis*, *D. glomerata* and *H. sabdariffa* plant powders (Deli *et al.*, 2019b).

39 88 Thus, the purpose of the present study was to investigate the nutritional quality on the basis of
40
41 89 proximal composition and flow properties (flow energy, compressibility, cohesion, flow factor,
42
43 90 etc.) of these powders, as powder flowability is an important functionality for industrial
44
45 91 manufacturing, handling, and storage. This was achieved with the FT4 powder rheometer
46
47 92 (Freeman Technology) (Gnagne *et al.*, 2017), which provides comprehensive series of methods
48
49 93 to characterize powder behavior. In this study, okra seeds were milled with an ultra-centrifugal
50
51 94 mill at three rotating speeds: 6 000, 12 000, and 18 000 rpm, then the three powder samples
52
53 95 were each separated in four granulometric sizes by sieving: $\geq 500 \mu\text{m}$, 315 - 500 μm , 180 - 315
54
55 96 μm , and $\leq 180 \mu\text{m}$. The influence of proximal composition and mean particle size of
56
57 97 granulometric classes of okra seed powders on their flowability was investigated, in order to
58
59 98 conclude on the impact of successive milling and sieving processes on okra seed powder
60
61 99 nutritional quality and flow characteristics.

100 2. Material and methods

101 **2.1. Material: okra seeds**

102 Okra seeds were purchased from farmers in Impfondo locality, in the north of the Republic of
103 the Congo (1°37'7.47"N; 18°3'44.459"E). Okra were shelled (the seeds were removed from
104 the pods), then seeds were sorted to remove impurities and sun-dried in the open air at 30 °C.
105 Dry seeds were stored at 10 °C until powder production.

106 **2.2. Powder production by milling and sieving**

107 500 g sun-dried okra seeds were milled with an ultra-centrifugal mill ZM 200 (Retsch, France)
108 at three different milling speeds (6 000, 12 000, and 18 000 rpm) at room temperature (25 °C)
109 using a 20-cm sieve drilled with 1-mm trapezoidal holes. Then, obtained powder was collected
110 in sealable polyethylene bags before performing sieving.

111 **2.3. Okra seed powder sieving**

112 Okra seed powders **issued resulting** from the milling step were sieved with an Analyzer 3
113 Spartan sieve shaker (Fritsch, Idar-Oberstein, Germany) **with** 0.5 mm vibration amplitude for
114 24 min using sieves of 180, 315, and 500 µm mesh sizes. Hence, the following granulometric
115 classes were produced: ≥ 500 µm, 315 - 500 µm, 180 - 315 µm, and ≤ 180 µm.

116 The material on each sieve was collected in sealable polyethylene bags, weighed to determine
117 the mass yield of each granulometric class, and stored at 10 °C until analysis.

118 **2.4. Physical properties of okra seed powders: particle size distribution**

119 The particle size distribution of okra seed powders (unsieved powders and granulometric
120 classes) were determined by laser diffraction granulometry using dry dispersion (Mastersizer
121 3000, Malvern Instruments France, Orsay, France) at room temperature (25 °C). Dispersion
122 conditions for all powders except the ≤ 180 µm powder samples were as follows: 100 % air
123 pressure at 4 bar, 3 mm hopper gap, and 40 % feed rate. For the ≤ 180 µm powder samples, the
124 following dispersion conditions were employed: 100 % air pressure at 4 bar, 4 mm hopper gap,
125 and 70 % feed rate. The chosen size estimator was the equivalent diameter in volume.

126 The d10, d50, and d90, which are characteristic diameters representative of the particle size
127 distribution, were calculated. They represent the diameters for which 10 %, 50 % and 90 %, respectively,
128 of the particle population have lower sizes. Last, the span was used to evaluate the
129 width of the particle size distribution; it was determined according to the following equation 1:

$$130 \text{ Span} = \frac{d90 - d10}{d50} \quad (\text{Eq. 1})$$

131 **2.5. Powder proximate composition**

132 The proximate composition of all granulometric classes as well as unsieved powders were
133 evaluated with standard methods. Fat content was determined by Soxhlet method using 5 g

1
2
3 134 powder, hexane as extraction solvent, and 8 h extraction duration (Castro *et al.*, 1998). Moisture
4
5 135 content was measured by weight loss after drying 3 g powder at 103 °C for 5 h (AFNOR
6
7 136 (Agence Française de NORmalisation), 1976). Ash content was evaluated by incinerating 3 g
8
9 137 powder in a muffle furnace at 550 °C for 12 h (ADPI (American Dry Product Institute,
10
11 138 Elmhurst, IL), 2002). Total protein content was determined by Kjeldahl method (Bradstreet,
12
13 139 1954). Carbohydrate content was calculated as the difference between 100 % and the contents
14
15 140 in other components; the associated standard error was deduced by propagation of uncertainties.

141 **2.6. Characterization of powder flow properties**

142 Rheological properties of okra seed powders were measured with the FT4 powder rheometer
143 (Freeman Technology, Worcestershire, UK) using 25 mm accessories. Measurement test of
144 stability, compressibility, aeration, and shear cell were performed with the standard method of
145 powder rheometer FT4 with some modifications (Gnagne *et al.*, 2017). Detailed descriptions of
146 FT4 standard tests can be found in Freeman (2007) and Leturia *et al.* (2014).

147 **2.6.1. Stability test**

148 The stability test is intended to determine if powder flowability in unconfined (low normal
149 stress) conditions is affected when making the powder flow. Indeed, powder agglomeration,
150 segregation, or attrition may change the energy needed to make the powder flow. Thus, the
151 energy required to make the powder flow was measured in flow conditions imposed by the
152 standard stability test seven times successively by rotating a blade down and up in the
153 conditioned powder bed. Thus, the stability test consists in the succession of test and
154 conditioning cycles. Conditioning was achieved by the gentle displacement of the powder bed
155 by the rotating blade. Conditioning and test cycles were carried out at 100 mm/s blade tip speed
156 and the powder sample was loaded into an assembly of two glass cylinders, the powder bed was
157 conditioned and then the upper cylinder was removed to obtain a precise volume (25 mL) of
158 conditioned powder for tests. From the measurements of force and torque of the blade, various
159 flow parameters can be extracted; the most important were:

- 160 - Basic flowability energy (BFE, mJ), defined as the flow energy measured upon downward
161 moving of the blade during test 7.
- 162 - Specific energy (SE, mJ/g): it defines the energy per gram needed to displace the conditioned
163 powder bed during upward moving of the blade. It represents a measure of how powder flows
164 in a low-stress environment.

1
2
3 165 - Conditioned bulk density (CBD, g/mL), which is a density measurement of the powder bed in
4
5 166 conditioned state. It is thus very similar to the classical powder aerated density.

7 167 **2.6.2. Compressibility test**

9 168 For the compressibility test, the powder sample was first placed in the measurement cell (i.e.
10
11 169 two overlaid 25 mm × 10 mL glass cylinders) and submitted to three conditioning cycles. After
12
13 170 that, the vessel was split to remove any excess powder and the piston was replaced by the vented
14
15 171 piston. Then, powder was slowly compressed by the vented piston moving vertically at 0.05
16
17 172 mm. s⁻¹ at different normal stresses ranging from 0.5 to 15 kPa, and the percentage change in
18
19 173 volume of the powder bed, corresponding to powder compressibility, was measured. Powder
20
21 174 compressibility at 15 kPa CP_{15kPa} was used to calculate compacted density (CD_{15kPa}), which is
22
23 175 the mass of powder per unit volume of the powder bed, including the volume of all interparticle
24
25 176 voids (Abdullah and Geldart, 1999) when applying 15 kPa normal stress. CD_{15kPa} was
26
27 177 calculated with equation 2:

$$27 \quad 178 \quad CD_{15kPa} = \frac{100}{(100 - CP_{15kPa})} \cdot CBD$$

30 179

32 180 **2.6.3. Aeration test**

34 181 The aeration test is a quick way to evaluate the fluidization ability of a powder. The aeration
35
36 182 test consists in introducing air into the powder and evaluating how this changes the flow energy
37
38 183 (Freeman, 2007; Leturia *et al.*, 2014; Gnagne *et al.*, 2017). The powder sample was placed in
39
40 184 the measurement cell (25 mm x 35 mL glass cylinder) fitted with a porous plate and three
41
42 185 conditioning cycles were carried out to achieve a homogeneous packing state of the powder
43
44 186 sample. The aeration test was then performed by measuring the flow energy of the powder bed
45
46 187 during downward moving of the blade through the powder bed at 100 mm.s⁻¹ tip speed for air
47
48 188 velocities ranging from 0 to 5 mm s⁻¹ by 0.5 mm. s⁻¹ steps. Between each test cycle, a
49
50 189 conditioning cycle was carried out to obtain a steady state of aerated powder. The most
51
52 190 important parameters measured by the aeration test are defined below:

53 191 - Aerated energy (AE, mJ), which is the flow energy measured during downward moving of the
54
55 192 blade through the powder bed at maximal air velocity (10 mm.s⁻¹).

56 193 - Aeration ratio (AR, dimensionless) that designates the factor by which the basic flowability
57
58 194 energy (i.e. at 0 mm.s⁻¹ air velocity) is reduced when aerating the powder bed at maximal air
59
60 195 velocity of 10 mm s⁻¹. It was calculated according to equation 3:

196

$$AR = \frac{BFE}{AE} \quad (\text{Eq. 3})$$

198 **2.6.4. Shear cell test**

199 Shear cell tests are intended to determine the shear stress required to initiate flow in a pre-
200 consolidated powder, using a specially designed shear head equipment. This test provides
201 indication of how easily a powder moves from a static state to dynamic flow in confined
202 environments (i.e. at high normal stress). During the FT4 shear cell test, the sample powder is
203 subjected to a pre-consolidation step at 9 kPa normal stress before subsequent shear tests are
204 carried out at normal stresses ranging from 7 to 3 kPa by 1 kPa steps (Freeman, 2007; Yu *et al.*,
205 2011; Fu *et al.*, 2012), in which the shear stress required to cause failure of the pre-consolidated
206 powder bed (thus making the powder flow) is measured. This succession of shear tests at
207 decreasing applied normal stresses permits to build the yield locus of the normal stress vs. shear
208 stress curve, from which powder flow properties such as flow factor and powder cohesion can
209 be derived.

210 Cohesion, expressed in kPa, represents the shear stress required to make the powder bed flow
211 in unconfined conditions: indeed, the cohesive interactions between particles must be broken
212 before they can be individually put in motion. Moreover, generally, the higher the
213 interparticular forces, the more the powder bed can be compressed, and the more energy must
214 be supplied to the powder to make it flow.

215 Flow factor (ffc) is defined as the ratio between the consolidation stress and unconfined yield
216 stress: The larger the ffc value, the better the powder flows (Jenike, 1964; Rhodes, 2008).

217 Powder flow behaviors can be classified as follows (Fu *et al.*, 2012):

218 $ffc < 1$: not flowing;

219 $1 < ffc < 2$: very cohesive;

220 $2 < ffc < 4$: cohesive;

221 $4 < ffc < 10$: easy flowing;

222 $10 < ffc$: free flowing.

223 **2.7. Statistical analysis**

224 All experiments were conducted in triplicate and results were expressed as mean \pm standard
225 deviation. Statistical analysis was performed to a one-factor analysis of variance using
226 Microsoft Excel 2016, significant differences were assessed with Tukey's HSD at a 5 %
227 confidence level with the DSAASTAT add-on.

228

229 3. Results and discussion

230 3.1. Impact of milling speed on okra seed powder granulometry

231 Particle size distributions of okra seed powders obtained at the three milling speeds (6 000,
232 12 000, and 18 000 rpm) are shown in figure 1.

233

234

Figure 1

235 First, it was observed that okra seed powders were constituted of two populations: one of large
236 particles between 200 and 3 000 μm , the other of fine particles between 5 and 200 μm . This is
237 typical for seed powders obtained by milling, as the different organs of seeds (seed coat, radicle,
238 cotyledon, etc.) differ in hardness, thus resulting in various particle sizes (Chen *et al.*, 2015).

239 Then, the shift to the left of the particle size distribution at higher milling speed shows that it
240 led to smaller particles, as expected. Besides, when the milling speed was increased, the
241 proportion of fine particles raised to the detriment of the proportion of large particles. This
242 shows that some large particles, presumably having elevated hardness, were converted in finer
243 ones at higher milling speed. Also, it can be guessed that the employed ultra-centrifugal mill
244 may not allow decreasing the particle size under 10 μm , in agreement with equipment
245 specifications.

246 Moreover, figure 1 confirms the effect of the milling speed on the particle size distribution of
247 okra seed powders. It is worth noting that a more intense milling was also associated to a
248 broader dispersion of the particle size distribution. Indeed, obtained powder was less
249 monomodal when milling speed was increased, as evidenced by the increase in span. This may
250 be indicative of the occurrence of different grinding mechanisms in the employed milling
251 process. From this granulometric analysis, three mesh sizes (180, 315, and 500 μm) were
252 chosen for sieving powders into granulometric classes so that each granulometric class
253 represent a significant mass fraction of the unsieved powder.

254 3.2. Efficiency of okra seed powder sieving

1
2
3 255 Before proximate analysis and characterization of flow properties of the powders, the efficiency
4
5 256 of the milling and sieving processes for the production of different granulometric classes were
6
7 257 verified by weighing powder samples after sieving (Table 1).
8

9 258 Table 1

10
11 259 Table 1 shows that the sieving process allowed an efficient fractionation of okra seed powders
12
13 260 into the different granulometric classes, as significant mass yields (generally above 10 %) were
14
15 261 found for all sieved powders. As expected from particle size distributions of unsieved powders
16
17 262 (figure 1), the $\geq 500 \mu\text{m}$ granulometric class was the most represented (around 50 % mass yield)
18
19 263 whatever the milling speed. This is consistent with the fact that seeds are material of rather high
20
21 264 hardness, making them difficult to grind, and resulting into powders containing a marked
22
23 265 proportion of large particles.

24 266 3.3. Particle size distribution

25
26 267 Particle size distribution of all granulometric classes and unsieved powder are displayed in
27
28 268 figures 2, 3, and 4 and associated granulometric parameters (d_{10} , d_{50} , d_{90} , and span) are
29
30 269 presented in table 2.

31
32 270 Figure 2

33
34 271 Figure 3

35
36 272 Figure 4

37
38 273 Figures 2, 3, 4 and table 2 show that okra seed powders were correctly fractionated by sieving
39
40 274 despite their rather large fat content (that will be discussed in the following subsection 3.4)
41
42 275 likely to make them sticky. Indeed, particle size distributions of sieved powders were generally
43
44 276 monomodal, with small span values (except for $\leq 180 \mu\text{m}$ granulometric classes whatever the
45
46 277 milling speed and the 180 - 315 μm granulometric class at 12 000 rpm milling speed).

47 278 The large width of particle size distributions (span) of $\leq 180 \mu\text{m}$ granulometric classes whatever
48
49 279 the milling speed and of the 180 - 315 μm granulometric class at 12 000 rpm milling speed may
50
51 280 be explained by the presence of numerous fine particles (likely coming from the attrition of
52
53 281 larger particles in the course of the milling process) that may adhere to larger ones and thus
54
55 282 may be difficult to separate into individual particles. Indeed, as it will be evidenced in the next
56
57 283 subsection (3.4) by proximate composition results, okra seed powders were rich in fat, which
58
59 284 may favor powder cohesion and result in their agglomeration during the milling process,
60
285 especially for fine particles, as they had a higher specific surface area and contained more fat.

1
2
3 286 Whatever it may be, the results of sieve fractionation (table 1) and particle size characteristics
4
5 287 of powder fractions (table 2) showed that the successive milling and sieving procedure was
6
7 288 effective in micronizing okra seeds and separating unsieved powders into sufficiently different
8
9 289 size classes. Indeed, the d50 of sieved powders (granulometric classes) was systematically
10
11 290 found between the mesh sizes of upper and lower sieves, which confirms the suitability of sieve
12
13 291 shaking for separating okra seed powders.

14 292 Milling speed contributed to reduce mean particle size and span of granulometric classes (cf.
15
16 293 figures 2, 3, and 4 and table 2), as expected. Both the decrease in particle sizes and the increase
17
18 294 in the width of the particle size distribution at higher milling speed were consistent with the
19
20 295 more elevated mass fractions of $\leq 180 \mu\text{m}$, $180 - 315 \mu\text{m}$, and $315 - 500 \mu\text{m}$ granulometric
21
22 296 classes (table 1).

23
24 297 Table 2

25 298 3.4. Proximate composition

26
27 299 In table 3, contents in moisture, fat, proteins, ash, and carbohydrates of all granulometric classes
28
29 300 and unsieved powders of okra seeds were evaluated to investigate the influence of milling speed
30
31 301 and sieving efficiency. Indeed, proximate composition may be indicative of the stickiness of
32
33 302 powder surface (for instance, surface fat generally tend to increase powder stickiness) (Langrish
34
35 303 *et al.*, 2006; Viviane *et al.*, 2007), which is known to greatly impair powder flowability.

36
37 304 Table 3

38
39 305 Moisture content of granulometric classes $\leq 180 \mu\text{m}$, $180 - 315 \mu\text{m}$, and $315 - 500 \mu\text{m}$ was
40
41 306 lower than 10 %, whereas unsieved powders and $\geq 500 \mu\text{m}$ samples had a moisture content
42
43 307 ranging between 10 and 13 %. It is generally considered that a moisture content lower than 10
44
45 308 % ensures powder stability during storage (Kaur, Kaushal and Sandhu, 2013). Then,
46
47 309 granulometric classes composed of smaller particles were expected to have better preservation
48
49 310 abilities than unsieved powders and $\geq 500 \mu\text{m}$ samples. Also, fat and protein contents
50
51 311 significantly decreased when the particle size was increased, unlike carbohydrates and ash that
52
53 312 increased. As regards the influence of milling speed, it appeared that the contents in fat and
54
55 313 proteins (resp., carbohydrates and ash) decreased (resp., increased) in $\leq 180 \mu\text{m}$ and $180 - 315$
56
57 314 μm granulometric classes and increased (resp., decreased) in $315 - 500 \mu\text{m}$ and $\geq 500 \mu\text{m}$
58
59 315 granulometric classes. Indeed, it was expected that a more intense milling enhances the size
60
316 reduction of plant material and thus the release of fat and proteins from plant cells (Becker *et*
317 *al.*, 2016; Zaiter *et al.*, 2016). This can be related to okra seed structure: indeed, the outer shell

1
2
3 318 is composed of a higher proportion of fibers than other parts of okra seeds. Then, as fibers are
4
5 319 more difficult to grind, larger particles may be enriched in this component, leading to higher
6
7 320 carbohydrate content. Consequently, the proportions of fat and proteins were decreased in larger
8
9 321 particles. Previous studies explained that plant parts containing a high proportion of fibers and
10
11 322 sugars (both belonging to carbohydrates) are harder to grind and thus result in larger particles
12
13 323 after milling, whereas plant parts with higher levels of fat and proteins are easier to grind and
14
15 324 thus lead to smaller particles (Becker *et al.*, 2016; Zaiter *et al.*, 2016). So, proximate
16
17 325 composition of okra seed powders was greatly dependent on granulometric class; in other
18
19 326 words, the mean particle size had a marked impact on proximate composition of okra powders.
20
21 327 The moisture content was decreased for powder samples constituted by small particles, because
22
23 328 they probably undergo a higher friction-induced heating during milling. Moreover, statistical
24
25 329 analysis permitted to show that large particles were richer in carbohydrates. Plant parts
26
27 330 containing a large proportion of fibers are expected to be more difficult to grind and result in
28
29 331 large particles. As fibers were included in total carbohydrates in the proximate composition
30
31 332 analysis, the larger content in carbohydrates of higher size granulometric classes of okra seed
32
33 333 powders may be due to a higher proportion of fibers. Bioactive compounds are often associated
34
35 334 with proteic and fatty fractions, thus the smaller granulometric classes of okra seeds are
36
37 335 expected to have higher contents in bioactive compounds (Becker, *et al* 2016; Deli, Ndjantou,
38
39 336 *et al.*, 2019). At higher milling speed, this differential distribution of macronutrients within
40
41 337 granulometric classes was enhanced: small particles contained even more proteins and fat and
42
43 338 less moisture, carbohydrates, and ashes. Thus, the discriminating power of the sieving process
44
45 339 in matter of proximate composition was improved at higher milling speed.

340 **3.5. Flow properties of okra seed powders**

341 **3.5.1. Stability and compressibility tests**

342 The results of stability tests for granulometric classes and unsieved samples of okra seed
343 powders are presented in table 4.

344 Table 4

345 Whatever the milling speed, unsieved powders presented significant differences with
346 granulometric classes in terms of BFE and SE. Moreover, the CBD of unsieved powders was
347 systematically found within the range of CBD of granulometric classes. The CBD of
348 granulometric classes decreased with particle size, indicating that the powder bed was less
349 compact for samples composed of smaller particles. This could be an indication of higher
350

1
2
3 350 cohesion of smaller granulometric classes, which is consistent with their probable stickiness
4
5 351 deriving from their higher fat content as well as with the fact that smaller particles are able to
6
7 352 establish more contact points with their surrounding environment (particles, walls), thus
8
9 353 impairing their flowability. At first glance, BFE results seemed in contradiction with this
10
11 354 conclusion, as BFE decreased with mean particle size and this could be interpreted as better
12
13 355 flowability of granulometric classes of smaller mean particle size. However, it should be
14
15 356 mentioned that powder bed with poor flow properties may also lead to low BFE values when
16
17 357 particle cohesion is so high that the blade only makes flow the small amount of powder located
18
19 358 in its vicinity (Leturia *et al.*, 2014; Gnagne *et al.*, 2017). Also, interpretations of powder
20
21 359 flowability on the basis of the BFE values may be distorted by important CBD differences.
22
23 360 Indeed, the stability test is performed to evaluate the flowability of a given volume of
24
25 361 conditioned powder bed, hence sample mass may vary between samples according to aerated
26
27 362 density (i.e. CBD) differences. When comparing powder sample differing in CBD, the SE
28
29 363 seems more appropriate to give information on powder flowability, as it corresponds to flow
30
31 364 energy normalized against powder mass. All okra seed powders exhibited SE values close to 5
32
33 365 mJ/g, indicating that studied powders had correct flow properties. For samples milled at 6 000
34
35 366 rpm, the lower SE (indicative of better flowability) was found for the 180 - 315 μm
36
37 367 granulometric class, while at 12 000 and 18 000 rpm milling speed, the best flow properties on
38
39 368 the basis of the SE was found for the highest granulometric class. This is consistent with the
40
41 369 fact that larger particles are expected to flow more easily (Petit *et al.*, 2017; Shi *et al.*, 2018).
42
43 370 Also, the better flowability of the 180 - 315 μm granulometric class for samples milled at 6 000
44
45 371 rpm may result from the lower span of this sample (cf. table 2).

46
47 372 Moreover, the influence of milling speed on powder flowability can be deduced by comparing
48
49 373 unsieved powders. Table 4 shows that the SE and the CBD increased, while the BFE decreased,
50
51 374 when the milling speed was increased. This trend was expected, because when the milling speed
52
53 375 increases, the mean particle size decreases and the interparticular interactions become so high
54
55 376 that powders are much cohesive and have a poorer flowability.

56
57 377 Table 4 and figures 5, 6, and 7 present the results of the compressibility tests. Whatever the
58
59 378 milling speed, compressibility markedly decreased with mean particle size, as expected: indeed,
60
379 it has previously been discussed that smaller particles are generally more cohesive, making
380 them more compressible. This confirms the better flow properties of powders composed of
381 larger particles. Unsieved powders had intermediate compressibility, comprised in the range of
382 compressibilities of granulometric classes. Compressibility of unsieved powders and

383 granulometric classes was increased at higher milling speed, consistently with the lower mean
384 particle size, and this was expected to lower powder flowability.

385 Final densities after compression at 15 kPa CD_{15kPa} ranged between 0.5 and 0.7 g/mL for all
386 powders, and these rather elevated values confirmed that the flowability of studied powders
387 was between correct and good. All these interpretations of compressibility results were valid
388 whatever the applied normal stress (cf. figures 5, 6, and 7).

389 Figure 5

390 Figure 6

391 Figure 7

393 3.5.2. Aeration and shear cell tests

394 Aeration results of granulometric classes and unsieved samples of okra seed powders are
395 presented in table 5.

396 Table 5

397 All investigated okra seed powders were not fluidizable in standard conditions of the FT4
398 aeration test (air velocity up to 10 m.s⁻¹). Indeed, very low aeration ratios AR and elevated
399 aerated energies AE were found, showing that the fluidized state was not achieved at maximal
400 air velocity employed in the standard FT4 test. This may be explained by the high mean particle
401 size of 180 - 315 μm , 315 - 500 μm , and $\geq 500 \mu\text{m}$ granulometric classes and the presumed
402 relatively high cohesion of $\leq 180 \mu\text{m}$ and 180 - 315 μm granulometric classes (Gnagne et al.,
403 2017; Petit et al., 2017). The low fluidizability of studied powders may constitute an advantage
404 for handling purposes at industrial scale, as loss of these powders by projection in the
405 surrounding air may be reduced. Aeration properties seemed not to be affected by changing the
406 milling speed, which is logical, as the order of magnitude of mean particle size of powder
407 samples was the same whatever the milling speed.

408 Shear test results are displayed in table 5 and figures 8, 9, and 10. Whatever the milling speed,
409 the flow factor decreased when decreasing the mean particle size of granulometric classes,
410 consistently with the increase in powder cohesion. Indeed, smaller particles are expected to be
411 more cohesive (more interparticular contact points) and thus flow more poorly. Investigated
412 powders ranged from cohesive to easy-flowing, according to Jenike classification (Jenike,
413 1964). Besides, the trends of decrease in flow factor and increase in cohesion at higher milling

1
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3 414 speed were in agreement with the decreased mean particle size of unsieved powders. The results
4
5 415 of the shear test met those of stability and compressibility tests, as powders with lower mean
6
7 416 particle size needed more energy to flow and were more compressible. This permits to state that
8
9 417 in our study the shear cell and compressibility tests were most suitable for sorting powders on
10
11 418 the basis of their flowability.

12 419 Figure 8

13 420 Figure 9

14 421 Figure 10

18 422 **4. Conclusion**

20 423 The objective of this work was to determine the impact of particle size class of okra seed
21
22 424 powders on their proximate composition and their flowability. Three different milling speeds
23
24 425 were used to produce powders that were each separated in four granulometric sizes by sieving.
25
26 426 It was found, as expected, that the mean particle size decreased when the milling speed was
27
28 427 increased. The sieving process was successful in separating unsieved powders into sufficiently
29
30 428 different size classes: a significant mass yield (generally above 10 %) was obtained for each
31
32 429 sieved powder. Mean particle size was found to be comprised between the upper and lower
33
34 430 sieves, confirming the efficiency of the sieving process. A strong correlation was found between
35
36 431 mean particle size and proximate composition: more proteins and fat and less water,
37
38 432 carbohydrates, and minerals were obtained for smaller granulometric classes. Also, the
39
40 433 influence of milling speed and mean particle size of sieved powders on flow properties was
41
42 434 denoted. Most samples were cohesive or easy-flowing. $\leq 180\mu\text{m}$, granulometric classes, having
43
44 435 a lower mean particle size and a higher fat content, were significantly more compressible and
45
46 436 cohesive than other samples. This information regarding the influence of milling speed and
47
48 437 mean particle size on flow properties of okra seed powders should be useful for the production
49
50 438 at industrial scale of okra seed powders, especially for handling, packaging, and storage
51
52 439 purposes. A compromise should be found between content in nutritional quality and/or content
53
54 440 in bioactive compounds, mass yields of the successive milling and sieving processes, and flow
55
56 441 properties.

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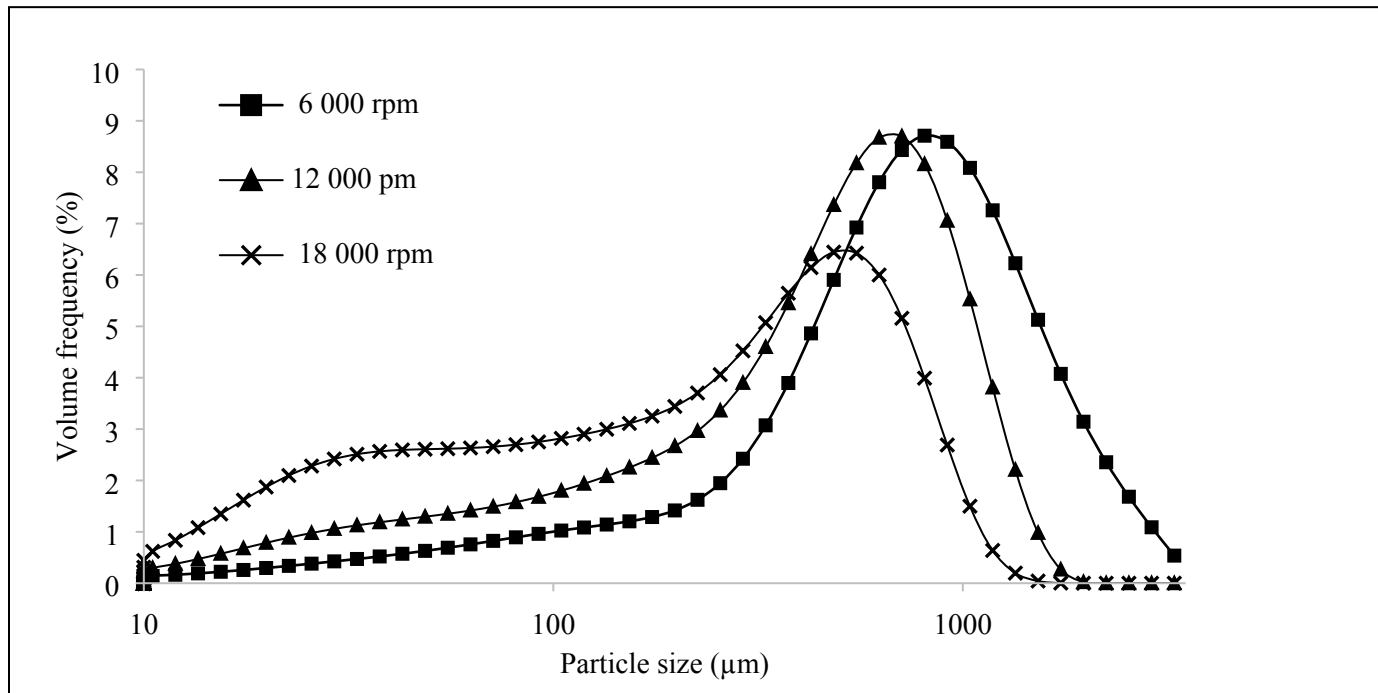


Figure 1: Particle size distributions of okra seed unsieved powders obtained at 6 000, 12 000, and 18 000 rpm milling speed.

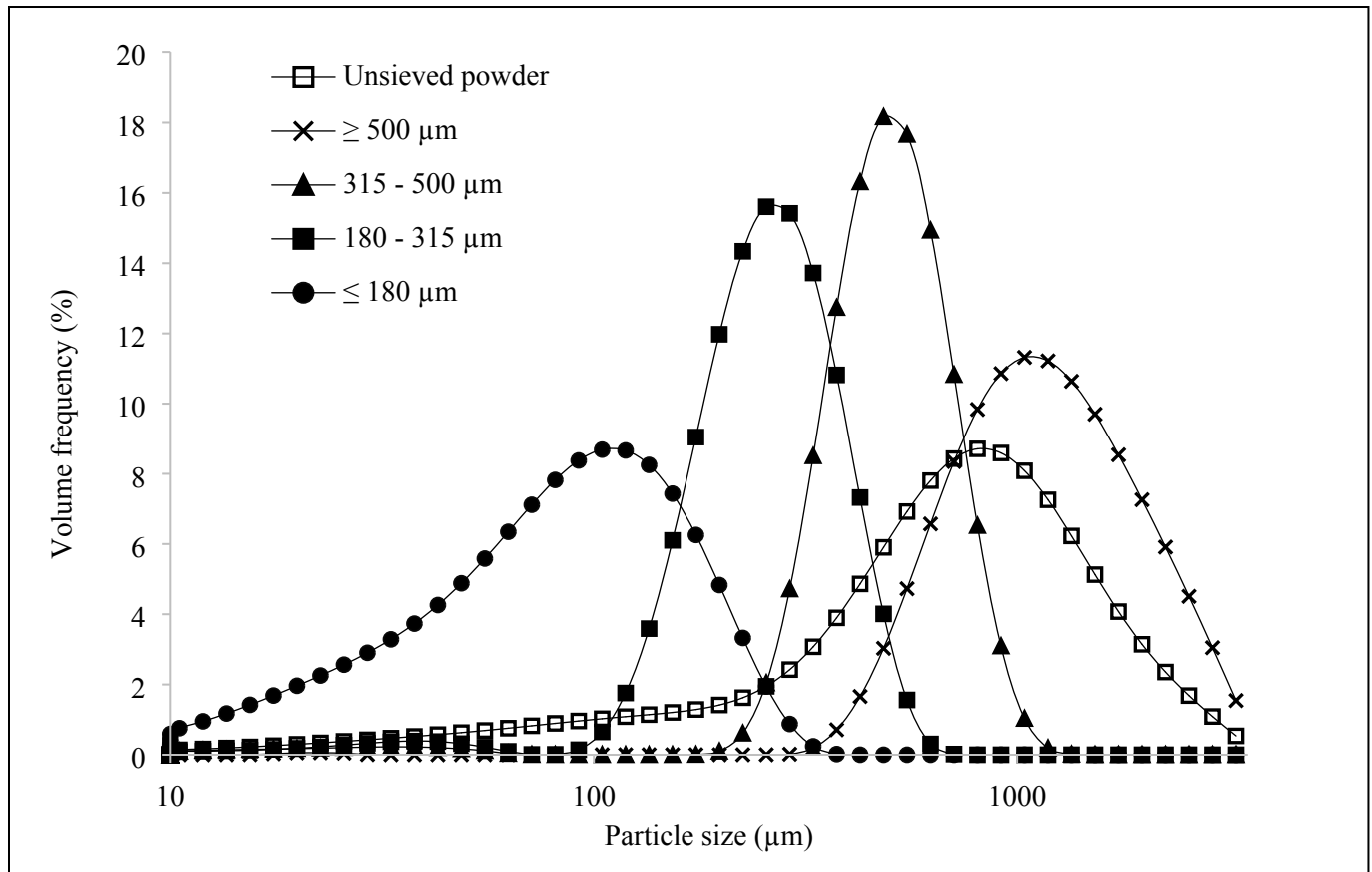


Figure 2: Particle size distributions of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 6 000 rpm.

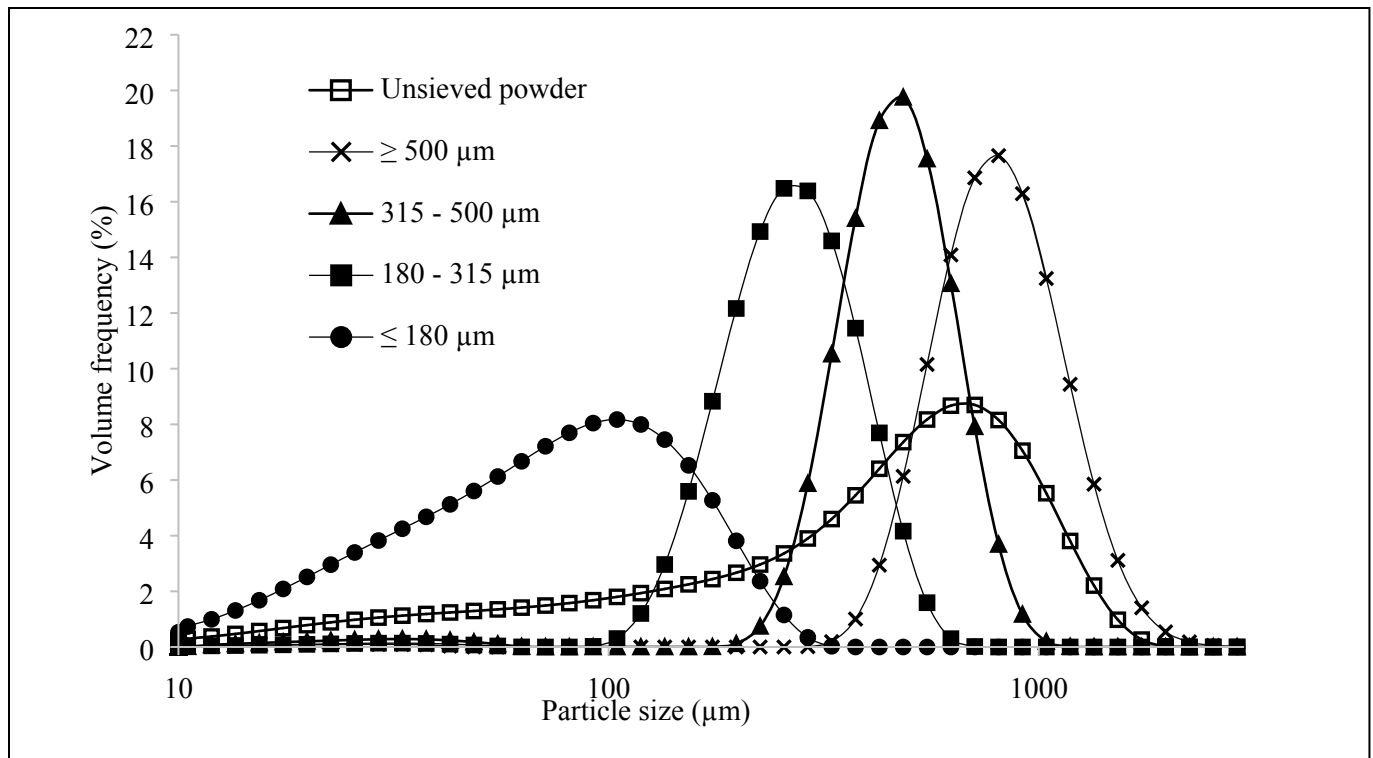


Figure 3: Particle size distributions of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 12 000 rpm.

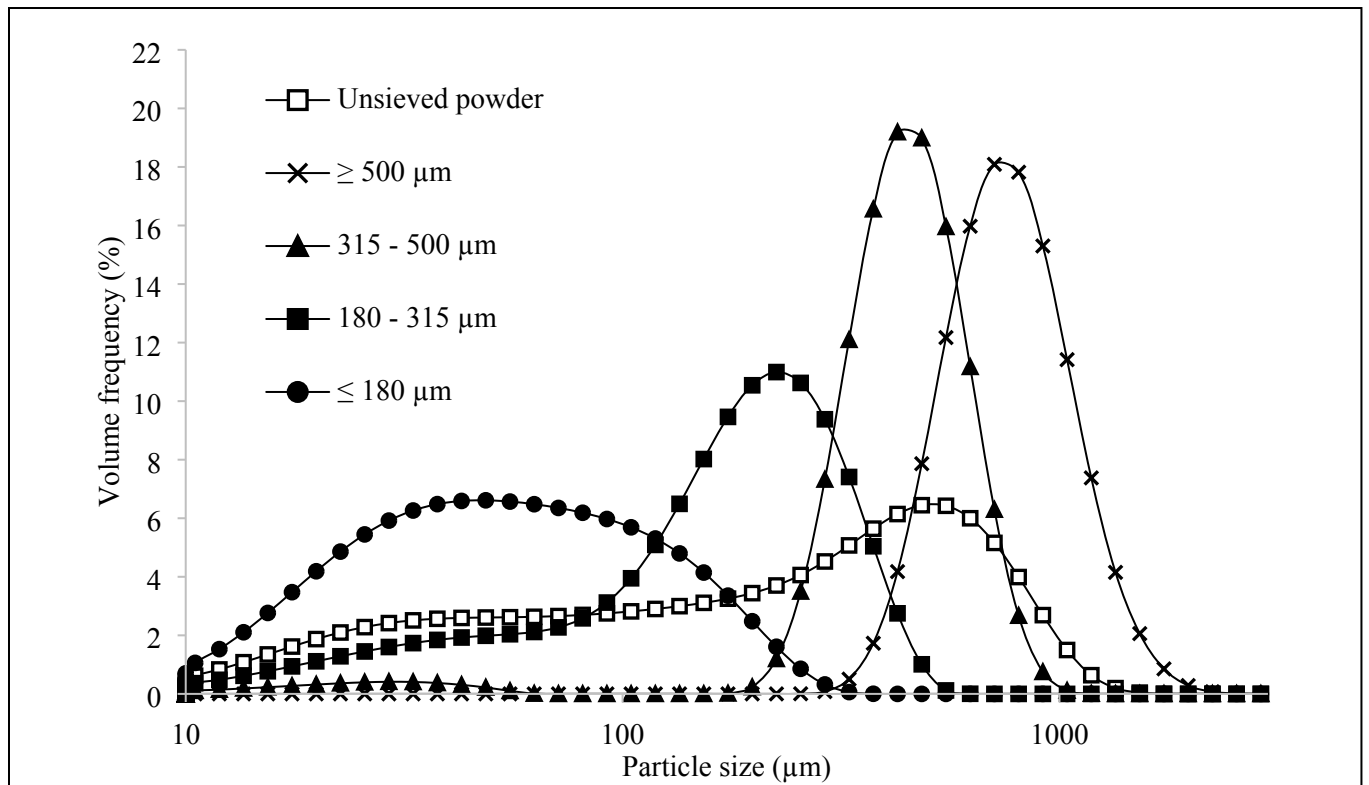


Figure 4: Particle size distributions of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 18 000 rpm.

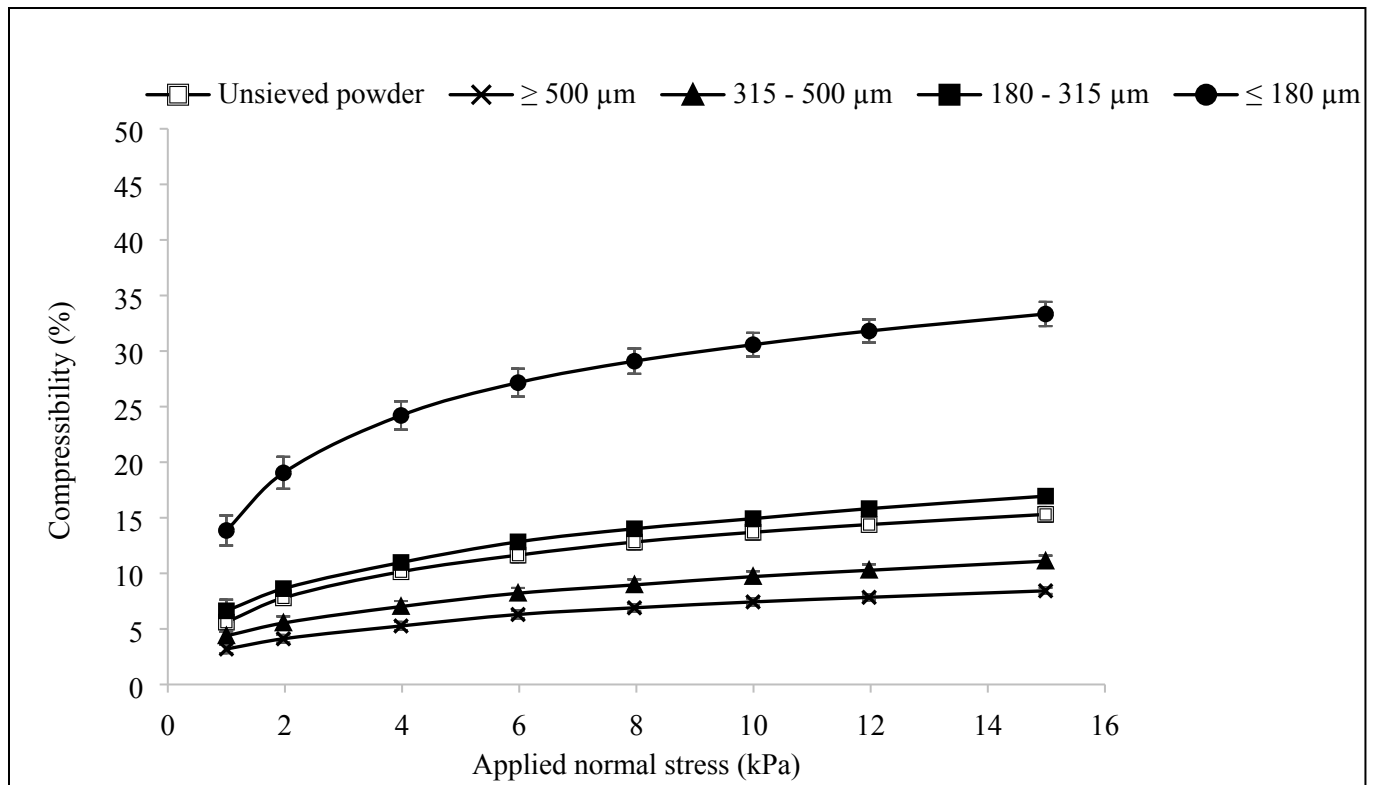


Figure 5: Compressibility of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 6 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

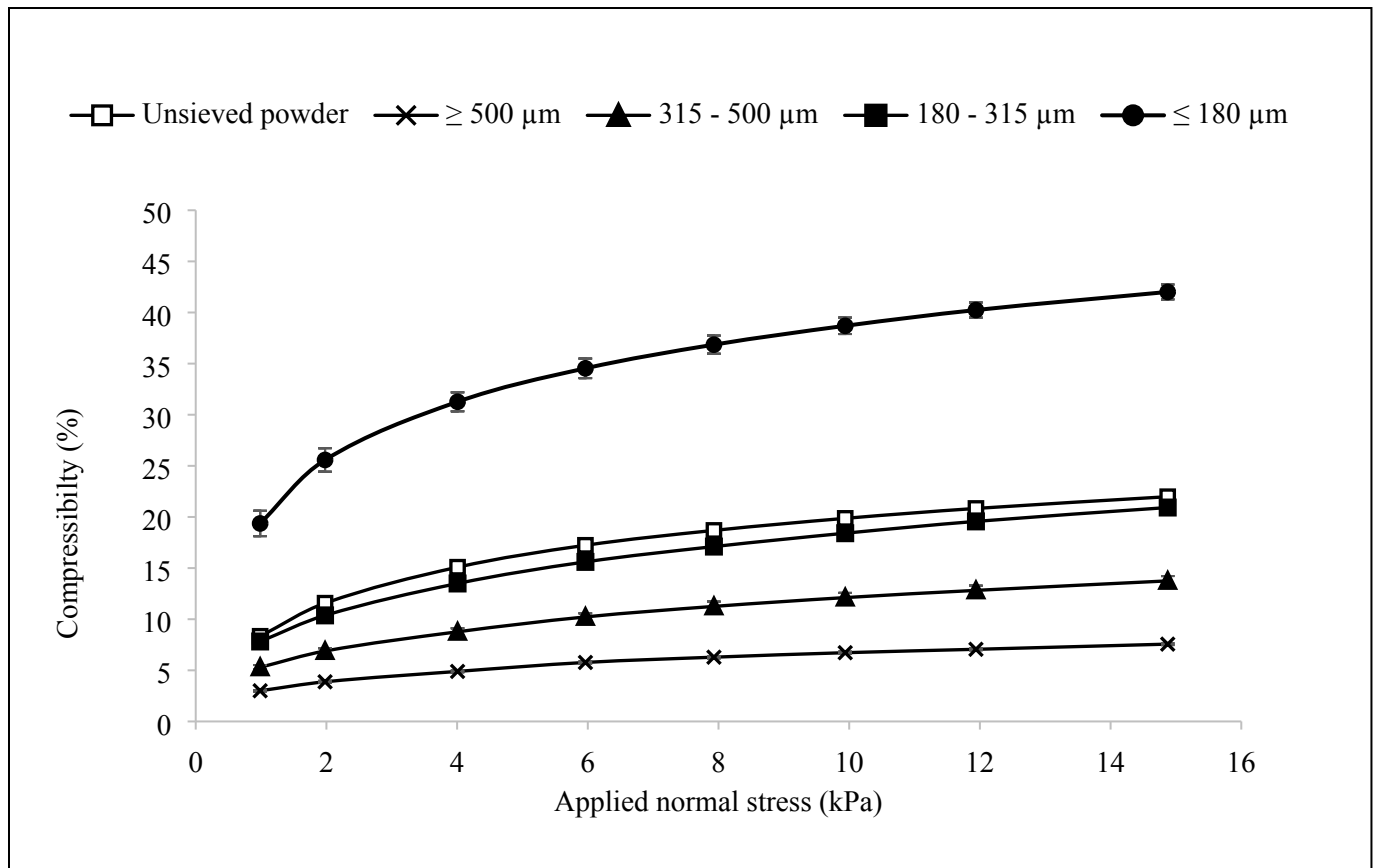


Figure 6: Compressibility of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 12 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

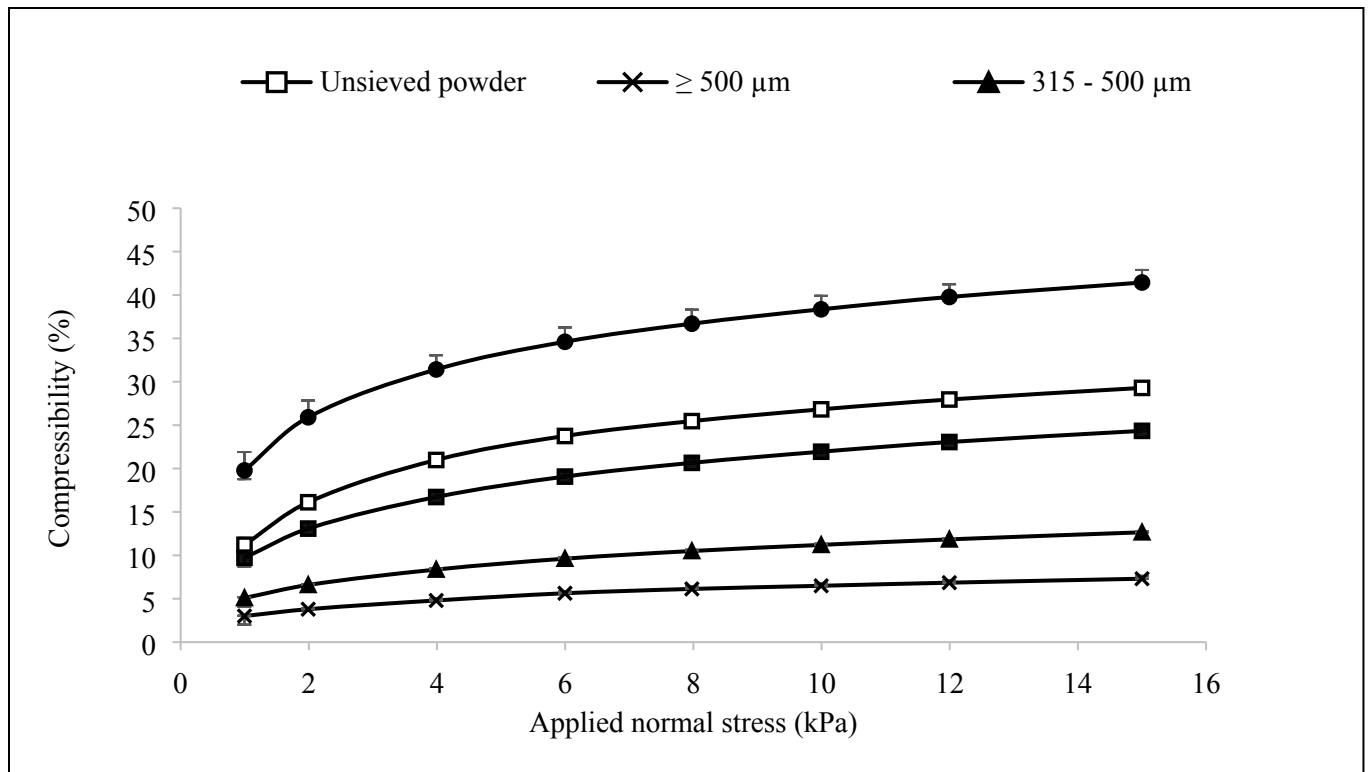


Figure 7: Compressibility of the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 18 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

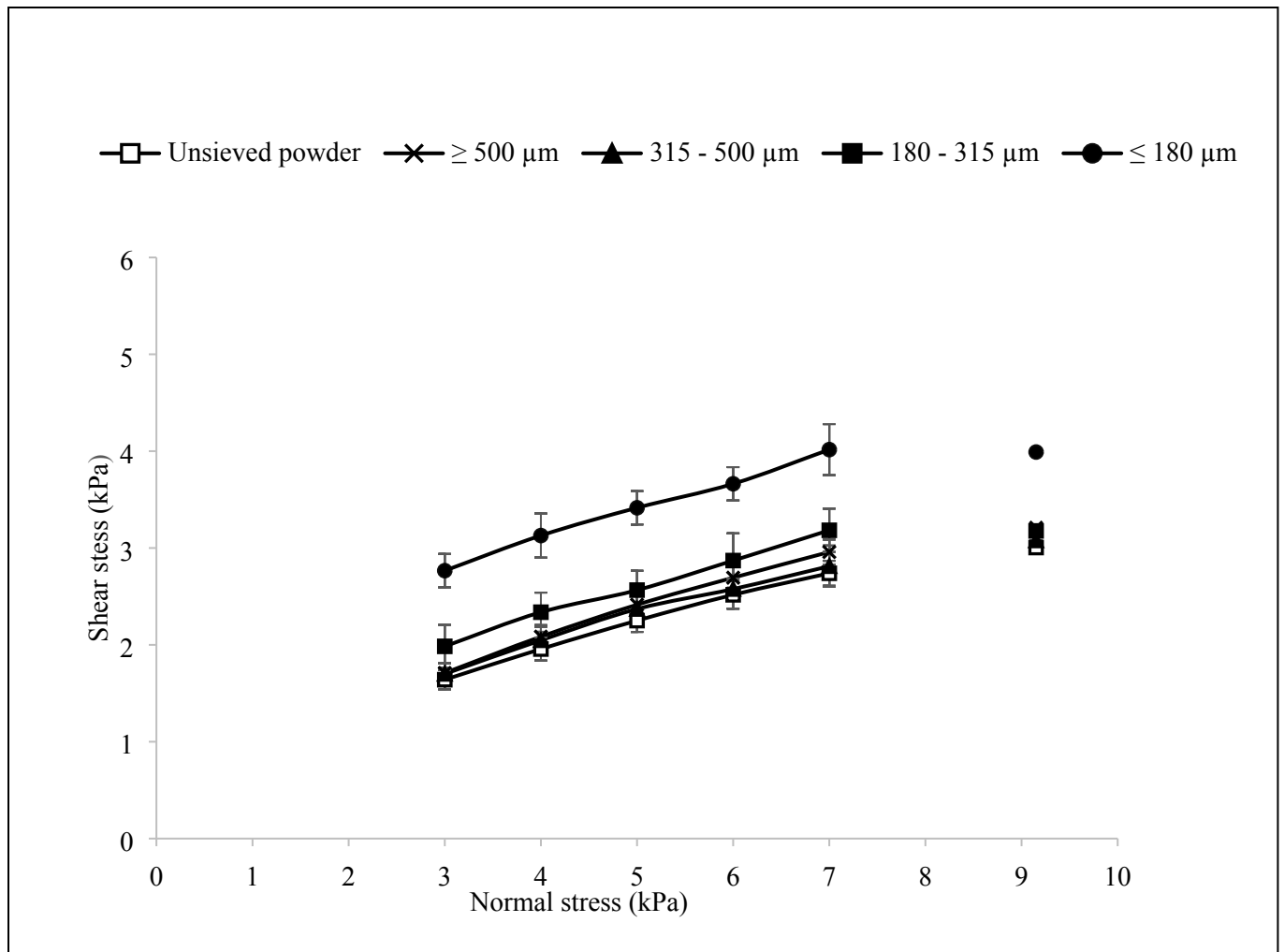


Figure 8: Shear test curve obtained after pre-shear at 9 kPa normal stress for the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 6 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

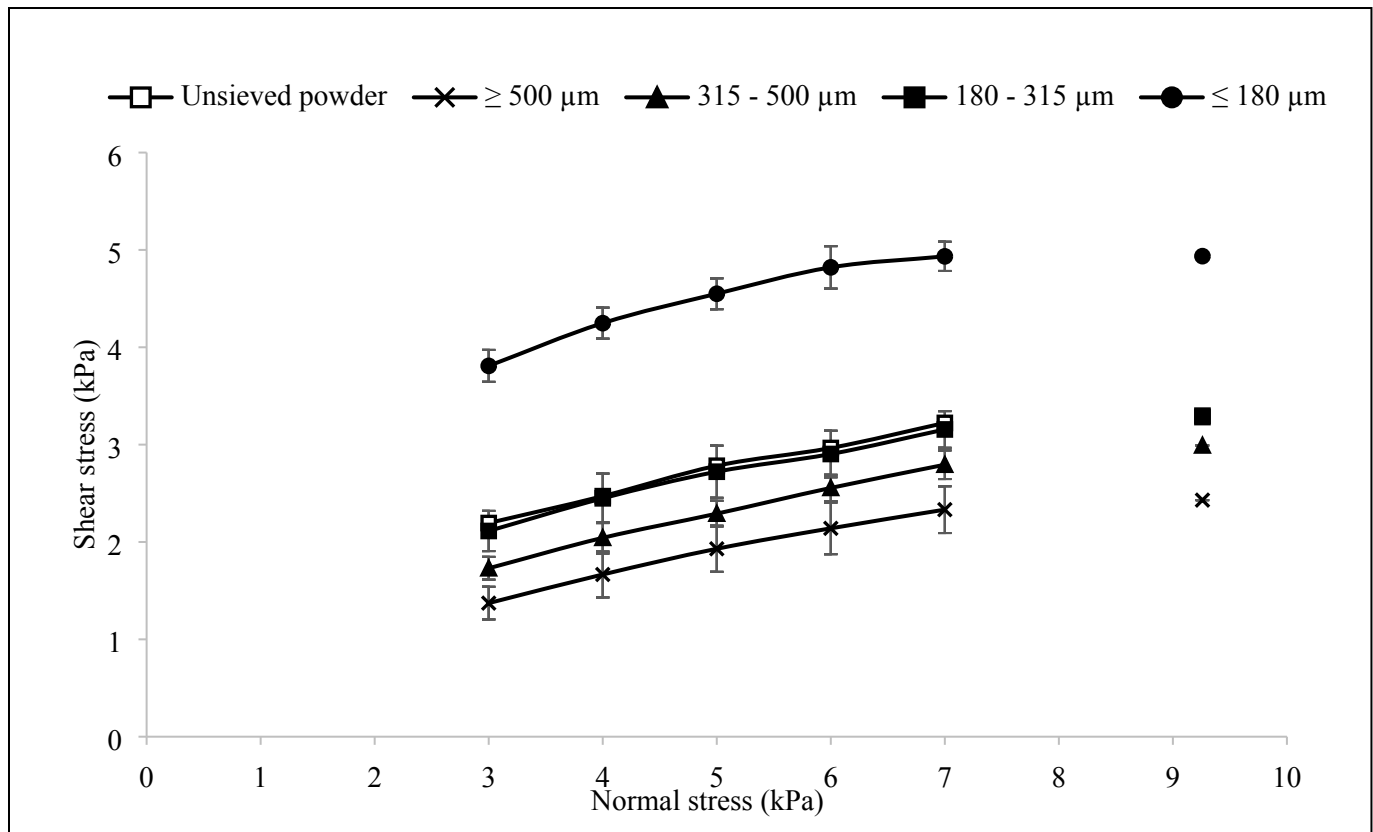


Figure 9: Shear test curve obtained after pre-shear at 9 kPa normal stress for the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 12 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

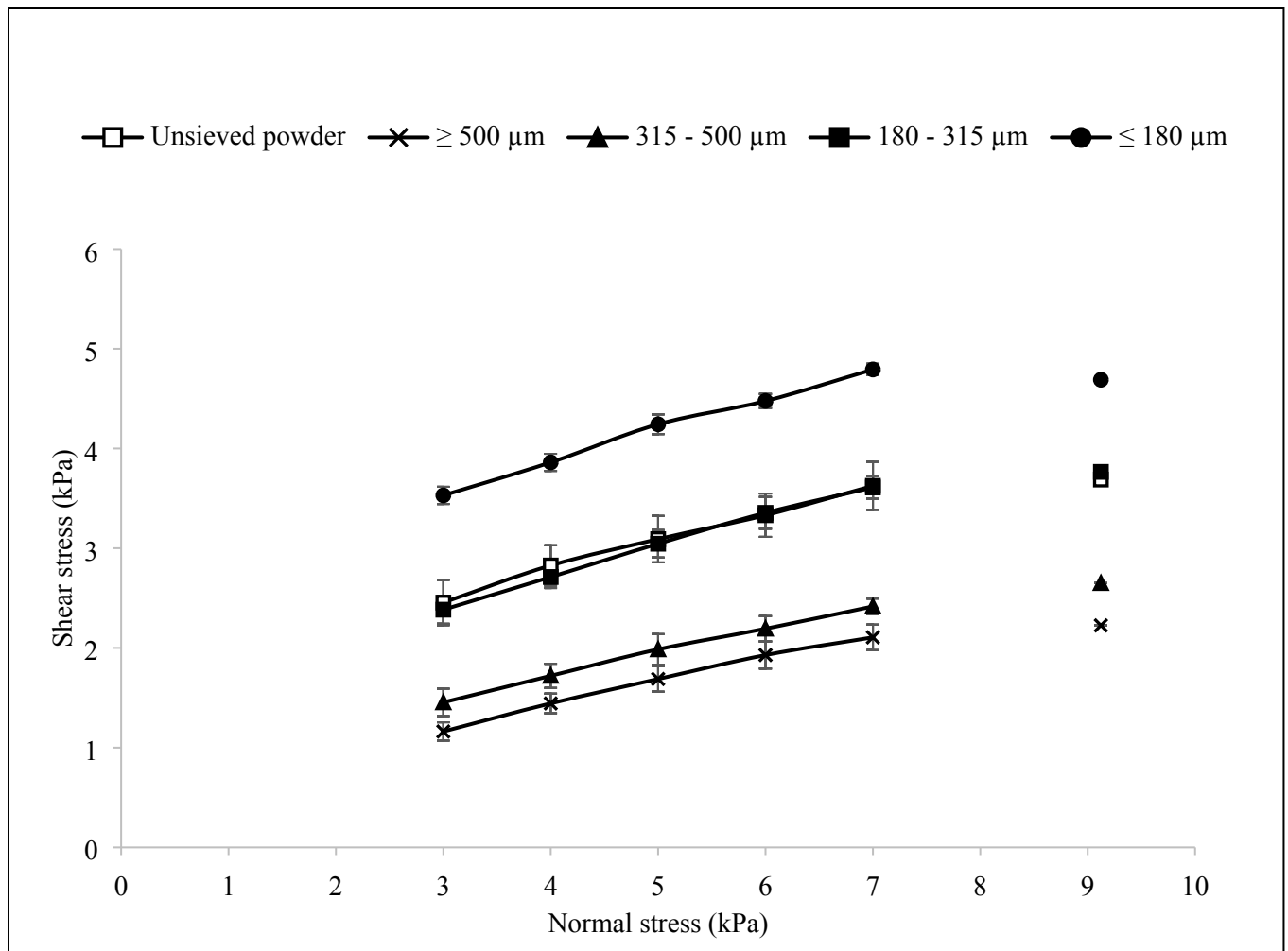


Figure 10: Shear test curve obtained after pre-shear at 9 kPa normal stress for the different granulometric classes and unsieved sample of okra seed powders obtained by milling at 18 000 rpm. Error bars represent standard deviations; some were smaller than the marker size and thus not visible.

Table 1: Sieved masses and mass fractions of the different granulometric classes and unsieved powders of okra seed powders obtained at 6 000, 12 000, and 18 000 rpm milling speed.

Milling speed (rpm)	6 000		12 000		18 000	
Granulometric class	Sieved mass (g)	Mass yield (%)	Sieved mass (g)	Mass yield (%)	Sieved mass (g)	Mass yield (%)
$\geq 500 \mu\text{m}$	317.53	59.13	164.18	50.26	86.83	42.47
315 - 500 μm	101.05	18.82	52.34	16.02	41.75	20.42
180 - 315 μm	51.16	9.53	44.68	13.68	46.32	22.66
$\leq 180 \mu\text{m}$	67.26	12.52	65.44	20.04	29.54	14.45
Unsieved powder (total)	537.00	100.00	326.64	100.00	204.44	100.00

Table 2: Granulometric parameters of the different granulometric classes and unsieved samples of okra seed powders obtained at 6 000, 12 000, and 18 000 rpm milling speed.

Milling speed (rpm)	6 000			
Granulometric classes	d10 (µm)	d50 (µm)	d90 (µm)	Span (-)
≥ 500 µm	589.33 ± 18.50 ^d	1100.00 ± 43.59 ^e	2173.33 ± 73.71 ^e	1.44 ± 0.02 ^c
315 - 500 µm	326.67 ± 6.03 ^c	498.33 ± 2.52 ^c	745.33 ± 2.52 ^c	0.87 ± 0.03 ^a
180 - 315 µm	152.00 ± 1.00 ^b	257.00 ± 1.00 ^b	406.00 ± 1.00 ^b	0.97 ± 0.01 ^b
≤ 180 µm	23.10 ± 0.20 ^a	85.30 ± 0.61 ^a	181.33 ± 0.58 ^a	1.87 ± 0.01 ^d
Unsieved powder	141.00 ± 7.81 ^b	723.33 ± 29.96 ^d	1670.00 ± 65.57 ^d	2.11 ± 0.02 ^e
Milling speed (rpm)	12 000			
Granulometric classes	d10 (µm)	d50 (µm)	d90 (µm)	Span (-)
≥ 500 µm	542.67 ± 5.69 ^e	801.33 ± 25.02 ^d	1243.33 ± 75.06 ^e	0.90 ± 0.05 ^b
315 - 500 µm	312.00 ± 1.00 ^d	463.33 ± 1.15 ^c	671.67 ± 4.93 ^c	0.78 ± 0.01 ^a
180 - 315 µm	165.67 ± 1.53 ^c	264.67 ± 1.53 ^b	409.33 ± 9.81 ^b	0.92 ± 0.03 ^b
≤ 180 µm	21.90 ± 0.10 ^a	75.60 ± 2.84 ^a	167.33 ± 6.35 ^a	1.95 ± 0.06 ^c
Unsieved powder	52.93 ± 0.58 ^b	457.67 ± 5.13 ^c	983.00 ± 7.81 ^d	2.03 ± 0.01 ^c
Milling speed (rpm)	18 000			
Granulometric classes	d10 (µm)	d50 (µm)	d90 (µm)	Span (-)
≥ 500 µm	527.67 ± 4.73 ^e	781.00 ± 14.18 ^e	1186.67 ± 37.86 ^d	0.84 ± 0.02 ^b
315 - 500 µm	290.00 ± 4.36 ^d	442.67 ± 12.86 ^d	644.67 ± 25.93 ^c	0.80 ± 0.02 ^b
180 - 315 µm	37.40 ± 0.17 ^c	181.00 ± 0.00 ^b	332.67 ± 2.08 ^b	0.48 ± 0.01 ^a
≤ 180 µm	17.83 ± 0.25 ^a	52.13 ± 0.65 ^a	149.33 ± 1.53 ^a	0.77 ± 0.01 ^b
Unsieved powder	25.70 ± 0.36 ^b	228.67 ± 7.51 ^c	702.67 ± 55.77 ^c	2.96 ± 0.16 ^c

For powder results at each milling speed separately, means ± standard deviations followed by the same superscripted letters in the same column were not significantly different at $p < 0.05$ according to Tukey's HSD test.

Table 3: Proximate composition (contents in moisture, lipids, proteins, ash, carbohydrates on wet basis) of the different granulometric classes and unsieved powders of okra seeds.

Milling speed (rpm)	6 000					
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	Unsieved powder [3]
Moisture (‰(w/w))	11.10 ± 0.07 ^b	9.59 ± 0.12 ^a	9.01 ± 0.67 ^a	9.21 ± 0.11 ^a	12.07 ± 0.05 ^c	9.45 ± 0.80
Fat (‰(w/w))	12.05 ± 0.07 ^b	23.61 ± 0.01 ^c	23.14 ± 0.43 ^c	23.34 ± 0.02 ^c	7.63 ± 0.20 ^a	24.90 ± 0.55
Proteins (‰(w/w))	16.45 ± 0.74 ^b	32.08 ± 0.81 ^d	30.07 ± 1.19 ^{cd}	28.77 ± 0.29 ^c	10.77 ± 0.62 ^a	24.85 ± 1.50
Carbohydrates (‰(w/w))	55.44 ± 0.82 ^b	28.33 ± 0.77 ^a	30.28 ± 1.63 ^a	32.12 ± 0.42 ^a	68.70 ± 8.04 ^c	36.83 ± 1.78
Ash (‰(w/w))	4.96 ± 0.04 ^a	6.39 ± 0.01 ^b	7.00 ± 0.66 ^b	6.56 ± 0.07 ^b	4.21 ± 0.46 ^a	5.68 ± 0.10
Milling speed (rpm)	12 000					
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	Unsieved powder [3]
Moisture (‰(w/w))	11.27 ± 0.08 ^c	9.15 ± 0.10 ^b	8.61 ± 0.08 ^a	9.22 ± 0.05 ^b	11.79 ± 0.06 ^d	9.45 ± 0.80
Fat (‰(w/w))	13.71 ± 0.89 ^b	26.69 ± 0.54 ^e	23.40 ± 0.27 ^d	19.21 ± 0.22 ^c	5.18 ± 0.17 ^a	24.90 ± 0.55
Proteins (‰(w/w))	17.04 ± 0.31 ^b	34.35 ± 0.62 ^e	30.61 ± 0.27 ^d	27.63 ± 1.12 ^c	7.84 ± 0.30 ^a	24.85 ± 1.50
Carbohydrates (‰(w/w))	51.01 ± 0.35 ^d	18.79 ± 0.68 ^a	30.53 ± 0.20 ^b	37.58 ± 1.19 ^c	70.33 ± 0.35 ^e	36.83 ± 1.78
Ash (‰(w/w))	5.12 ± 0.15 ^b	6.73 ± 0.04 ^d	6.23 ± 0.04 ^c	5.83 ± 0.09 ^c	4.09 ± 0.35 ^a	5.68 ± 0.10
Milling speed (rpm)	18 000					
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	Unsieved powder [3]
Moisture (‰(w/w))	10.69 ± 0.09 ^c	9.56 ± 0.08 ^a	10.15 ± 0.02 ^b	11.32 ± 0.06 ^d	13.20 ± 0.17 ^e	9.45 ± 0.80
Fat (‰(w/w))	14.57 ± 0.69 ^c	27.80 ± 0.51 ^e	23.51 ± 0.08 ^d	12.17 ± 0.15 ^b	3.43 ± 0.43 ^a	24.90 ± 0.55
Proteins (‰(w/w))	18.37 ± 0.12 ^b	35.01 ± 0.26 ^c	28.62 ± 0.24 ^d	19.72 ± 0.72 ^c	5.83 ± 0.53 ^a	24.85 ± 1.50
Carbohydrates (‰(w/w))	50.99 ± 1.23 ^c	24.73 ± 0.68 ^a	33.33 ± 0.88 ^b	52.94 ± 2.52 ^c	73.19 ± 0.76 ^d	36.83 ± 1.78
Ash (‰(w/w))	5.18 ± 0.04 ^c	6.80 ± 0.01 ^e	5.94 ± 0.06 ^d	4.39 ± 0.16 ^b	3.75 ± 0.28 ^a	5.68 ± 0.10

Table 4: Results of stability and compressibility tests for unsieved samples and granulometric classes of okra seed powders produced at 6 000, 12 000, and 18 000 rpm milling speed.

Milling speed (rpm)	6 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm
BFE (mJ)	285.00 ± 2.27 ^d	93.12 ± 2.00 ^a	164.96 ± 10.12 ^b	197.49 ± 14.34 ^c	265.58 ± 7.92 ^d
SE (mJ/g)	6.70 ± 0.24 ^c	5.94 ± 0.14 ^{bc}	4.64 ± 0.61 ^a	4.91 ± 0.31 ^{ab}	5.50 ± 0.50 ^{ab}
CBD (g/mL)	0.53 ± 0.02 ^d	0.33 ± 0.01 ^a	0.44 ± 0.00 ^b	0.50 ± 0.01 ^c	0.58 ± 0.00 ^e
CD _{15kPa} (g/mL)	0.63 ± 0.02 ^d	0.50 ± 0.01 ^a	0.53 ± 0.00 ^b	0.57 ± 0.02 ^c	0.64 ± 0.00 ^d
CP _{15kPa} (%)	15.30 ± 0.67 ^c	33.34 ± 1.09 ^d	16.95 ± 0.00 ^c	11.10 ± 0.50 ^b	8.43 ± 0.28 ^a
Milling speed (rpm)	12 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm
BFE (mJ)	259.04 ± 6.43 ^c	89.88 ± 7.19 ^a	190.37 ± 2.47 ^b	252.10 ± 6.53 ^c	248.57 ± 7.03 ^c
SE (mJ/g)	7.08 ± 0.13 ^c	7.08 ± 0.11 ^c	5.45 ± 0.07 ^b	5.56 ± 0.23 ^b	4.50 ± 0.61 ^a
CBD (g/mL)	0.53 ± 0.01 ^c	0.33 ± 0.03 ^a	0.42 ± 0.01 ^b	0.50 ± 0.01 ^c	0.61 ± 0.02 ^d
CD _{15kPa} (g/mL)	0.67 ± 0.01 ^b	0.57 ± 0.06 ^a	0.53 ± 0.00 ^a	0.57 ± 0.01 ^a	0.66 ± 0.02 ^b
CP _{15kPa} (%)	21.98 ± 0.18 ^c	42.01 ± 0.89 ^d	20.92 ± 0.62 ^c	13.75 ± 0.58 ^b	7.56 ± 0.11 ^a
Milling speed (rpm)	18 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm
BFE (mJ)	219.48 ± 3.48 ^b	88.09 ± 17.89 ^a	204.28 ± 17.54 ^b	311.58 ± 7.16 ^c	299.17 ± 21.76 ^c
SE (mJ/g)	7.12 ± 0.41 ^b	6.88 ± 0.61 ^b	6.49 ± 0.19 ^{ab}	6.14 ± 0.21 ^{ab}	5.68 ± 0.22 ^a
CBD (g/mL)	0.47 ± 0.00 ^c	0.33 ± 0.03 ^a	0.42 ± 0.01 ^b	0.51 ± 0.01 ^d	0.60 ± 0.01 ^e
CD _{15kPa} (g/mL)	0.66 ± 0.01 ^b	0.56 ± 0.04 ^a	0.56 ± 0.01 ^a	0.59 ± 0.01 ^a	0.65 ± 0.02 ^b
CP _{15kPa} (%)	29.29 ± 0.74 ^d	41.44 ± 1.77 ^e	24.35 ± 0.42 ^c	12.66 ± 0.16 ^b	7.31 ± 0.39 ^a

Means ± standard deviations followed by the same superscripted letters in the same row were not significantly different at $p < 0.05$ according to Tukey's HSD test.

Table 5: Aeration and shear cell test results for unsieved powders and granulometric classes of okra seed powders obtained at 6 000, 12 000, and 18 000 rpm milling speed.

Milling speed (rpm)		6 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	
Aerated energy AE (mJ)	85.77 ± 13.53 ^b	21.12 ± 2.56 ^a	44.26 ± 2.40 ^a	51.45 ± 12.88 ^a	108.48 ± 20.79 ^b	
Aeration ratio AR (-)	1.25 ± 0.07 ^a	2.53 ± 0.19 ^b	1.39 ± 0.13 ^a	1.16 ± 0.09 ^a	1.12 ± 0.10 ^a	
Cohesion (kPa)	0.84 ± 0.09 ^a	1.88 ± 0.18 ^b	1.12 ± 0.19 ^a	0.92 ± 0.08 ^a	0.81 ± 0.10 ^a	
Flow factor ffc (-)	5.29 ± 0.53 ^c	2.41 ± 0.16 ^a	3.91 ± 0.62 ^b	4.88 ± 0.42 ^{bc}	5.32 ± 0.59 ^c	
Milling speed (rpm)		12 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	
Aerated energy AE (mJ)	104.87 ± 26.69 ^a	58.21 ± 25.21 ^a	73.12 ± 9.82 ^a	80.95 ± 11.52 ^a	64.62 ± 1.39 ^a	
Aeration ratio AR (-)	1.53 ± 0.15 ^b	2.14 ± 0.23 ^c	1.26 ± 0.03 ^{ab}	1.06 ± 0.01 ^a	0.99 ± 0.08 ^a	
Cohesion (kPa)	1.45 ± 0.22 ^b	3.05 ± 0.19 ^c	1.39 ± 0.32 ^b	0.96 ± 0.15 ^{ab}	0.69 ± 0.18 ^a	
Flow factor ffc (-)	3.14 ± 0.59 ^{ab}	1.60 ± 0.09 ^a	3.34 ± 0.74 ^{ab}	4.65 ± 0.82 ^{bc}	6.44 ± 2.42 ^c	
Milling speed (rpm)		18 000				
Powder sample	Unsieved powder	≤ 180 μm	180 - 315 μm	315 - 500 μm	≥ 500 μm	
Aerated energy AE (mJ)	75.25 ± 1.37 ^b	31.91 ± 3.95 ^a	56.05 ± 18.34 ^{ab}	76.50 ± 8.16 ^b	61.15 ± 5.81 ^b	
Aeration ratio AR (-)	1.47 ± 0.04 ^a	2.09 ± 0.13 ^b	1.38 ± 0.30 ^a	1.18 ± 0.03 ^a	1.09 ± 0.20 ^a	
Cohesion (kPa)	1.64 ± 0.26 ^b	2.61 ± 0.20 ^c	1.48 ± 0.17 ^b	0.76 ± 0.22 ^a	0.48 ± 0.11 ^a	
Flow factor ffc (-)	2.80 ± 0.40 ^a	1.80 ± 0.05 ^a	3.06 ± 0.28 ^a	6.11 ± 1.24 ^b	8.98 ± 2.09 ^b	

Means ± standard deviations followed by the same superscripted letters in the same row were not significantly different at $p < 0.05$ according to Tukey's HSD test.

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3 Conflict of interests:
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5 The authors declare that they have no conflict of interest.
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For Peer Review

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3 **Ethics statements:**
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5 No experiment was performed on humans or animals. So, ethics approval was not required for
6 this research.
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3 Data Availability Statement:
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5 Research data are not shared.
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For Peer Review

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3 The authors wish to thank the reviewers for their new helpful comments. Detailed
4 point-by-point response can be found hereafter, with reviewer comments in black (line
5 numbers associated with reviewer comments correspond to submitted manuscript)
6 and answers in blue (line numbers related to manuscript revision correspond to revised
7 manuscript). In revised manuscript, additions were performed in blue and deletions in
8 red crossed words.
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15 Reviewer #1

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17 The revision is satisfactory.

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19 Thank you for your work in reviewing the manuscript.
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24 Reviewer #2

25
26 Although the work presents interesting, it is quite hard to understand the paper due to
27 poor communication. therefore i couldn't identify the main goal of this paper.Overall,
28 this is not a well-written manuscript. There were no sufficient information about the
29 previous study findings presented for readers to follow the present study rationale and
30 procedures. Finally, I recommend that the paper should not be accepted for the
31 publication in the present from.
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36 Thank you for your work in reviewing the manuscript, the authors have tried to take
37 into account these remarks in the second revised manuscript.
38
39

40 - L47: this sentence is not required it is repeating

41
42 The sentence in l41-43 indicates that okra (in general, i.e. all plant parts being
43 considered) provides carbohydrates, protein, fat, minerals, and vitamins that are usually
44 deficient in staple foods. The sentence in l47 concerns more specifically okra seeds,
45 which is the studied material. Thus, it was decided to keep this sentence giving further
46 information about the composition of okra seeds.
47
48
49

50 L49-50: Seeds are rich in proteins (45%) and lipids (34%), thus use as a food for a
51 malnutrition children in Nigeria. (reference).
52
53

54 In order to reduce the length of this sentence, the authors preferred to delete
55 "classifying is as a nutrient-rich plant". Also, "A research program initiated by Nigeria"
56 was corrected by "A Nigerian research program".
57
58

59 - L52-54: rewrite, English is not good
60

1
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3 This sentence was rewritten (cf. l51-53).
4

5 - L56: this sentence is not clear
6

7 Indeed, this convoluted sentence was removed.
8

9
10 - L58: you should discuss about this before you discuss the aim of this paper? you need
11 to explain why you need to study this and the importance of this area and its
12 relationship to human health as well as to okra
13

14
15 The link between nutritional quality and granulometric class has been added in l55-61,
16 which permits to better introduce the aim of the study. The link with human health
17 (food supplements) was added in l64-65.
18

19
20 - L59: this sentence and previous sentence has a big jump
21

22 "flow properties" was removed as it was not introduced previously. The link with the
23 distribution of nutritional compounds within granulometric classes was made through
24 the production of food supplements (cf. l64-65). The reason for studying also flow
25 properties has been developed in l74-79.
26

27
28 - L64: not clear why
29

30 Indeed, this should now be clearer with the mention of "food supplements" in l64-65.
31

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33 - L66: I what are the bioactive compounds you are going to extract not clear.
34

35 Indeed, this was out of the scope of the study, and "bioactive" as "replaced" by
36 "nutritional" (cf. l67-68).
37

38
39 - L67: if you introduce new cofounders you need to explain them.
40

41 The authors did not understand this comment, nevertheless this sentence was out of
42 the scope of the study and it was removed.
43

44
45 L68: I can't understand your English.
46

47 This sentence was deleted.
48

49
50 L70: not clear
51

52 The authors did not understand what is not clear in this sentence, and thus it as kept
53 unchanged.
54

55
56 L73: not clear
57

58 This sentence was corrected in l83-84.
59
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3 L78: your introduction and the purpose is not matching rewrite.

4 With changes made in the second revised manuscript, the authors hope that the link
5 between bibliographical data and study aim is now clearer.
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10 L92-93: rewrite

11 The authors did not understand why this sentence should be rewritten, so it was kept
12 unchanged.
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16 L94: it is important do proof reading of your paper

17 This sentence appears clear enough, so the authors do not understand why this
18 comment was formulated by reviewer #2. Maybe the fact that the sentence was
19 justified and not aligned to the left? This was changed in l105.
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22

23 L98: this sentence is too long rewrite

24 The authors did not find that this sentence is too long (42 words, 245 characters
25 including spaces), and only one idea is presented in this sentence, so it was decided to
26 keep this sentence unchanged.
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30 L101: what do you mean by issued

31 This was corrected by "resulting from"
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35 L104: you English is very poor so hard to understand.

36 The authors really did not understand this comment, as concerned sentence cannot be
37 clearer.
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41 L225-226: I can't understand what you mean by two populations.

42 It is very classical to talk about particle populations when discussing particle size
43 distribution results. A particle population gathers all particles having similar
44 characteristics, like size in the case of granulometric analysis. See here for instance:
45 http://www-odp.tamu.edu/publications/178_SR/Volume/CHAPTERS/SR178_24.PDF
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