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Original article

Effect of particle size on flow behaviour and physical properties of semi-ripe plantain (*AAB Musa* spp) powders

Kady Rosine Irie,^{1,2} Jérémy Petit,^{2*} Eliane Hadiowe Gnagne,¹ Olivier Kouadio Kouadio,¹ Claire Gaiani,² Joël Scher² & Georges N'Guessan Amani¹

1 Laboratoire de Biochimie Alimentaire et Technologies des Produits Tropicaux, UFR STA, Université Nangui Abrogoua, 02 BP 801 Abidjan 02, Abidjan, Côte d'Ivoire

2 LIBio (Laboratoire d'Ingénierie des Biomolécules), Université de Lorraine, F-54000, Nancy, France

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Summary This work was conducted to study the influence of particle size and shape on the flow behaviour of semi-ripe plantain powders. An improved plantain variety (Big Ebanga), two hybrids (FHIA 21 and PITA 3) and a local variety (Corne 1, as reference material) were powdered by successive drying, milling and sieving into three granulometric classes: unsieved (<500 µm), fine fraction (<180 µm; class 1) and coarse fraction (between 180 and 500 µm; class 2), thus differing in particle size distribution. Physical properties (water activity, particle size distribution, shape factors) were characterised, and flow properties (basic flowability energy, compressibility, fluidisability, cohesion, etc.) were investigated using the FT4 powder rheometer. Convexity of unsieved and fine powders was slightly higher than for coarse powders. Fine powders of all plantain varieties were found to have the lowest water activity and the lowest flowability in unconfined conditions. Unsieved and fine powders presented a fluidised state at the end of the FT4 standard aeration test and their minimum fluidisation velocity was low, confirming the good fluidisability of these powders.

Keywords Powder, plantain hybrids, sieving, particle size, shape factor, flowability.

Introduction

Plantain is an important food crop that significantly contributes to the food security of populations in Africa, particularly in Central, East and West Africa (Nkendah & Akyeampong, 2003; Fongang Fouepe *et al.*, 2016). It is consumed at different stages of maturity (green, semi-ripe and ripe) under many different forms (Happi Emaga *et al.*, 2007). Thus, consumers may prefer one variety to another depending on the target end use (Gilbert *et al.*, 2009). Ripe plantains, richer in starch at this maturity state, are eaten boiled, steamed, baked, fried, roasted or dried. Plantains are also used to make flour (from stages 1 and 2 of ripening) or powder (from stages 4 and 5 of ripening), which are employed as dietary supplements in developing countries. Semi-ripe plantain powder, which is a less known plantain product, has a high sugar content and may be useful in dishes requiring a sweet taste. In addition to health and sensory reasons,

ripening stages are also important for technical constraints of powder manufacturing (Alkarkhi *et al.*, 2011). Many authors (Ngalani & Crouzet, 1995; Eke-Ejiofor & Kiin-Kabari, 2012) have used semi-ripe plantain powder (at ripening stages 4 and 5) in the manufacture of breads, biscuits and instant powders for making composite breads. Banana and plantain pulps and flours have been shown to behave differently in the production of food products such as cakes and extruded products. Amongst the main powder functionalities known to impact on the manufacturing of food products from powdered ingredients, flowability is surely one of the most determinants for many processing steps, such as storage, transport and dosage. However, according to Fitzpatrick *et al.* (2004), and Lu *et al.* (2015), powders are probably the least predictable of all materials with regard to their flow behaviour because of the large number of influential physicochemical and process factors.

Several experimental and theoretical studies on the flow properties of food powders have been carried out in the past years. For instance, Gnagne *et al.* (2017) studied the flow properties of foutou and fofou

*Correspondent: Fax: +33 (0)3 83 59 57 72; e-mail: jeremy.petit@univ-lorraine.fr

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(plantain-based doughs consumed in West Africa) powders with a FT4 powder rheometer. Fu *et al.* (2012) evaluated the physical and flow properties of three lactose powders that differed in particle shape or size, confirming that in addition to particle size, the particle shape also had a significant influence on powder flow properties. Kerr *et al.* (2000) also studied the effect of milling (with a focus on particle size) on functional and physicochemical properties of cowpea flour.

Many other research studies have been undertaken in order to highlight the influence of the particle size distribution of different plant powders on physicochemical properties (Nura *et al.*, 2011, Lui *et al.*, 2015; Deli *et al.*, 2019a), antioxidant activity and polyphenol content (Zaiter *et al.*, 2016; Becker *et al.*, 2016; Becker *et al.*, 2017; Deli *et al.*, 2019b). However, the influence of particle size on the bulk and shear properties of plantain powders still needs to be clarified before the production of plantain powder can be implemented at industrial scale. Sieve fractionation is a new approach to improve the physical and functional properties of powders made from plants. This study deals with new plantain hybrids of high agronomic potential with a view to enhance the domestic or industrial uses of plantain powders. In fact, the adoption by traditional plantain consumers of powders produced from plantain hybrids rich in nutrients (lipids, proteins, minerals and carbohydrates) would contribute to a considerable reduction in post-harvest losses, guarantee a year-round availability of plantain and provide a sustainable solution to the food security issue in West and Central Africa.

In this paper, the influence of plantain variety and granulometric class on physical (water activity, particle size distribution, shape factors: sphericity, convexity and aspect ratio) and flow properties (basic flowability energy, compressibility, fluidisability, cohesion, flow factor, etc.) was investigated. An improved plantain variety (Big Ebanga from Cameroon), two hybrids (FHIA 21 and PITA 3 from Honduras and Nigeria, respectively) and a local variety (Corne 1 from Ivory Coast) serving as control were used.

Materials and methods

Material

This study was carried out with one improved hybrid variety (Big Ebanga, AAB *Musa sp.*), two new hybrid varieties (FHIA 21 and PITA 3, AAAB *Musa sp.*) and one traditional variety (Corne 1, AAB *Musa sp.*) used as a reference, at stages 4 and 5 of ripening, that is more yellow than green and yellow with green necks, respectively (Lii *et al.*, 1982). Corne 1 was used as a reference variety because it is one of the most widely used traditional varieties in Ivory Coast, which is the most available and has a relatively short production

cycle between 10 and 12 months. Fruits were supplied by the Research Station on Fruits and Citrus Fruits of National Center of Agronomic research (CNRA), located 20 km east of Abidjan (Ivory Coast). They were artificially ripened to stages 4 and 5 using a 20 % (v/v) aqueous solution of ethylene glycol (Sigma-Aldrich, Steinheim, Germany) according to the protocols described by Goonatillake (2008) and Gnagne *et al.* (2017). They were all harvested at maximal maturity, when at least one ripe fruit appeared on the bunch. Starting from the date of flowering, this stage corresponds to 82–90 days for the three hybrids. The control variety Corne 1 was harvested after 80 days.

Plantain powder processing

Powders were produced according to the protocol described by Gnagne *et al.* (2017). First, plantain fingers were precooked for 15 min in distilled water at 100 °C containing 0.5 % (w/v) citric acid (Sigma-Aldrich) to limit enzymatic browning and make peeling easier. After that, pulps were cooked in a buffer solution containing 0.3 % (w/v) sodium pyrophosphate (Sigma-Aldrich) at pH = 4.5, sliced into 10-mm cubes using a stainless-steel knife and dried in a convected air oven (MINERGY, ATIE PROCESS, Pont-de-Larn, France) at 65 °C for 8 h to reach between 8 and 10 % (w/w) moisture content. Dried plantains were milled in a first grinder (I2T, Abidjan, Ivory Coast) equipped with a 500-µm sieve and then in a second grinder (Forplex, Béthune, France) to obtain fine powders that were finally packaged in airtight polyethylene bags stored at 10 ± 1 °C for subsequent analyses.

Following particle size distribution results, the powders were separated in two distinct granulometric classes. Class 1 corresponded to fine particles: it gathered all particles passing through a 180-µm sieve. Class 2 corresponded to retained particles between 180 and 500 µm sieves. Sieving of 50 g batches during 10 min was performed at 1 mm amplitude with the Analyseste 3 apparatus (Fritsch, Idar-Oberstein, Germany), operating by vertical vibration.

Methods

Physical properties

Water activity. The water activity (a_w) of powder samples was determined using the HygroPalm a_w -meter (HP23 AW-A, Rotronic, Croissy-Beaubourg, France) in standard mode using approximately 10 g of powder in a thermostated room (20 ± 1 °C). The equilibration times required to achieve a constant water activity ranged between 5 and 10 min depending on the sample.

Particle size distribution. A Mastersizer 3000 laser granulometer (Malvern Instruments Ltd., Malvern, United

Kingdom) in dry mode (Aero S module) was used at room temperature (20 ± 1 °C) for the determination of particle size distributions of plantain powder samples. About 5 g sample was analysed after air dispersion at 1–1.5 bar in the following conditions: 100 % air pressure, 40 % feed rate and 1 mm hopper length. This allowed reaching between 2 and 20 % obscuration as requested when using dry analysis. Volume-based particle size distributions were characterised by classical granulometric parameters D_{10} , D_{50} and D_{90} , where D_x means that x % of the volume of particle has a diameter lower than D_x . The span, which describes the width of the particle size distribution, was also recorded (equation 1).

$$\text{Span} = \frac{D_{90} - D_{10}}{D_{50}} \quad (1)$$

Shape factors. The shape of powder particles was analysed by image analysis with a QICPIC morphogranulometer (Sympatec Inc., Clausthal-Zellerfeld, Germany) supplied with Windox 5.0 software and a dry dispersion unit. Samples (from 5 to 10 g) were fed into the high-speed dry disperser where sample particles were accelerated up to 100 m s^{-1} via a Venturi tube located in the dispersion line. This allowed dispersing and aerosolising sample particles by particle–particle and particle–wall collisions, along with centrifugal forces caused by velocity gradients. Then, particles entered the measurement zone and were finally collected by a NilfiskTM vacuum system (Nilfisk-Advance A/S, Sognevej, Denmark). The diameter of a circle of equal projection area (EQPC), defined as the diameter of a circle that has the same area as the projection area of the particle, was used as size estimator. Sphericity was determined as the ratio of the perimeter of the circle of equivalent projection area to the real particle perimeter. The sphericity value ranged between 0 (extremely irregular particles such as rod-like particles) and 1 (perfectly spherical particles). Convexity, an important shape parameter describing the compactness of a particle was defined as the ratio of the equivalent projection area to the area of the particle convex hull. Without any concave region, particle convexity is maximal and equals 1. Aspect ratio, another shape factor calculated as the ratio between minimal and maximal Feret diameters (where a Feret diameter is defined as the distance between the two parallel planes restricting the object perpendicular to the direction of particle size measurement), was also determined. It gives an indication of the elongation of a particle.

Flow properties

The study of powder flow properties consisted in performing standard stability, compressibility, aeration

and shear cell tests of the FT4 powder rheometer using 25-mm accessories (Freeman Technology Ltd., UK). Every FT4 measurement was preceded by a step of powder bed conditioning, involving the downward and upward movement of a blade at 100 mm s^{-1} tipspeed through the powder bed, in order to remove excess stress or entrapped air.

During both downward and upward rotational movement of the blade, the torque and axial force required to move the blade through the powder bed were recorded and used by the software to calculate the total energy input required to make the powder bed flow. For the stability test, seven cycles of conditioning and test were realised by the rotating movement of the blade into and through a powder bed, and the stabilised flow energy of test 7 was taken as the basic flowability energy (BFE). The specific energy, SE (mJ g^{-1}), was evaluated as the energy needed to displace the conditioned powder bed during upward moving of the blade divided by the mass of sampled powder. The stability test also allows measuring the conditioned bulk density (CBD), a parameter similar to the apparent density of the powder bed, by weighting the mass of a given powder volume after conditioning.

The aeration test was carried out by measuring the flow energy in aerated conditions at 100 mm s^{-1} blade tipspeed: air was circulated upward through the powder bed at velocities ranging from 0 to 10 mm s^{-1} by 2 mm s^{-1} steps. Powder sample was placed in the measurement cell fitted with a porous plate. Prior to the aeration test, three conditioning cycles were carried out to achieve a homogeneous initial state of the powder bed. A complete test was then performed by measuring the flow energy of the powder bed, and a conditioning cycle was carried out between each aeration test cycle to achieve a steady state of aerated powder. Various fluidisation parameters were determined from the aeration test:

- the aeration energy, AE (mJ), which is the flow energy at 10 mm s^{-1} air velocity;
- the aeration ratio, AR (dimensionless), which is defined as the ratio between the BFE (measured at 0 mm s^{-1} air velocity) and the AE. AR and AE are considered as good indicators of powder fluidisability: easily fluidisable powders may have high AR and low AE.
- the normalised aeration sensitivity, NAS (s mm^{-1}), which is a measure of powder sensitivity to aeration, especially at small air velocities. It is defined as the maximal difference in normalised test energy between the two consecutive points of the test, divided by the change in air velocity.

Fluidisation occurred for all powders, so the minimum fluidisation velocity (MFV) was also recorded. It is the minimum air velocity needed to establish

fluidisation in the powder bed. It was considered that fluidisation occurred when the flow energy was close to zero, that is inferior to 10 mJ in the conditions of the aeration test.

The compressibility test was carried out by applying increasing normal stresses with a vented piston to a conditioned powder bed and measuring the volume change. Powder sample was first placed in the measurement cell and submitted to three conditioning cycles. After that, the vessel was split to remove any excess powder and the dynamic blade was changed with the 23.5-mm vented piston. Then, powder was slowly compressed by the vented piston vertically moving at 0.05 mm s^{-1} and applying levels of normal stresses from 0.5 to 15 kPa while measuring the volume change. Compressibility, CPS (%), was determined as the percentage change in volume after compression at an applied normal stress of 15 kPa. Then, the compacted density, CD (g mL^{-1}), was calculated according to the following equation (equation 2):

$$\text{CD}(\text{g/mL}) = \frac{\text{CBD}}{1 - \frac{\text{CPS}}{100}} \quad (2)$$

Shear tests were carried out using the rotational shear cell accessory of the FT4 powder rheometer. The powder sample was first conditioned to achieve a homogeneous initial state and slowly precompacted under a normal stress of 9 kPa with a vented piston. Then, the vented piston was changed for the shear cell head and the powder sample was recompressed to remove any disturbances caused by the split and ensure that the sample surface was properly consolidated. After this preconsolidation step, the sample was presheared at 9 kPa to achieve a critically consolidated state. Measurements consisted in recording the shear stress required to cause the failure of the precompacted powder bed at applied normal stresses decreasing from 7 to 3 kPa by 1 kPa steps. Powder cohesion, C_m (kPa), was evaluated as the equivalent shear stress at zero normal stress (i.e. intercept of the linear regression of the shear stress vs. applied normal stress curve), and the flowability index (also called flow factor), ff (dimensionless), was calculated as the ratio between the major principal stress and the unconfined yield stress, using the yield locus approach. The classification of powders by Jenike (1964) according to the ff value was then applied: $ff < 1$: not flowing; $1 < ff < 2$: very cohesive; $2 < ff < 4$: cohesive; $4 < ff < 10$: easy-flowing; and $10 < ff$: free-flowing.

Statistical analysis

All analyses of this study were carried out in triplicates, and reported values were means $\pm$ standard deviations. One-way ANOVA was conducted to evaluate

the presence of statistically significant differences between samples using Sigma Stat 7.1 (Systat Software, Inc., Chicago, IL, USA). Tukey's HSD test at $P \leq 0.05$ was used for the comparison of sample means.

Results and discussion

Physical parameters

Water activity

Determination of water activity is one of the most frequent analyses carried out to evaluate the quality of industrial food products, in particular regarding their preservation ability (Mathlouthi, 2001). pH and water activity are often listed as the main factors affecting storage stability and shelf life of food materials, including food systems rich in carbohydrates such as plantain-based powders (Labuza *et al.*, 1972). For foods with low and medium water content, water activity may be useful in controlling various functional properties (flowability, reconstitutability, etc.) and the rate of food alteration reactions (Labuza *et al.*, 1972; Troller & Christian, 1978). The water activity of studied powders, ranging between 0.283 and 0.539 (Table 1), significantly differed according to particle size and plantain variety. Hence, all investigated powders can be considered as biochemically and microbiologically stable, as their water activity remained below 0.6 which represents a threshold for the growth of most microorganisms (moulds, yeasts and bacteria) (Tapia *et al.*, 2007; Deli *et al.*, 2019b). In general, class 1 powders had the lowest water activity; thus, they may have the best preservation ability amongst produced plantain powders. Also, this low-to-moderate water activity may be favourable to good flow properties; indeed, according to Emery *et al.* (2009), a low water content of powders might have a positive effect on their flowability, as it can both eliminate particle electrostatic charges and decrease surface roughness. Moreover, at higher water activity, powders are expected to become more cohesive and agglomerate (Bravo-Osuna *et al.*, 2007). Most studies report a decrease in powder flow at increased moisture content (Bravo-Osuna *et al.*, 2007; Sandler *et al.*, 2010).

For unsieved powders, the reference variety Corne 1 had the highest water activity. For class 2 powders, the highest water activity values were found for FHIA 21 and the reference variety Corne 1. Also, the reference variety presented the lowest water activity for class 1 powders.

Particle size distribution

Particle size distributions and weight proportions of unsieved powders and their granulometric classes are displayed in Fig. 1 and Table 1. Particle size

Table 1 Granulometric parameters, water activity and sieved mass fractions of unsieved powders and size classes prepared from three plantain hybrids (Big Ebanga, FHIA 21 and PITA 3) and the reference variety Corne 1

Powder type	Plantain variety	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)	Span (–)	Water activity (–)	Sieved mass fractions (%)
Unsieved powder (< 500 μm)	Big Ebanga	22.17 ^b ± 0.32	90.67 ^d ± 1.89	281.00 ^e ± 4.36	2.86 ⁱ ± 0.02	0.454 ^f ± 0.002	–
	FHIA 21	40.30 ^d ± 0.53	173.67 ^f ± 1.15	383.00 ^f ± 2.65	1.97 ^{ef} ± 0.02	0.447 ^{ef} ± 0.014	–
	PITA 3	20.73 ^{ab} ± 0.25	93.27 ^d ± 0.12	260.00 ^d ± 2.00	2.57 ^h ± 0.02	0.446 ^{ef} ± 0.003	–
	Corne 1	22.37 ^b ± 0.25	75.60 ^c ± 1.15	205.00 ^c ± 4.00	2.40 ^g ± 0.04	0.539 ^h ± 0.001	–
Class 1 (< 180 μm)	Big Ebanga	29.37 ^c ± 0.31	68.73 ^b ± 0.46	133.33 ^a ± 1.53	1.51 ^d ± 0.02	0.433 ^{de} ± 0.006	81.06
	FHIA 21	36.17 ^d ± 0.95	132.67 ^e ± 1.15	246.33 ^d ± 5.13	1.59 ^d ± 0.04	0.392 ^c ± 0.006	74.34
	PITA 3	22.50 ^b ± 0.60	79.17 ^c ± 2.12	172.33 ^b ± 5.86	1.89 ^e ± 0.04	0.361 ^b ± 0.003	92.61
	Corne 1	15.03 ^a ± 0.06	53.93 ^a ± 0.15	124.67 ^a ± 0.58	2.04 ^f ± 0.01	0.283 ^a ± 0.001	72.96
Class 2 (180 – 500 μm)	Big Ebanga	183.67 ^e ± 2.25	263.00 ^g ± 1.00	376.00 ^f ± 1.00	0.73 ^b ± 0.02	0.428 ^d ± 0.001	17.01
	FHIA 21	210.33 ^g ± 3.21	306.67 ^j ± 3.79	447.00 ^g ± 15.52	0.75 ^b ± 0.06	0.500 ^g ± 0.002	24.99
	PITA 3	211.67 ^f ± 0.58	284.33 ^h ± 1.53	384.33 ^f ± 2.52	0.61 ^a ± 0.01	0.439 ^{de} ± 0.001	6.45
	Corne 1	195.00 ^f ± 5.57	296.33 ⁱ ± 5.51	450.00 ^g ± 3.61	0.86 ^c ± 0.02	0.499 ^g ± 0.001	25.87

–, not applicable

Means with different superscripted letters in the same column were significantly different according to Tukey's HSD test ($p < 0.05$; $n = 3$).

distributions of plantain powders were monomodal. The mean particle sizes D₅₀ of class 1 and 2 granulometric fractions ranged from 75.60 to 173.67 μm and were comprised in the size ranges of employed sieves. D₁₀ and D₉₀ were also close to the mesh sizes of lower

and upper sieves, respectively, when applicable. Both results showed that the sieving procedure was successful in obtaining well different granulometric classes (Malela *et al.*, 2016). Table 1 also shows the weight proportions of granulometric classes, measured after

Figure 1 Particle size distributions of granulometric classes of powders of three plantain hybrids (Big Ebanga, FHIA 21 and PITA 3) and the reference material Corne 1.

sieving. A majority of particles of unsieved powders was able to pass through a 180- μm sieve, as sieved mass fraction of class 1 was around 80 % whatever the plantain variety. PITA 3 hybrid led to the highest yield (92.61 %) for class 1 powders and therefore the lowest yield (6.45 %) for class 2 powders.

Shape factors

Figure 2 shows the convexity, sphericity, and aspect ratio of unsieved powders and their granulometric fractions. Depending on plantain variety, convexity ranged between 0.88 and 0.94, aspect ratio between

0.66 and 0.71, whereas no significant difference was observed for sphericity (found between 0.84 and 0.86). In the literature, many studies have shown that plantain powder is usually composed of small particles of circular or angular shape (Rayo *et al.*, 2015; Gnagne *et al.*, 2017). These results were close to those obtained by Gnagne *et al.* (2017) for powders of three plantain cultivars.

Convexity of unsieved and class 2 powders was slightly higher than for class 1 powders. Whatever the plantain variety, class 2 powders had the highest values of convexity (0.93–0.94), intermediate values were found for unsieved powders (0.90–0.91), and class 1 powders had the lowest convexity (0.88–0.90). Regarding the aspect ratio, it ranged between 0.68 and 0.71 for unsieved powders, from 0.66 to 0.71 for class 1 powders and from 0.71 to 0.72 for class 2 powders. Thus, class 1 powders were slightly more elongated than class 2 powders. The degree of correlation between mean particle size and shape factors was investigated by calculating Pearson's correlation coefficients. A high positive correlation was found between D_{50} and convexity ($r = 0.97$, $P < 0.05$). This shows that larger particles were more convex, which can be understood as more compact and less elongated.

Flow properties of plantain powders

Flow properties of unsieved powders and their granulometric classes are presented in Fig. 3 and Tables 2 and 3. The range of basic flowability energies BFE, between 112.75 and 250.93 mJ, was typical for powders of medium-high flowability (Jan *et al.*, 2018). Significant differences were observed between powders. In fact, whatever the granulometric class, the FHIA 21 variety had the highest BFE value. BFE seemed to slightly increase with particle size, as previously reported in the literature (Lu *et al.*, 2015). Thus, from this criterion, class 1 powder beds appeared to have a better dynamic flowability. As regards the specific energy SE, it ranged from 2.25 to 4.83 mJ g⁻¹ for all investigated powders, consistently with results reported by Gnagne *et al.* (2017) for plantain-based powders. This indicated that all studied powders were not much cohesive. SE values of unsieved powders were comprised between 3.22 (FHIA 21) and 4.17 mJ g⁻¹ (Big Ebanga). For class 1 powders, the FHIA 21 hybrid showed the lowest SE value (3.51 mJ g⁻¹), while the control variety Corne 1 showed the highest SE value (4.83 mJ g⁻¹). SE decreased with particle size, in agreement with the literature (Jan *et al.*, 2018), suggesting that powder cohesion was lowered for large particles (Fu *et al.*, 2012; Ghoroi *et al.*, 2013; Gnagne *et al.*, 2017). This may be understood by the fact that interparticular contact points are generally less numerous for large

Figure 2 Convexity (a), sphericity (b) and aspect ratio (c) of unsieved plantain powders and their granulometric fractions. Bars topped with different letters corresponded to significantly different results according to Tukey's HSD test ($P < 0.05$).

Figure 3 Compressibility (i.e. percentage of bulk density change) versus normal stress of unsieved plantain powders and their granulometric fractions.

Table 2 Results of stability and aeration tests of unsieved powders and granulometric classes prepared from three plantain hybrids (Big Ebanga, FHIA 21 and PITA 3) and the reference variety Corne 1

Powder type	Plantain variety	Basic flowability energy, BFE (mJ)	Specific energy, SE (mJ g ⁻¹)	Conditioned bulk density, CBD (g mL ⁻¹)	Aerated energy, AE (mJ)	Aeration ratio, AR (-)	Normalised aeration sensitivity, NAS (s mm ⁻¹)	Minimum fluidisation velocity, MFV (mm s ⁻¹)
Unsieved powder	Big Ebanga	146.61 ^b ± 6.94	4.17 ^f ± 0.06	0.75 ^{ab} ± 0.01	1.45 ^a ± 0.37	137.26 ^b ± 36.47	1.01 ^{bc} ± 0.03	1.60 ^a ± 0.40
	FHIA 21	180.33 ^{cd} ± 3.04	3.22 ^c ± 0.07	0.85 ^h ± 0.01	1.53 ^a ± 0.15	246.17 ^c ± 36.47	0.92 ^b ± 0.12	2.33 ^a ± 0.58
	PITA 3	154.74 ^{bc} ± 5.48	3.80 ^{ef} ± 0.02	0.80 ^g ± 0.01	4.76 ^a ± 0.67	47.37 ^a ± 8.99	1.30 ^d ± 0.04	1.20 ^a ± 0.01
	Corne 1	174.95 ^{cd} ± 4.15	3.42 ^{cde} ± 0.09	0.79 ^{fg} ± 0.01	6.91 ^a ± 1.10	57.28 ^{ab} ± 2.68	0.47 ^a ± 0.01	1.47 ^a ± 0.46
Class 1 (< 180 µm)	Big Ebanga	174.95 ^{bc} ± 4.15	3.77 ^{def} ± 0.08	0.77 ^{de} ± 0.05	2.08 ^a ± 0.77	122.19 ^b ± 28.71	1.19 ^{bcd} ± 0.01	1.33 ^a ± 0.23
	FHIA 2	178.45 ^{cd} ± 5.12	3.51 ^{cde} ± 0.05	0.78 ^{def} ± 0.01	0.85 ^a ± 0.13	352.71 ^d ± 24.23	0.92 ^b ± 0.06	1.60 ^a ± 0.01
	PITA 3	141.95 ^b ± 4.80	3.55 ^{cde} ± 0.09	0.77 ^{cde} ± 0.01	1.33 ^a ± 0.57	257.33 ^c ± 67.51	1.25 ^{cd} ± 0.01	1.20 ^a ± 0.01
	Corne 1	112.75 ^a ± 4.18	4.83 ^g ± 0.07	0.74 ^a ± 0.01	10.80 ^a ± 0.84	12.43 ^a ± 1.95	1.18 ^{bcd} ± 0.22	1.07 ^a ± 0.46
Class 2 (180–500 µm)	Big Ebanga	191.50 ^d ± 11.91	3.40 ^{cd} ± 0.27	0.75 ^{abc} ± 0.01	68.87 ^b ± 5.76	3.20 ^a ± 0.54	0.42 ^a ± 0.05	–
	FHIA 2	250.93 ^e ± 19.29	3.56 ^{cde} ± 0.26	0.80 ^{fg} ± 0.01	124.09 ^c ± 6.05	4.66 ^a ± 0.25	0.53 ^a ± 0.12	–
	PITA 3	143.77 ^b ± 6.25	2.25 ^a ± 0.03	0.79 ^{efg} ± 0.01	112.95 ^c ± 5.22	5.64 ^a ± 0.50	0.58 ^a ± 0.06	–
	Corne 1	167.42 ^{bcd} ± 14.95	2.77 ^b ± 0.20	0.76 ^{bcd} ± 0.01	120.63 ^c ± 21.91	2.85 ^a ± 0.38	0.37 ^a ± 0.12	–

–, not applicable, that is minimal fluidisation velocity was not reached in the conditions of the standard FT4 aeration test (up to 10 mm s⁻¹). Means with different superscripted letters in the same column were significantly different according to Tukey's HSD test ($P < 0.05$).

particles. Conditioned bulk density (CBD), ranging from 0.74 to 0.85 g mL⁻¹, did not vary much between investigated samples. Plantain variety had no significant influence on the CBD.

Unsieved and class 1 powders showed a fluidised state at the end of the standard FT4 aeration test, and their minimum fluidisation velocity varied between 1.07 and 2.33 mm s⁻¹.

Sensitivity to small aeration rates (NAS) was comprised between 0.37 and 1.30 s mm⁻¹, with significant differences amongst samples. The inability to be

fluidised at air velocities up to 10 mm s⁻¹ and the low NAS values of class 2 powders may be justified by their greater proportion of large particles, which are more difficult to suspend in the air than non-cohesive small particles. In fact, class 2 powders had average sensitivity to aeration (aeration ratio (AR) comprised between 2 and 6), whereas the other powders (unsieved and class 1) were very sensitive to aeration (AR > 10), which is a sign of lower cohesion of the latter powders. Indeed, as a general rule, the larger the aeration ratio and the lower the aerated energy, the less

Table 3 Results of compressibility and shear cell tests of unsieved powders and granulometric classes prepared from three plantain hybrids (Big Ebanga, FHIA 21 and PITA 3) and the reference variety Corne 1

Powder type	Plantain variety	Compressibility at 15 kPa, CPS (%)	Compacted density at 15 kPa, CD (g mL ⁻¹)	Cohesion, C _m (kPa)	Flowability index, ff (–)
Unsieved powder (< 500 µm)	Big Ebanga	11.97 ^f ± 0.93	0.85 ^{def} ± 0.02	0.35 ^{cde} ± 0.01	12.41 ^{abc} ± 0.43
	FHIA 2	5.55 ^c ± 0.13	0.90 ^g ± 0.01	0.19 ^{ab} ± 0.01	22.81 ^{bcd} ± 1.27
	PITA 3	7.22 ^d ± 0.37	0.86 ^{ef} ± 0.00	0.33 ^{bcd} ± 0.03	13.25 ^{abcd} ± 0.92
	Corne 1	7.48 ^d ± 0.40	0.86 ^{ef} ± 0.01	0.28 ^{abcd} ± 0.02	15.26 ^{abcd} ± 1.30
Class 1 (< 180 µm)	Big Ebanga	8.88 ^e ± 0.13	0.85 ^{def} ± 0.00	0.17 ^a ± 0.01	24.38 ^{def} ± 0.95
	FHIA 2	4.54 ^{bc} ± 0.19	0.82 ^c ± 0.01	0.18 ^{ab} ± 0.03	23.30 ^{cdef} ± 3.12
	PITA 3	5.79 ^c ± 0.21	0.82 ^c ± 0.01	0.21 ^{abc} ± 0.06	21.90 ^{bcd} ± 6.74
	Corne 1	11.40 ^f ± 1.02	0.83 ^{cde} ± 0.00	0.37 ^{de} ± 0.02	11.62 ^{ab} ± 0.46
Class 2 (180–500 µm)	Big Ebanga	3.03 ^a ± 0.28	0.77 ^a ± 0.01	0.47 ^e ± 0.09	9.99 ^a ± 1.76
	FHIA 2	3.56 ^{ab} ± 0.02	0.83 ^{cde} ± 0.00	0.46 ^e ± 0.07	11.76 ^{abc} ± 4.50
	PITA 3	2.45 ^a ± 0.09	0.81 ^{bc} ± 0.00	0.15 ^a ± 0.04	29.00 ^{ef} ± 8.23
	Corne 1	2.77 ^a ± 0.42	0.78 ^{ab} ± 0.01	0.13 ^a ± 0.02	34.64 ^f ± 6.88

Means with different superscripted letters in the same column were significantly different according to Tukey's HSD test ($P < 0.05$).

cohesive the powder (Freeman, 2013a). It can be seen that AE was increased at higher mean particle size. Aerated energy AE varied from 0.85 to 120.63 mJ and was not significantly different according to plantain variety within each granulometric class.

When increasing applied normal stress from 1 to 15 kPa, compressibility CPS increased from 1% to 3–4% for class 2 powders, 5–12% for class 1 powders and 6–12% for unsieved powders. For unsieved powders, the highest CPS (11.97 %) was obtained for the FHIA 21 hybrid and the lowest (5.55 %) for the Big Ebanga hybrid. For class 1 powders, the Corne 1 control variety had the highest CPS value (11.40 %), whereas the FHIA 21 hybrid had the lowest CPS value (4.54 %). No significant plantain variety effect was observed on CPS of class 2 powders. CPS allows to evaluate the density change of a powder under application of normal stress. It is not directly a measurement of flowability, but nevertheless relates to flow behaviour occurring in many process environments, such as storage in hoppers and big bags or processing behaviour during roller compaction (Fu *et al.*, 2012; Freeman, 2013b). Generally, highly compressible powders require more energy to flow (Salameh *et al.*, 2016). The compressibility of unsieved and class 1 powders was greater than that of class 2 powders. This may be due to the lower mean particle sizes of unsieved and class 1 powders, as low particle size powders have a tendency to be more cohesive and entrap large amounts of air, thus making them more sensitive to compression (Salameh *et al.*, 2016). Also, powders constituted by several particle populations and/or presenting a large dispersion of particle sizes around the mean value (i.e. having a high span) may likely be more compressible (Salameh *et al.*, 2016; Gnagne *et al.*, 2017).

The compacted density (CD) ranged from 0.77 to 0.90 g mL⁻¹, with unsieved powders showing the highest values. Such high values of CBD and CD are expected to be favourable to powder flowability (Jan *et al.*, 2018).

Last, the shear cell test permitted to confirm the very low cohesion of studied powders, which ranged from 0.13 to 0.47 kPa, similarly to Gnagne *et al.* (2017). All investigated samples were free-flowing powders, with *ff* values comprised between 9.99 and 34.44. No clear effect of plantain variety or mean particle size on cohesion and flowability index was observed. Also, it was noticed that shear and compressibility test results were in good agreement: the higher the compressibility of the powder bed, the higher the cohesion deduced from shear cell test, and the lower the powder flowability.

Both particle shape and particle size distribution significantly influenced the shear properties of the samples. *D*₅₀ and convexity appeared to be negatively correlated with SE ($r = -0.70$), NAS ($r = -0.94$ and -0.92 , respectively) and CPS at 15 kPa ($r = -0.85$ and -0.76 , respectively). Also, CPS at 15 kPa was found to be positively correlated with SE ($r = 0.84$) and NAS ($r = 0.80$). This shows that large particles of plantain powders had better flow properties and were less sensitive to aeration and compaction.

Conclusion

In the present work, the influence of particle size and plantain variety on physical and flow properties of powders prepared from semi-ripe plantain was investigated. As regards physical properties, water activity was lower for class 1 (< 180 µm) granulometric class than for unsieved and class 2 (between 180–500 µm) powders. Convexity tended to increase for larger particles, and a

positive correlation was even found between D_{50} and convexity. All studied powders were not much cohesive and could be considered as free-flowing. Fluidisation occurred only for class 1 and unsieved powders, suggesting that the interparticular cohesive forces were relatively low for these powders and that class 2 powders were too large to be fluidised in the conditions of the standard FT4 aeration test. Compressibility decreased with particle size for all plantain varieties. Shape factors and particle size distribution significantly influenced the flow properties of plantain powders.

This study permitted to show that differences in particle size distribution influence physical properties of plantain powders such as a_w , D_{50} and convexity and significantly affected all flow properties. So, it would be interesting to determine the influence of the particle size on the rheological properties (that can also influence flow properties) in order to give a clear orientation of the applications of these powders either in pastry, in infantile porridge, or for the reconstitution of dishes such as foutou or fofou.

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Author contribution

Kady Rosine IRIE: Formal analysis (supporting); Writing-original draft (supporting); Writing-review & editing (supporting). **Jeremy Petit:** Data curation (supporting); Visualization (supporting); Writing-review & editing (supporting). **Eliane H. GNAGNE:** Methodology (supporting); Visualization (equal). **Olivier Kouadio KOUADIO:** Project administration (lead); Supervision (supporting); Visualization (supporting). **Claire GAIANI:** Supervision (supporting); Visualization (supporting). **Joël Scher:** Project administration (supporting); Resources (supporting); Supervision (supporting); Validation (supporting). **Georges N'Guessan AMANI:** Project administration (supporting); Resources (supporting); Supervision (supporting); Validation (supporting).

Conflict of interest

The authors declare that they have no conflict of interest.

Ethical approval

'Ethics approval was not required for this research'.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

References

- Alkarkhi, A.F.M., Ramli, S.B., Yong, Y.S. & Easa, A.M. (2011). Comparing physicochemical properties of banana pulp and peel flours prepared from green and ripe fruits. *Food Chemistry*, **129**, 312–318.
- Bravo-Osuna, I., Ferrero, C. & Jiménez-Castellanos, M.R. (2007). Influence of moisture content on the mechanical properties of methyl methacrylate-starch copolymers. *European Journal of Pharmaceutics and Biopharmaceutics*, **66**, 63–72.
- Becker, L., Zaiter, A., Petit, J. et al. (2016). Improvement of antioxidant activity and polyphenol content of *Hypericum perforatum* and *Achillea millefolium* powders using successive grinding and sieving. *Industrial Crops and Products*, **87**, 116–123.
- Becker, L., Zaiter, A., Petit, J. et al. (2017). How do grinding and sieving impact on physicochemical properties, polyphenol content, and antioxidant activity of *Hieracium pilosella* L. powders? *Journal of Functional Foods*, **35**, 666–672.
- Deli, M., Petit, J., Nguimbou, R.M., Beaudelaire Djantou, E., Njintang Yanou, N. & Scher, J. (2019a). Effect of sieved fractionation on the physical, flow and hydration properties of *Boscia senegalensis* Lam., *Dichrostachys glomerata* Forssk. and *Hibiscus sabdariffa* L. powders. *Food Science and Biotechnology*, **7**, 3618–3628.
- Deli, M., Ndjantou, E.B., Ngatchic Metsagang, J.T., Petit, J., Njintang Yanou, N. & Scher, J. (2019b). Successive grinding and sieving as a new tool to fractionate polyphenols and antioxidants of plants powders: Application to *Boscia senegalensis* seeds, *Dichrostachys glomerata* fruits, and *Hibiscus sabdariffa* calyx powders. *Food Science & Nutrition*, **7**, 1795–1806.
- Eke-Ejiofor, J. & Kiin-Kabari, D.B. (2012). Effect of substitution on the functional properties of flour, proximate and sensory properties of wheat/plantain composite bread. *International Journal of Agricultural Sciences*, **10**, 281–284.
- Emery, E., Oliver, J., Pugsley, T., Sharma, J. & Zhou, J. (2009). Flowability of moist pharmaceutical powders. *Powder Technology*, **189**, 409–415.
- Fitzpatrick, J.J., Iqbal, T., Delaney, C., Twomey, T. & Keogh, M.K. (2004). Effect of powder properties and storage conditions on the flowability of milk powders with different fat contents. *Journal of Food Engineering*, **64**, 435–444.
- Fongang Fouepe, G.H., Folefack, D.P., Pane Pagui, Z., Bikoi, A. & Noupadja, P. (2016). Transformation et commercialisation des chips de banane plantain au Cameroun : une activité artisanale à forte valeur ajoutée. *International Journal of Biological and Chemical Sciences*, **10**, 1184.
- Freeman Technology (2013a). Stability method, Approved by: Quality Manage, Template D101 (pp. 8.), Issue C, W7011.
- Freeman Technology (2013b). Compressibility, Approved by: Quality Manage, Template D101 (pp. 5), Issue B, W7008.
- Fu, X., Huck, D., Makein, L., Armstrong, B., Willen, U. & Freeman, T. (2012). Effect of particle shape and size on flow properties of lactose powders. *Particuology*, **10**, 203–208.
- Ghoroi, C., Han, X., To, D., Jallo, L., Gurumurthy, L. & Davé, R.N. (2013). Dispersion of fine and ultrafine powders through

- surface modification and rapid expansion. *Chemical Engineering Science*, **85**, 11–24.
- Gibert, O., Dufour, D., Giraldo, A. *et al.* (2009). Differentiation between cooking bananas and dessert bananas. 1. Morphological and compositional characterization of cultivated Colombian *Musa*-*ceae* (*Musa* sp.) in relation to consumer preferences. *Journal of Agricultural and Food Chemistry*, **57**, 7857–7869.
- Gnagne, E.H., Petit, J., Gaiani, C., Scher, J. & Amani, G.N. (2017). Characterisation of flow properties of foutou and fofou flours, stable foods in West Africa, using FT4 powder Rheometer. *Journal of Food Measurement and Characterization*, **11**, 1128–1136.
- Goonatilake, R. (2008). Effects of diluted ethylene glycol as a fruit-ripening agent. *Global Journal of Biotechnology and Biochemistry*, **3**, 8–13.
- Happi Emaga, T., Andrianaivo, R.H., Wathélet, B., Tchango, J.T. & Paquot, M. (2007). Effects of the stage of maturation and varieties on the chemical composition of banana and plantain peels. *Food Chemistry*, **103**, 590–600.
- Jan, S., Ghoroi, C. & Saxena, D.C. (2018). Characterization of bulk and shear properties of basmati and non-basmati rice flour. *Journal of the Science of Food and Agriculture*, **98**, 667–673.
- Jenike, A.W. (1964). Storage and flow of solids, Engineering experiment station, University of Utah, Bulletin 123.
- Kerr, W.L., Ward, C.D.W., McWatters, K.H. & Resurreccion, A.V.A. (2000). Effect of milling and particle size on functionality and physicochemical properties of cowpea flour. *Cereal Chemistry Journal*, **77**, 213–219.
- Labuza, T.P., Mc Nally, L., Gallagher, D., Hawkes, J. & Hurtado, F. (1972). Stability of intermediate moisture foods. 1. Lipid Oxidation. *Journal of Food Science*, **37**, 154–159.
- Lii, C.Y., Chang, S.M. & Young, Y.L. (1982). Investigation of the physical and chemical properties of banana starches. *Journal of Food Science*, **47**, 1493–1497.
- Liu, Y., Guo, X., Lu, H. & Gong, X. (2015). An investigation of the effect of particle size on the flow behavior of pulverized coal. *Procedia Engineering*, **102**, 698–713.
- Lu, H., Guo, X., Liu, Y. & Gong, X. (2015). Effect of particle size on flow mode and flow characteristics of pulverized coal. *KONA Powder and Particle Journal*, **32**, 143–153.
- Malela, K.E., Petit, J., Nzikou, J.M. & Scher, J. (2016). Physico-chemical characterisation and phytochemical study of *Grewia coriacea* Mast powders: focus on polyphenols. *IJISSET - International Journal of Innovation Science, Engineering & Technology*, **3**, 14.
- Mathlouthi, M. (2001). Water content, water activity, water structure and the stability of foodstuffs. *Food Control*, **12**, 409–417.
- Ngalani, J.A. & Crouzet, J. (1995). Utilisation de farine de banana plantain dans la fabrication de pains, biscuits et farines instantanées. *Cahiers Agricultures*, **4**, 61–64.
- Nkendah, R. & Akyeampong, E. (2003). Socio-economical data of banana production in Central and Western Africa. *Infomusa*, **12**, 8–13.
- Nura, M., Kharidah, M., Jamilah, B. & Roselina, K. (2011). Textural properties of laksa noodle as affected by rice flour particle size. *Food Research International*, **18**, 1309–1312.
- Rayo, L.M., Chaguri e Carvalho, L., Sardá, F.A.H., Dacanal, G.C., Menezes, E.W. & Tadini, C.C. (2015). Production of instant green banana flour (*Musa cavendishii*, var. Nanicaão) by a pulsed-fluidized bed agglomeration. *LWT - Food Science and Technology*, **63**, 461–469.
- Tapia, M.S., Alzamora, S.M. & Chirife, J. (2007). Effects of water activity (aw) on microbial stability: as a hurdle in food preservation. In book: *Water Activity in Foods: Fundamentals and Applications*.
- Troller, J.A. & Christian, J.H.B. (1978). *Water activity and food*. Pp. 13–47 New York: Academic Press.
- Salameh, C., Banon, S., Hosri, C. & Scher, J. (2016). An overview of recent studies on the main traditional fermented milks and white cheeses in the Mediterranean region. *Food Reviews International*, **32**, 256–279.
- Sandler, N., Reiche, K., Heinämäki, J. & Yliruusi, J. (2010). Effect of moisture on powder flow properties of theophylline. *Pharmaceutics*, **2**, 275–290.
- Zaiter, A., Becker, L., Petit, J. *et al.* (2016). Antioxidant and anti-acetylcholinesterase activities of different granulometric classes of *Salix alba* (L.) bark powders. *Powder Technology*, **301**, 649–656.

Graphical Abstract

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Summary of effect on particle size in the physical parameters and flow properties of semi-ripe plantain hybrids powders.