Acta Agrophysica, 2006, 7(2), 477-485

ANALYSIS OF THE ACCURACY OF SINGLE-KERNEL DENSITY MEASUREMENTS MADE WITH A SIMPLE EXPERIMENTAL SET-UP*

Ryszard Siuda, Mikołaj Rozwadowski

Institute of Mathematics and Physics, Technical and Agricultural University
ul. S.Kaliskiego 7, 85-796 Bydgoszcz, Poland
e-mail: siuda@atr.bydgoszcz.pl

A b s t r a c t. An experimental set-up consisting of a commercial electronic microbalance and a home-made air micropycnometer was used for the determination of single-kernel (SK) density of wheat grain via SK mass and volume measurements. The measurements were made for two representative samples taken from two lots of wheat grain differing in mean mass (45.3 and 30.4 mg). It was found that results obtained for SKs of lower mass were considerably affected by measurement inaccuracy (up to ca. 15% of the density for the lightest kernels). This effect can be removed by an appropriate procedure. Corrected results showed that mean density for kernels from the sample of massive kernels was lower (1.282 g cm^{-3}) than for the sample of light kernels (1.363 g cm^{-3}). Kernels from both samples showed linear increase of SK density with mass (the slope of the dependence was ca. 0.0017 g cm^{-3} mg^{-1}). Corrected standard deviation from the linear dependence was larger for lighter kernels (0.066 and 0.041 g cm^{-3}, respectively) and was noticeably reduced compared to the value before correction for measurement inaccuracy (0.100 and 0.047 g cm^{-3}, respectively).

K e y w o r d s: single-kernel density, single-kernel mass, single-kernel volume, single-kernel physical properties

INTRODUCTION

Single kernel (SK) density is a physical property correlated with grain quality in many respects. For instance, Tkachuk *et al.* [4] showed that sprouted, shrunken, and broken kernels were highly concentrated in the least dense fraction of grain. Another report by Tkachuk *et al.* [5] showed distinct negative correlation between kernel density and the intensity of visible symptoms of fusarium head blight (also

* The work has been partly supported from research projects BS-16/2005.

known as scab) present in wheat. These findings allowed the authors to use specific gravity table for improving grain quality by removal of lower density kernels. Density of grain kernels affects also some other parameters, such as resistance to fungal infestation, kernel hardness, flour quality, etc. For these reasons, SK density measurements are still important in evaluation of grain quality.

Kernels contain some voids and pores. As a consequence, measured SK density may depend on which method for SK volume determination is used. Liquid pycnometers tend to underestimate SK volume because penetration of pores with the liquid is limited due to surface tension. Fang and Campbell [2] evaluated this effect at the level of ca. 6 to 10%, in dependence on the liquid used. Gas pycnometers provide better opportunity for reliable SK volume evaluation, provided no closed voids are present. This assumption, however, is often not valid. Therefore, gas pycnometers tend to overestimate SK volume, and, as a consequence, underestimate SK density. If one is interested in determination of the actual value of SK bulk material density, the best method seems to be that used by Chang [1]. The author used crushed kernels and helium-gas pycnometer to determine the true volume of particles in powdered material of grain. Resulting density was referred to as true density, while the apparent density was defined as the ratio of SK mass to its volume determined with the same pycnometer for whole kernels. According to this distinguishing, we will deal hereafter with the apparent SK density which will be simply called density or SK density.

Present contribution is focused on evaluation of the accuracy of SK density derived from SK mass and volume measurements made with a set of two instruments: a commercial electronic balance and a home made air-micro-pycnometer. As the analysis of the measurements shows that measurement accuracy influences the values of light SK density, the method for subtraction of this effect from results of the measurements is described as well.

EXPERIMENTAL DETAILS, RAW RESULTS

Measurements

The SK mass measurements were made with a commercial electronic balance (WPS/50/C2, Radwag, Poland) allowing readout at position of 0.1 mg. This position for a given SK, however, was subject to fluctuations by more than neighbouring figures only. Therefore, for four single kernels differing visibly in size, 15 measurements were made in order to evaluate the actual uncertainty of the measurements. In a similar way, evaluation of the uncertainty for measurements of SK volume were made. In both cases uncertainty was assumed to be equal to the standard deviation of the results of measurements. The same definition for uncertainty of any other quantity holds hereafter.

SK volume was measured with a simple home made air micropycnometer based on Boyle-Mariotte law. The principle of the measurement of the instrument is based on the idea described in [3], and similar to that described in [1]. The volume of SK is calculated from the formula:

$$v = v_o (1 - \frac{p_2}{p_1 - p_2}) , \tag{1}$$

where v_o is a volume constant for a given instrument (457.3 μl for the present instrument), p_1 and p_2 are two pressures measured at the cycle of manipulations needed for the SK volume measurement.

Pre-preparation of raw data

Measurements were made for each SK from two samples taken at random from two lots of wheat grain differing in mean values of SK mass. The sample taken from the lot of more massive kernels contained 173 kernels, while the sample taken from the lot of less massive kernels contained 195 kernels. The dependence of SK density versus mass (cf. Fig. 1) showed some data points in both samples considerably deviating from the general pattern.

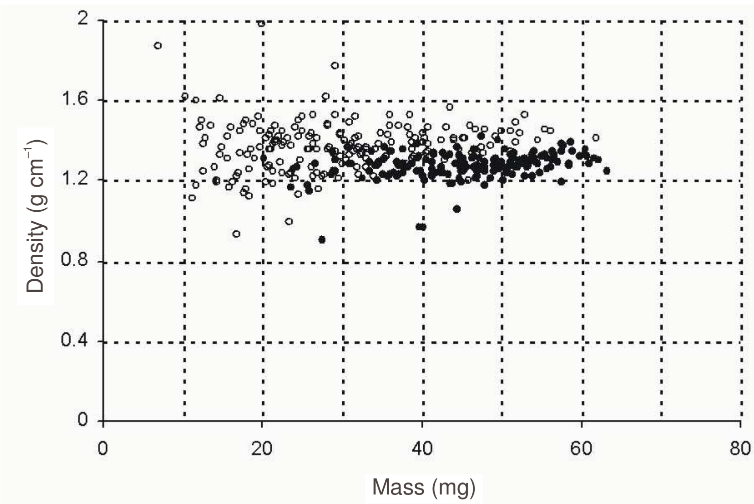

Fig. 1. Single kernel density versus mass for raw data: (•) for massive, and light (o) kernels

These points can be classified as outliers. Most likely the reasons for the presence of outliers were possible mistakes in readouts, and/or in writing (by hand) the results of readouts, as well as closed voids present in some kernels. In order to prevent results of further analysis from disturbance due to adverse influence of outlying data, they were rejected from both series, as well as ca. 2% of the data that gave the most extreme values for measured SK volume. Figure 2 shows the plots of density versus SK mass for both samples with rejected outliers. All considerations in the following apply to corrected series, containing now 166 (sample of massive kernels) and 188 (light kernels) data points.

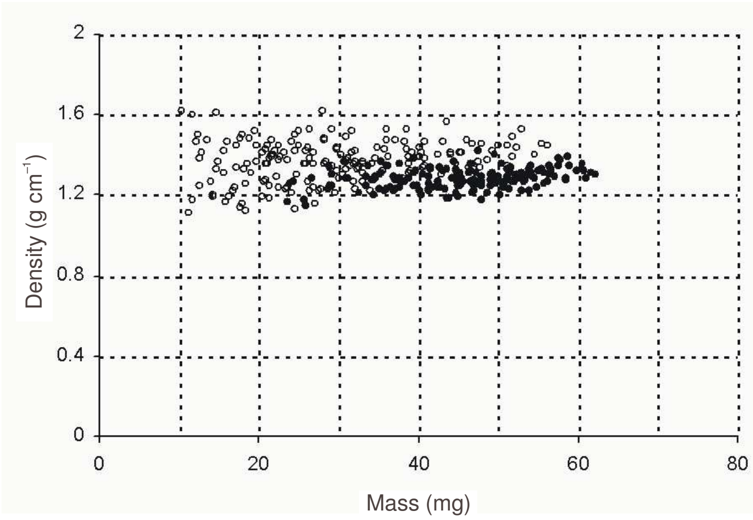

Fig. 2. Same as in Fig. 1 but after rejection of outliers

METHOD FOR DETERMINATION OF ACTUAL SK DENSITY

Uncertainty of the measurements

SK density was calculated from the measurements of mass and volume. It can be seen from Figure 2 that data points tend to scatter more with the decrease of SK mass. This is especially visible for the series of less massive kernels. The scatter of data points can be expected to be caused by two sources of variability. The first source can be referred to the property of the set of kernels (the actual SK variability), while the second one can be referred to the uncertainty of the measurements. Therefore, in order to evaluate the actual SK variability, it is necessary to subtract the variability caused by the uncertainty of the measurements from the results of measurements. This needs the latter factor to be evaluated and then extracted from raw results of the measurements.

With this aim, for four single kernels the measurements of mass and volume were repeated 15 times in order to determine uncertainties $u_C(m)$ and $u_C(v)$ for the mass and volume, respectively. Basing on the definition of density, $u_C(d)$ were calculated as well. The results are set in Table 1. By virtue of density definition, the dependence between the uncertainties is:

$$\left(\frac{u_C(d)}{d}\right)^2 = \left(\frac{u_C(m)}{m}\right)^2 + \left(\frac{u_C(v)}{v}\right)^2. \tag{2}$$

This dependence can be used in order to calculate relative uncertainty of d from relative uncertainties of m and v. It follows from Table 1 that $u_C(m)$ and $u_C(v)$ are practically independent of v and m, at least within the range where the measurements were made. Mean values for the uncertainties are 0.0733 mg and 1.10 µl, respectively. These findings give a possibility to predict $u(d)/d$ dependence on SK mass. The dependence is:

$$\frac{u_C(d)}{d} = \frac{1}{m}\sqrt{(u_C(m))^2 + (d \cdot u_C(v))^2}, \tag{3}$$

i.e. assuming SK density to be approximately constant, $u(d)/d$ is inversely proportional to SK mass. Numerical values from Table 1 lead to a simple relationship

$$u_C(d)/d = 150/m\ (\%), \tag{3a}$$

characterising the uncertainty of SK density in the present measurements.

Table 1. Means and standard deviations for SK mass and volume measurements and calculated SK density obtained for four single kernels and series of 15 measurements

Measurements of	Single kernel 1	2	3	4	Mean
Mass (mg)					
m (mean)	12.91	20.86	41.66	59.50	
$u_C(m)$	0.0779	0.0715	0.0729	0.0710	0.0733 ±0.0031
Volume (µl)					
v (mean)	9.44	16.08	30.52	42.53	
$u_C(v)$	1.14	1.13	1.09	1.05	1.10 ±0.04
Density (g cm^{-3})					
d (mean)	1.373	1.299	1.365	1.400	1.359 ±0.043
$u_C(d)$	0.165	0.0914	0.0487	0.0350	
$u(d)/d$ (%)	12.0	7.04	3.57	2.50	

Correction for the effect of the measurements

The two factors mentioned above contributing to the observed variability can be assumed to contribute independently. Thus one can write:

$$u_{act}^2(d) = u_{obs}^2(d) - u_{meas}^2(d), \tag{4}$$

and calculate the effect of actual variability, provided the measure of the observed variability is known. The latter factor can be found from experimental $d=d(m)$ dependence as follows. First, linear approximation of this relationship has to be determined. The approximations resulted in:

$$d_{appr}^h = 1.2043 + 0.0017 \cdot m \quad \text{and} \quad d_{appr}^l = 1.3194 + 0.0016 \cdot m\ , \tag{5}$$

for heavy (massive) and light kernels, respectively. Next, for each kernel of a given mass its density from relevant approximation (5) is calculated, as well as the difference between the approximated and measured densities, Δ_d. Because of (3a) the dependence of squared differences, $(\Delta_d)^2$, on SK mass can be expected to obey the formula:

$$u_{obs}^2(d) = \frac{a}{m^2} + b\ . \tag{6}$$

Constants a and b can be determined as follows: the whole range of mass is divided into several subranges of equal width, the mean value of $(\Delta_d)^2$ in each subrange calculated, and the values of a and b determined by the least squares approximation of Eq. (6) to the series of the means. This procedure was applied to the data for massive kernels only (because the effect of measurement uncertainty was considerably greater) and repeated for several numbers of mass subranges. The resultant a and b values gave the mean values $a = 4.32 \pm 0.30$ and $b = 0.0027 \pm 0.0004$. Because of Eq. (3a), the squared product of 1.50 and the mean density 1.36 (see Tab. 1) provides an alternative evaluation of the value of a. This calculation gives 4.16, i.e. the value that agrees quite well with 4.32. This means that the proposed approach for the explanation of the observed scatter in data points seems to be reasonable, and can be really attributed to the inaccuracy of the measurements.

The observed distribution of SK density can be now corrected for the measurement effect in the following way. The difference between the approximated and measured SK density for a SK of given mass, Δ_d, has to be reduced by a factor that takes into account the proportion between the variation caused by measuring effect and actual variability in SK properties. Putting Eq. (6) and evaluation Eq. (3a) of $u_{meas}(d)$ into Eq. (4) one gets:

$$u_{act}(d) = u_{obs}(d)\sqrt{1-\left(\frac{u_{meas}(d)}{u_{obs}(d)}\right)^2}$$
$$= u_{obs}(d)\sqrt{1-\left(\frac{4.24}{4.24+0.0027\cdot m^2}\right)^2} \tag{7}$$

where $a=(4.32+4.16)/2=4.24$ and $b=0.0027$ are used. The same relationship should also be fulfilled by the observed Δ_d and its value Δ_d^{act} expected when the effect of measurements is absent. Using Eq. (7) the scatter of data points was recalculated for both samples of grain and standard deviation of Δ_d^{act} determined. The results are shown in Figure 3. One should be aware, however, that positions of particular data points in the figure cannot be interpreted directly, because removing of measurement inaccuracy does not have deterministic character, contrary to statistic parameters that can be directly interpreted. In Table 2 the values of standard deviations for raw results of SK density, for not corrected, Δ_d, and corrected, Δ_d^{act}, data are set together. A visible lowering of the scatter of data points for light kernels after correction can be found by comparing Figs 2 and 3.

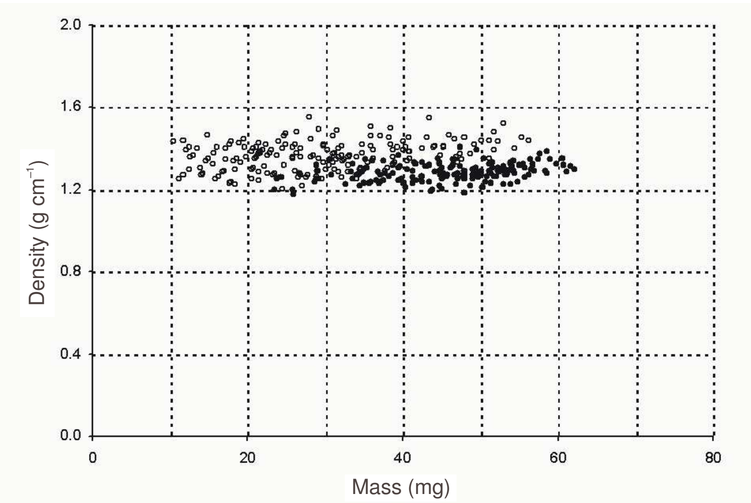

Fig. 3. Same as in Fig. 2 but after SK density correction for the effect of measurement inaccuracy

This result is described quantitatively by considerably lowering relevant standard deviations that can be found in Table 2.

Table 2. Mean SK mass and density and standard deviations for samples of massive and light kernels. Standard deviations of the density were calculated for raw data and for deviations from approximated dependence (5) of density on mass before and after correction removing the scatter of data points due to inaccuracy of measurements

Type of kernels	Mean SK mass (mg)	Mean SK density (g cm^{-3})	Density standard deviation for		
			raw data	not corrected	corrected
massive	45.3 ± 8.9	1.282	0.049	0.047	0.041
light	30.4 ± 11.5	1.363	0.102	0.100	0.066

The final results from the measurements presented above, made on two samples of grain, and from the analysis of measurement inaccuracy allow one to state that SK density values in the samples were found to increase a little with SK mass in accordance with the formula given by Eq. (5). Slopes in linear relationships for both samples were practically the same, contrary to standard deviations that were considerably greater for light kernels. The latter finding holds even after correction for inaccuracy of the measurements.

CONCLUSIONS

1. Presented analysis shows that the set of simple measuring devices can be effectively used for the determination of SK density.

2. Direct results of the measurements for lightest kernels are considerably affected by inaccuracy of the measurements, however, this effect can be removed by the procedure described and applied in the paper.

3. It has been found that, on average, massive SKs were less dense than light SKs in both samples.

4. SK density in the sample of massive kernels was lower than in the sample of lighter kernels.

5. Internal variability of SK density was found to be considerably greater for the sample of light kernels.

REFERENCES

1. **Chang C.S.:** Measuring density and porosity of grain kernels using a gas pycnometer. Cereal Chemistry, 65, 13-15, 1988.
2. **Fang C., Campbell G.M.:** Effect of measurement method and moisture content on wheat density measurement. Food and Bioproducts Processing, 78, 179-186, 2000.
3. **Kohlrausch F.:** Laboratory Physics (in Polish). PWN Warszawa, 1959.
4. **Tkachuk R., Dexter J.E., and Tipples K.H.:** Removal of sprouted kernels from hard red spring wheat with a specific gravity table. Cereal Chemistry, 68, 390-395, 1991.
5. **Tkachuk R., Dexter J.E., Tipples K.H., Nowicki T.W.:** Removal by specific gravity table of tombstone and associated trichotecenes from wheat infected with Fusarium Head Blight. Cereal Chemistry, 68, 428-431, 1991.

ANALIZA DOKŁADNOŚCI POMIARÓW GĘSTOŚCI POJEDYNCZYCH ZIARNIAKÓW WYKONANYCH ZA POMOCĄ PROSTEGO ZESTAWU POMIAROWEGO

Ryszard Siuda, Mikołaj Rozwadowski

Instytut Matematyki i Fizyki, Akademia Techniczno-Rolnicza
ul. S.Kaliskiego 7, 85-796 Bydgoszcz
e-mail: siuda@atr.bydgoszcz.pl

S t r e s z c z e n i e. Zestaw pomiarowy złożony z wagi elektronicznej oraz własnej konstrukcji piknometeru powietrznego został użyty do wyznaczania gęstości pojedynczych ziarniaków poprzez pomiar ich masy i objętości. Pomiary przeprowadzono dla reprezentatywnych próbek wziętych z dwu bardziej licznych prób ziarna pszenicy różniącego się średnią masą ziarniaków (45,3 i 30,4 mg). Stwierdzono, że wyniki otrzymane dla lekkich ziarniaków były znacząco obciążone niedokładnością pomiarów (nawet do ok. 15% wartości gęstości w przypadku najlżejszych ziarniaków). Ta niedokładność pomiarów może być skorygowana za pomocą odpowiedniej procedury. Skorygowane wyniki wykazały, że ziarniaki z próbki cięższych ziarniaków miały mniejszą średnią gęstość (1,282 $g\cdot cm^{-3}$) niż ziarniaki z próbki ziarniaków lżejszych (1,363 $g\cdot cm^{-3}$). W obu próbkach stwierdzono liniowy wzrost gęstości ziarniaków ze wzrostem ich masy (współczynnik nachylenia ok. 0,0017 $g\cdot cm^{-3}\cdot mg^{-1}$). Standardowe odchylenie wokół liniowych zależności po korekcji niedokładności pomiarów było większe dla próbki ziarniaków lżejszych (odpowiednio 0,066 i 0,041 $g\cdot cm^{-3}$) i wyraźnie zmalało w stosunku do wartości przed korekcją (odpowiednio 0,100 i 0,047 $g\cdot cm^{-3}$).

S ł o w a k l u c z o w e: gęstość pojedynczego ziarniaka, masa pojedynczego ziarniaka, objętość pojedynczego ziarniaka, właściwości fizyczne pojedynczego ziarniaka