Acta Scientiarum
http://www.periodicos.uem.br/ojs/
ISSN on-line: 1807-8621
Doi: 10.4025/actasciagron.v47i1.69343

Accuracy assessment of bulk density measurement methods across different soil management practices: sample volume- and paraffin temperature-related errors

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ABSTRACT. Soil bulk density (BD) serves as a crucial physical property for characterizing soils and assessing the quality of their management systems. Various methods, including the Core, Clod, and Jolly balance (JBM) methods, are employed for BD measurement. However, these methods can yield significantly different measurements due to analytical errors. This study aims to assess the accuracy of these methods in a clayey Oxisol under different management conditions, while also identifying primary experimental errors in BD determination and strategies for their mitigation. Different statistical approaches were employed to analyze the impacts of sample volume, paraffin temperature, and management systems on BD determination methods. Method accuracy exhibited variation among management systems, particularly notable in secondary forest (SF) areas. In these areas, Core-based BD measurements were 37% lower than those obtained by the Clod and JBM methods. This disparity can be attributed to the higher macroporosity observed in SF, leading to greater sample volume loss and smaller volumes analyzed by the Clod and JBM. A correlation between paraffin temperature for sample coating and clod volume was observed, with paraffin temperature affecting BD measurements only in clods larger than 69.9 cm^3. The paraffin temperature inducing the lowest mean error for larger clods was 92°C. For clods smaller than 69.9 cm^3, BD measurement errors arose due to inadequate sample volume. Representative elementary volume was identified as a means to mitigate BD overvaluation by the Clod method. A volume of 99 cm^3 proved effective in reducing mean BD errors to 5%, making it suitable for both field sampling and laboratory analytical procedures.

Keywords: Core method; Jolly balance method; paraffin-coated clod method; representative elementary volume; analysis of covariance.

Received on August 19, 2023.
Accepted on March 1, 2024.

Introduction

Soil quality encompasses physical, chemical, and biological properties that reflect soil functionality (Aratani, Freddi, Centurion, & Andrioli, 2009). Among the physical properties, soil bulk density (BD) is crucial for agronomic studies as it informs about porosity, hydraulic conductivity, air diffusivity, and soil compaction. BD is also integral to calculations of soil nutrient and carbon stocks, water availability, and irrigation requirements (Al-Shammary et al., 2018).

Bulk density is influenced by factors such as textural class, mineralogy, organic matter content, soil use, and management practices (Silva et al., 2020; Pareja-Sánchez et al., 2017). High BD may signal the need for mechanical soil decompaction in soil management (Gonçalves, Marasca, Souza, Tavares, & Silva, 2013; Drescher et al., 2017), underscoring the importance of its accurate determination.

Grossman and Reinsch (2002) and Al-Shammary et al. (2018) highlighted several methods for BD determination, including those employing samples with standardized volumes such as the Core method (CrM), and those with irregular volumes such as the paraffin-coated clod method (CdM). Alternative methods have been proposed (Auler, Pires, Brinatti, & Saab, 2017; Camargo, Pires, Brinatti, & Saab, 2020), including nuclear methods (Pires, 2018), photogrammetry (Whiting, Salley, James, Karl, & Brungard, 2020), and pedotransfer functions (Freitas, Armindo, Pires, Swinka Filho, & Ribeiro Júnior, 2019), both for regular and irregular volume samples. Still, given their However, it is important to note that the selection of methods should consider the specific characteristics of the site where they will be applied, the timing of application, and factors such as laboratory and field measurements, as well as associated costs (Pires, Mooney, Auler, Atkinson, & Sturrock, 2019a).

Core, the standard sampling method, faces criticism for potential soil compaction during penetration (Pires et al., 2004) and sampling limitations in certain soil conditions (Grossman & Reinsch, 2002). Clod, the second most utilized technique (Al Shammary et al., 2018), involves coating samples with paraffin and measuring volume displacement using Archimedes' Principle (Auler et al., 2017). While advantageous for studying aggregate BD (Ahmadi, Neyshabouri, Rouhipour, & Asadi, 2011; Uteau, Pagenkemper, Peth, & Horn, 2013; Auler et al., 2017), Clod may overestimate BD due to paraffin infiltration and overlooking macropores, but these errors are not easily quantified (Pires et al., 2019a).

The influence of elementary sizes on soil physical property measurements, particularly in the context of nuclear methods for determining bulk density (BD), has been extensively discussed in the literature (Pires et al., 2004; Ferreira, Borges, & Pires, 2015). Although it is acknowledged that clod volume poses a limitation to the accuracy of BD measurements using the CdM, research on the specific impact of clod volume on BD measurements remains lacking (Al-Shammary et al., 2018).

Theoretically, changes in paraffin temperature could impact the depth to which paraffin penetrates clods during coating. According to Rossi, Hirmas, Graham, and Sternberg (2008), fluctuations in paraffin temperature, as well as soil texture, may influence the extent of paraffin penetration into soil pores, leading to changes in coating thickness and potentially resulting in overestimated results. Despite the widespread recognition of this hypothesis (Auler et al., 2017; Pires et al., 2019a; Camargo et al., 2020), few studies have directly measured this effect. Pires et al. (2019a) conducted a demonstration using eight samples and documented the effects of paraffin temperature on coating. However, other researchers either do not specify temperature control details or fail to identify an ideal temperature to mitigate this issue.

Auler et al. (2017) introduced the Jolly Balance Method (JBM) as an alternative for BD measurements of paraffin-coated clods, aiming to mitigate overestimation compared to the CrM. The JBM uses dynamometers instead of analytical balances to gauge clod force. It operates by balancing the restoring force on a spring scale with the force exerted by a soil clod submerged in a fluid, typically water, to ascertain clod density. This method integrates Archimedes' principle and Hooke's Law. Density calculations account for spring deflections of the material suspended in air, both before and after coating, and while submerged in water, with consideration for water density. Following measurements on soil samples, the authors concluded that the JBM, employing a dynamometer and Hooke's Law, yields more accurate BD measurements compared to the CdM, as the estimation of buoyancy force is more precise. However, as the JBM relies on coated samples, variations in paraffin temperature can potentially impact measurement accuracy.

Given the significance of exploring alternative methods for determining BD and pinpointing sources of measurement error, this study aimed to: i) compare the accuracy of BD determination methods across various soil management practices and identify properties contributing to analytical errors; ii) investigate the potential interdependency between sample volume and paraffin temperature impacting BD measurements; and iii) characterize the relationship between sample volume and BD measurements to determine a representative elementary volume. It is crucial to note that identical soil samples were used for all methods. Samples were collected from diverse management systems to broaden the spectrum of BD values, facilitating more robust data comparisons without confounding variations attributable to sample disparities between BD determination methods.

Material and methods

Experiment location and description

The study site is at the Institute for Rural Development (IDR) of Paraná in Ponta Grossa, Paraná State, Brazil (25°09′ S, 50°09′ W, 865 m above sea level). According to Köppen's classification, the region has a mesothermal climate, with humidity in both summer and winter and cold winters (Cfb). The average yearly temperature is 18.5°C, and annual rainfall is 1,545 mm (Alvares, Stape, Sentelhas, Gonçalves, & Sparovek, 2013). The soil is classified as "Latossolo Vermelho" in the Brazilian Soil System (Santos et al., 2018), or Rhodic Hapludox in the US Soil Taxonomy (Soil Survey Staff, 2014). The soil profiles are deep and well-structured, with high porosity and good drainage (Sá et al., 2015). Key minerals include gibbsite, kaolinite, hematite, goethite, and quartz (Gonçalves et al., 2008). Table 1 details soil chemical properties and clay contents.

Table 1. Soil chemical properties, organic carbon (SOC) content from 0-0.10 m[(1)] depth layers, and clay content from 0-0.20 m[(2)] depth layers of a Rhodic Hapludox under different soil management systems (Secondary Forest [SF], No-tillage [NT], Minimum tillage [MT], and Conventional tillage [CT]).

Soil management	pH	Al^{3+}	Ca^{2+}	Mg^{2+}	K^{+}	SOC	Clay
	$CaCl_2$	$cmol_c\ dm^{-3}$				$g\ dm^{-3}$	$g\ kg^{-1}$
SF	3.97	1.35	2.23	1.80	0.31	36.23	593
NT	4.20	0.75	3.30	3.63	0.33	29.84	681
MT	4.41	1.02	2.02	1.30	0.29	29.09	637
CT	3.99	1.95	3.45	6.22	0.39	29.02	619

[(1)]Adapted from Kazmierczak et al. (2020). [(2)]Adapted from Sá et al. (2015).

The history of the study site, with a slope ranging from 0.02 to 0.07 m m^{-1}, reveals that a portion of the native forest was converted into pastureland in 1967, with a plowing depth of 20 cm. After 11 years of grazing, the area transitioned to crop cultivation in 1978. In 1981, soil cultivation practices involving both no-tillage (NT) and conventional tillage (CT) were implemented in separate 10,000 m^2 plots. Thereafter, in 1989, the CT area was subdivided, and minimum tillage (MT) practices were introduced. CT involved the use of a disk plow to mobilize the soil at a depth of 0-0.20 m, followed by two leveling harrows. In MT, a power harrow was applied at the same depth, followed by two leveling harrows. NT practices restricted soil disturbance to the sowing line (Sá et al., 2015). The secondary forest (SF) resulted from natural succession processes since 1940 and consists of species typical of the Mixed Ombrophilous Forest, also known as Atlantic rainforest *sensu stricto* (Liebsch & Mikich, 2009), along with introduced *Eucalyptus* spp. during restocking.

Soil sampling

In November 2017, 36 years after the implementation of management systems, undisturbed soil samples were collected using volumetric rings with heights ranging from 3 to 5 cm and an external diameter of 5 cm. An Uhland sampler was used to collect samples at the 0-0.10 m layer. A total of 8 samples were randomly collected from each management system and SF. Samples from the management systems were specifically collected between rows of corn crops, with 100 days of tillage in both CT and MT systems. These sampling locations were chosen to investigate potential variations in BD measurements, thereby facilitating the assessment of BD determination method accuracy.

Bulk density measurements

After collection, the samples were sent to the laboratory, where 32 samples were divided into two groups for investigation of: i) the effect of sample volume on BD determination, independent of paraffin temperature; and ii) the effect of paraffin temperature on sample coating. Each group consisted of 4 samples from each management system and SF.

Before subdivision, the samples were saturated by capillary rise and subjected to a tension of -6 kPa on a tension table (model M1-0801, Eijkelkamp®) to determine microporosity (Flint & Flint, 2002a). After achieving hydraulic equilibrium, the samples were dried in an oven with forced air circulation (105°C for 48 hours) for BD determination by the CrM, which calculates the ratio between dry soil mass and volumetric ring volume (Grossman & Reinsch, 2002). The CrM was adopted as the standard method. Total porosity was measured using the ratio between the BD values obtained by the different methods and particle density, obtained by helium pycnometry (Flint & Flint, 2002b). Macroporosity (Ma) was calculated as the difference between total porosity and microporosity (Flint & Flint, 2002a).

After drying and measuring the dry soil mass, and determining BD by the CrM, the samples were carefully removed from the volumetric rings on a Petri dish, and soil mass loss after volumetric ring removal was recorded. Next, the volume of soil with preserved structure, hereafter referred to as clod, was analyzed using other BD determination methods.

Paraffin-coated Clod, using an analytical balance (accuracy of 0.01 g), and the JBM, using dynamometers (accuracy of 0.1 or 0.2 N) and a JBM, were employed as proposed by Auler et al. (2017).

For JBM measurements, dynamometers were calibrated with metal specimens of masses ranging from 10 to 180 g (0.1 to 1.8 N). Specimen masses were determined using an analytical balance, and dynamometer deflection was measured using a digital caliper ruler (accuracy of 0.01 mm). Elastic constants (K) were obtained by applying Hooke's law to these data (Auler et al., 2017).

The first group of samples consisted of clods with volumes between 30.2 and 77.3 cm³, which were attached to a cotton thread for mass measurement. Mass measurements for Clod and force measurements for JBM were then carried out. Clods were submersed four times in granulated paraffin (Synth®), whose melting temperature is 65 ± 3°C, for coating. After complete coating, mass and force measurements were taken in air and distilled water (18.5°C).

The second group of samples, consisting of clods with volumes ranging from 31.9 to 81.3 cm³, was prepared analogously to the first group. However, samples were coated in paraffin at temperatures of 65 ± 3, 90 ± 3, 120 ± 3, and 150 ± 3°C, based on the results of the first group. In both groups, paraffin was melted in an aluminum flask on a heating plate, and the temperature was controlled using a digital thermometer (accuracy of 0.1°C) placed inside the paraffin container. After coating the clods of the second group, mass and force measurements were conducted as previously described.

Bulk density was calculated according to equations 1 and 2, for Clod and JBM, respectively, using mass and force data:

$$BD_{CdM} = m_c \left[\left(\left(m_{pc\ in\ air} - m_{pc\ sub} \right) WD^{-1} \right) - \left(\left(m_{pc\ in\ air} - m_c \right) PD^{-1} \right) \right]^{-1} \quad (1)$$

$$BD_{JBM} = [\Delta L\ (\Delta L - \Delta L')^{-1}]\ WD \quad (2)$$

where: m_c is the clod mass (g); $m_{pc\ in\ air}$ and $m_{pc\ sub}$ are the paraffin-coated clod masses in air and submersed in water (g), respectively; WD and PD are water and paraffin densities; ΔL and $\Delta L'$ are the dynamometer deflections for the clod in air and submersed in water (N). Note that $\Delta L'$ already disregards the effect of the paraffin buoyant force on the coated clod. Details of $\Delta L'$ calculations are shown in Auler et al. (2017).

Statistical analysis

Data underwent descriptive statistical analysis and were tested for normality of residuals using the Shapiro-Wilk test and for homogeneity of variances using the Bartlett test. Once the assumptions were met, analysis of variance (ANOVA), covariance (ANCOVA), polynomial regression, and linear correlation (Pearson) were performed.

ANOVA was applied considering a completely randomized design (CRD) in a 3 × 4 factorial arrangement (three methods and four soil management systems), with four repetitions for the first group of samples. ANCOVA was applied to the second group of samples, considering the volume of the Clod and JBM samples as a covariate that would affect coating temperature. In both ANOVA and ANCOVA, when the F-value was significant, Tukey's test was used for multiple comparisons of qualitative factors (methods or management systems), and polynomial regression analysis was used for quantitative factors (paraffin temperatures).

Linear correlation analyses were performed with BD results of the different determination methods and with other variables that could affect the results (macroporosity, sample volume, and soil volume loss). All statistical analyses were conducted using R, version 4.0.1 (R Core Team, 2020).

Samples that were not influenced by the pore system or paraffin temperature were selected for the estimation of a representative elementary volume (REV) of clods for BD determination by the Clod method (CdM). To determine the REV, the following approaches were considered: (1) the clod mean and (2) median, considering an infinite population (Miot, 2011); (3) the direct estimation by linear regression between clod volume and BD mean error by the CdM compared to the CrM; (4) the increase of clod volume; and (5) the mean clod volume proportional to the increase in data range (Borges et al., 2018).

Results

Effects of bulk density determination methods on soil management systems: first group of soil samples

The bulk density (BD) of clayey soil was assessed using three analytical methods (Core, Clod, and JBM) with the same soil samples across three soil management systems (NT, MT, and CT) and a secondary forest (SF). As anticipated, Core yielded the lowest BD values ($p < 0.05$) compared to Clod and JBM across all soil management systems (Table 2). No significant differences in BD values were observed between

Clod and JBM, irrespective of the management system. Notably, SF samples exhibited the lowest BD values when analyzed using the Core method (CrM), whereas they displayed the highest BD values when analyzed using Clod and JBM (Table 2). This suggests that these methods may not be suitable for accurately determining BD in areas covered by natural vegetation. Additionally, SF exhibited the largest standard deviations (SD) when analyzed by the CrM, while NT exhibited the highest SD when analyzed by the Clod and JBM methods (Table 2).

Table 2. Descriptive statistics of bulk density (BD) measurements by the Core method (CrM), Clod method (CdM), and Jolly balance method (JBM) across different soil management systems (SMS) at the same coating conditions.

Method	SMS	BD (g cm^{-3})				
		Mean	SD	Min	Max	Median
CrM	SF	0.87 Bb	0.07	0.82	0.98	0.84
	NT	0.94 Bab	0.02	0.91	0.97	0.95
	MT	0.96 Ba	0.02	0.93	0.98	0.97
	CT	0.94 Bab	0.02	0.92	0.93	0.93
CdM	SF	1.37 Aa	0.03	1.33	1.42	1.38
	NT	1.24 Ab	0.04	1.20	1.31	1.23
	MT	1.22 Ab	0.01	1.20	1,.23	1.22
	CT	1.20 Ab	0.02	1.17	1.23	1.20
JBM	SF	1.38 Aa	0.04	1.32	1.44	1.38
	NT	1.21 Ab	0.06	1.15	1.30	1.18
	MT	1.19 Ab	0.03	1.15	1.21	1.19
	CT	1.17 Ab	0.04	1.12	1.21	1.18

SF, NT, MT, and CT comprise the areas under secondary forest, no-tillage, and minimum and conventional tillage systems, respectively. SD, Min, and Max are the standard deviation, minimum values, and maximum values, respectively. Means followed by the same uppercase (BD measurement methods) and lowercase (soil management systems) letters did not differ from each other by the Tukey's test ($p \leq 0.05$).

Clod ($p = 0.06$) and JBM ($p = 0.04$) exhibited a negative correlation in comparison to the CrM (Figure 1). Interestingly, there was a similarity in the linear coefficients of Clod (2.97) and JBM (3.34), but a notable difference in the angular coefficients (-1.84 for Clod and -2.27 for JBM). This suggests that soils with lower BD tended to have overvalued measurements. Notably, the largest errors were observed in SF samples.

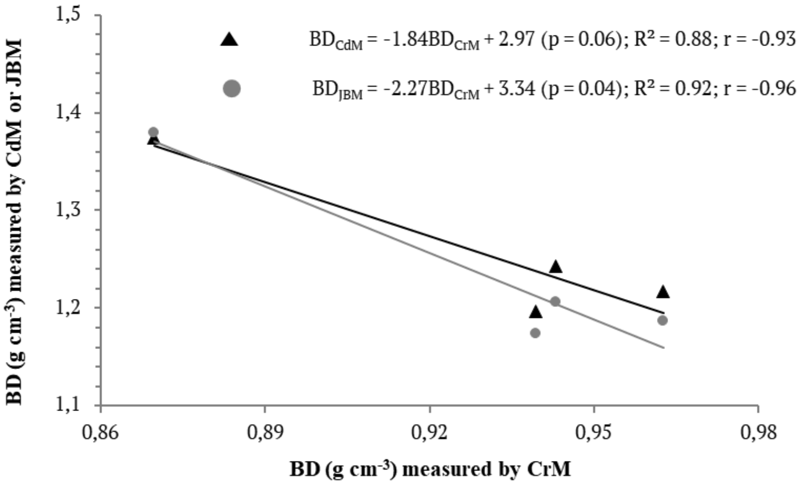

Figure 1. Linear correlation between bulk density (BD) measurements by the Clod (CdM [▲]) or Jolly balance (JBM [●]) method with BD measurements by the Core method (CrM), regardless of the soil management system.

Soil pore system data (Table 3) may elucidate the volume losses observed after BD determination by the CrM, which consequently resulted in smaller sample volumes for Clod and JBM measurements, particularly for SF samples (Table 2). As previously noted, total porosity (TP), macroporosity, and microporosity were solely determined using the CrM. Notably, the averages of TP, macroporosity, and microporosity exhibited significant differences among the soil management systems. SF and CT exhibited the highest TP and macroporosity values, while NT and MT demonstrated the highest microporosity levels. Of particular importance are the relationships observed between macroporosity and microporosity, with the macroporosity of SF and CT being 38 and 79% higher than those of MT and CT, respectively (Table 3).

Table 3. Descriptive statistics of total porosity, macroporosity, and microporosity in the 0-0.10 m depth layer of a clayey Rhodic Hapludox under different soil management systems.

Statistics	SF	NT	MT	CT
		Total porosity (cm^3 cm^{-3})		
Mean	0.62 a	0.57 b	0.59 ab	0.62 a
SD	0.03	0.01	0.01	0.01
Min	0.57	0.56	0.58	0.60
Max	0.64	0.59	0.60	0.62
Median	0.63	0.57	0.58	0.62
		Macroporosity (cm^3 cm^{-3})		
Mean	0.25 a	0.14 b	0.18 ab	0.26 a
SD	0.05	0.03	0.01	0.02
Min	0.17	0.12	0.17	0.22
Max	0.32	0.20	0.20	0.27
Median	0.27	0.12	0.18	0.27
		Microporosity (cm^3 cm^{-3})		
Mean	0.37 b	0.43 a	0.41 ab	0.36 b
SD	0.03	0.03	0.01	0.01
Min	0.32	0.39	0.40	0.35
Max	0.40	0.45	0.41	0.38
Median	0.37	0.44	0.41	0.35

SF, NT, MT, and CT comprise the areas under secondary forest, no-tillage, and minimum and conventional tillage systems, respectively. SD, Min, and Max are the standard deviation, minimum values, and maximum values, respectively. Means followed by the same letters (soil management systems) did not differ from each other by the Tukey's test ($p \leq 0.05$).

As a result of the variations in macroporosity, there was a notable linear increase in soil volume loss following the removal of samples from the CrM. This indicates that greater macroporosity corresponds to greater soil volume losses (Figure 2a). Furthermore, BD measures demonstrated an inverse correlation with sample volume, as observed in both Clod ($p < 0.001$) and JBM ($p = 0.002$) (Figure 2b).

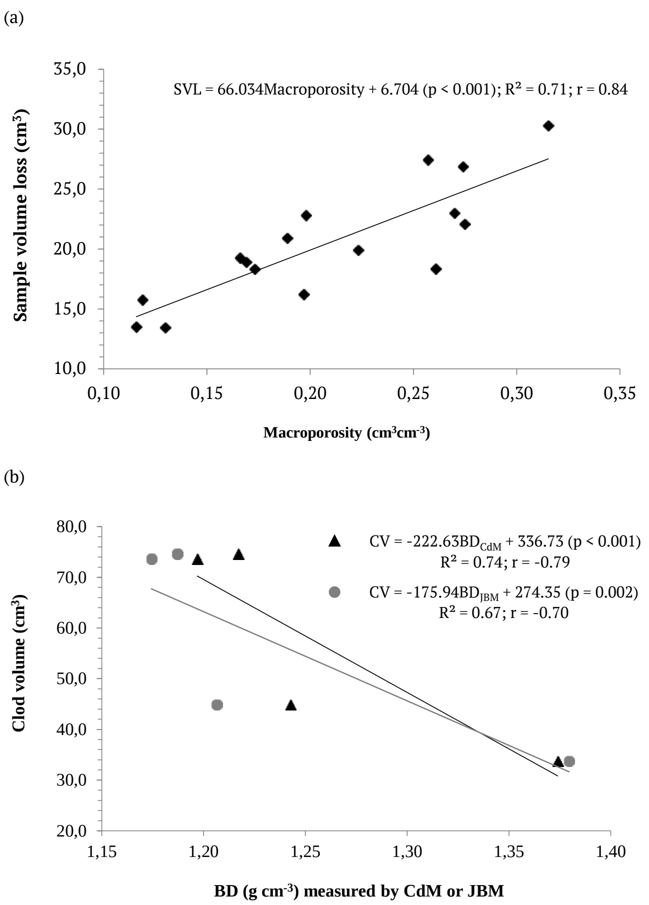

Figure 2. Linear correlation between the sample volume loss (SVL) after bulk density (BD) determination by the Core method (CrM) and the soil macroporosity (a); and linear correlation between the clod volume (CV) and the BD measurement by the Clod (CdM [▲]) and the Jolly balance (JBM [▲]) methods, regardless of the soil management system.

Effect of paraffin temperature on clod coating and interdependency of sample volume on BD determination: second group of samples

The second group of samples, which comprised clods with slightly larger volumes (from 31.9 to 81.3 cm^3) compared to the first group (30.2 to 77.3 cm^3), showed a positive linear correlation of BD measurements by the Clod ($r = 0.63$, $p = 0.05$) and JBM ($r = 0.60$, $p = 0.007$)) methods with those measured by the CrM, regardless of paraffin temperature (Figure 3a). Consequently, there was a trend where larger clod volumes resulted in smaller overvaluation of BD by the Clod ($r = -0.76$, $p < 0.001$) and JBM ($r = -0.74$, $p < 0.001$, Figure 3b) methods.

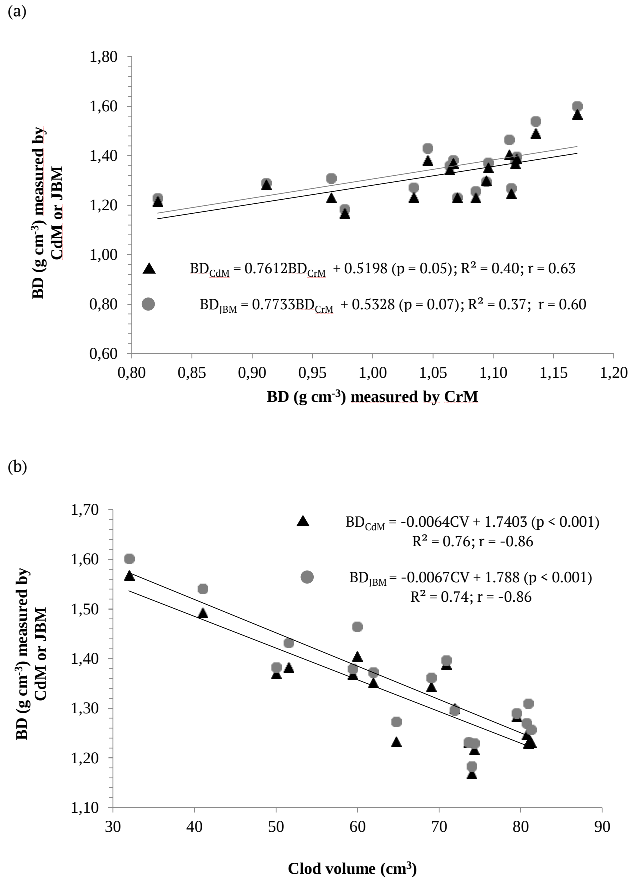

Figure 3. Linear correlation between the bulk density (BD) measurement by the Clod method (CdM [▲]) or Jolly balance method (JBM [●]) with those by the Core method (CrM) (a); and linear correlation with BD measurements by the Clod volume (CV) (b), regardless of the paraffin temperature.

These individual correlations were crucial for ANCOVA's performance. Moreover, the effect of clod volumes was assessed for all temperatures utilized. After ANCOVA, the influence of paraffin temperature was observed, although it tended to be masked by the impact of clod volume. Therefore, it became imperative to identify: (1) the minimum clod volume that is both representative of BD measurements and uninfluenced by paraffin temperature; and (2) the effect of paraffin temperature across different clod volumes.

Clod volume classes were determined using quartile distribution, considering the first quartile (31.2-59.5 cm^3), the interquartile (59.5-74.2 cm^3), and the third quartile (74.2-81.3 cm^3). For the first class, paraffin temperature showed no effect on BD measurement (1.44 ± 0.09 g cm^3) by the Clod ($p = 0.87$) and JBM ($p = 0.79$) methods. In the second class, a non-significant trend towards a quadratic effect was noted ($p = 0.39$), likely due to the small sample size ($n = 8$). Regression analysis could not be performed for the third class because only 4 samples coated at temperatures of 65 and 120°C had a volume greater than 74.2 cm^3. When the t-Student test was applied to BD values (1.23 ± 0.01 g cm^{-3}) for these temperatures, the temperature did not affect BD measurements ($p = 0.14$). Thus, due to the data set, we deduced that the separation in quartiles was not appropriate.

Clod volume classes were redefined based on the median. Two classes were defined: volumes below (31.9-69.9 cm^3) and above (69.9-81.3 cm^3) the median. This classification proved to be satisfactory, as it distributed the dataset homogeneously. For the first class, there was no effect of paraffin temperature on BD measurements for Clod ($p < 0.001$) and JBM ($p < 0.001$). However, a significant quadratic effect was observed for both methods in the largest volume class (Figure 4).

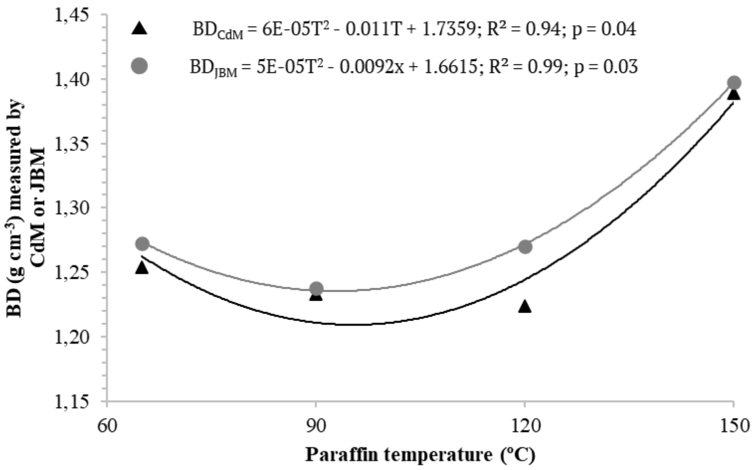

Figure 4. Effects of paraffin temperature (°C) on clod waterproofing for bulk density (BD) measurements by the Clod (CdM [▲]) and Jolly balance (JBM [●]) methods for volumes above 69.9 cm^3.

According to the fitted regression (Figure 4), temperatures near 65°C may induce BD overvaluation by 21.7% and 13.6% using the Clod and JBM methods compared to the CrM. BD overvaluation reduces as paraffin temperature increases, reaching mean errors of 18.4% for Clod and 18.7% for JBM at 91.7 and 92.0°C, respectively. There was again an increase in BD overvaluation, reaching mean errors close to 27% for both methods at temperatures near 150°C. BD overvaluation was higher by the JBM method than by the CdM. However, compared to the CdM, BD overvaluation mean errors through the JBM method were 1.9, 0.4, 4.6, and 0.9% at temperatures of 65, 90, 120, and 150°C, respectively, which are values that could be disregarded.

Covariance analysis was performed, considering clod volume as a covariate, but separating the dataset into these two classes. The assumption of linearity between BD and the clod volume of 69.9-81.3 cm^3 (above the median) was not significant (p = 0.34), suggesting that interferences in BD measurements were only a function of paraffin temperature. In the smallest volume class (31.9-69.9 cm^3), the errors in BD measurements continued to be dependent on clod volume (p = 0.002). This result underscores the need for determining a minimum clod volume for BD determination by the Clod and JBM methods.

Determining representative elementary volume for BD measurement by the Clod method using five simulation methods

After determining how paraffin temperature affects clod coating and the relationship between clod volume and BD measurements, our objective was to determine the representative elementary volume (REV) for BD measurements with minimal overvaluation. To do so, we employed five methods: estimating the REV by considering (1) the mean clod volumes (64.8 ± 14.3 cm^3, n = 29) and (2) the median clod volumes (71.9 ± 14.3 cm^3, n = 29), based on an infinite population (Miot, 2011); (3) direct estimation through simple linear regression between clod volume and BD mean error by the CdM concerning the CrM; (4) estimation based on the increase in clod volume relative to the smallest sample volume (31.9 cm^3), as proposed by Borges et al. (2018); and (5) determining the average clod volume proportional to the increase in data range (n+1). In all these simulations, only the clods that were not influenced by the coating temperature (second group of samples) and the SF (first group of samples) were used. Due to the similarity between the results obtained by the Clod and JBM methods, simulations were conducted solely for Clod, as it exhibited lower overvaluations.

The representative elementary volume (REV) values, estimated by mean and median clod volumes and according to the equation adjusted by linear regression (REV = (ME-44.083) / -0.2656; R^2 = 0.31, p = 0.002), increased considerably within the range of mean allowable errors from 30 to 1% (Table 4). Among these three estimation methods, the median estimation yielded the smallest volumes. REV values estimated by the infinite population mean were 57.4% lower than those obtained by the fitted regression, within the allowable errors of 30 to 10%. For the highest level of accuracy, the difference between these estimation methods was 6% (Table 4).

BD measurements were considered to have reached a REV when the relative difference between BD measured by the Clod and CrM was below 5% in at least 3 consecutive volume variations (Borges et al., 2018).

Based on the increase in sample volume and a minimum volume of 31.9 cm³, REV was observed to begin at 55.7 cm³ (Figure 5a). Considering the augmentation in mean clod volume by expanding the dataset (n+1), the REV was determined to be 50 cm³ (Figure 5b). However, for both methods of REV estimation, the mean error of BD measurements stood at 30%. Under this mode of representative volume determination, estimation at higher confidence levels of accuracy is not attainable, owing to the inherent BD overvaluation of the CdM compared to the Core one for this soil, thus establishing the minimum mean error at 26.9% (Figure 5).

Table 4. Estimation of representative elementary volume (REV) for soil clods using mean or median clod volumes from an infinite population, and via simple linear regression between clod volumes and mean error of BD measurements by the Clod and Core methods (3) within the admissible error range of confidence intervals.

Statistics	BD measurement admissible error						
	30%	25%	20%	15%	10%	5%	1%
Mean clod volume error (cm³)	45.3	48.6	51.8	55.1	58.3	61.5	64.1
Median clod volume error (cm³)	50.3	53.9	57.5	61.1	64.7	68.3	71.2
t - value	2.809	3.125	3.491	3.937	4.525	5.449	7.351
REV (cm³) based on mean clod volume	35.7	41.3	48.3	57.8	72.1	99.0	172.9
REV (cm³) based on median clod volume	32.2	37.2	43.5	52.1	65.0	89.2	155.8
REV (cm³) based on linear regression	53.0	71.8	90.7	109.5	128.3	147.1	162.2

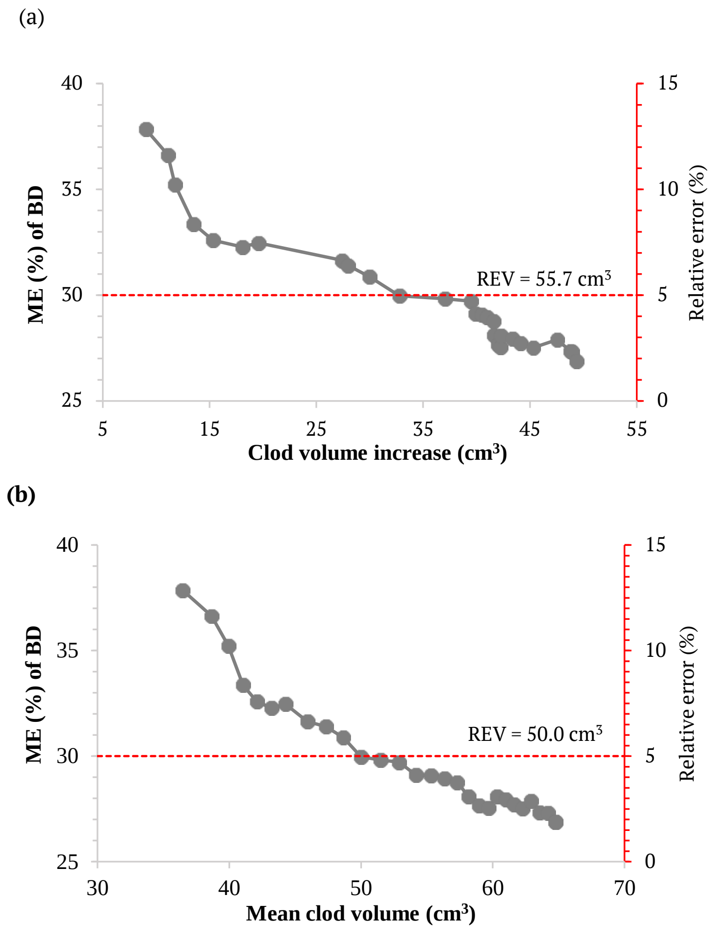

Figure 5. Representative elementary volume (REV) of clods for bulk density (BD) measurement by the CdM, considering a BD mean error (ME) compared to the CrM, based on individual volume increases (a) and mean volume increase with population increase (n+1) (b).

Discussion

Relationships between pore system, volume losses, and clod volume affect BD measurements in soil management systems

When compared to the CrM, the Clod and JBM methods overvalued BD measurements in all soil management systems, particularly for SF (Table 2); however, JBM exhibited higher accuracy compared to Clod

(Figure 1). These findings corroborate those of Auler et al. (2017), highlighting the importance of more accurate buoyancy estimation for better sample volume determination.

Different BD determination methods yielded distinct values for each soil management system (Table 2). The CrM effectively distinguished between management systems concerning SF, which exhibited the lowest BD compared to MT and the highest total porosity, macroporosity, and lowest microporosity than the NT method (Table 3). This outcome aligns with observations commonly reported in areas without anthropic interference. Similarly, increases in total porosity and macroporosity in more superficial layers (0-0.10 m) post-tillage are frequently documented, corroborating CT and MT results. The increase in BD in NT was also anticipated (Kazmierczak et al., 2020; Borges et al., 2019). Although the total porosity and macroporosity of SF and CT were similar, BD values of CT tended to be higher than those of SF, likely due to the higher SOC content in the regeneration forest (Table 1) (Sá et al., 2015).

However, when BD was determined by the Clod or JBM method, the relationship observed for SF was inverse (Table 2), indicating that these methods may not be suitable for determining BD under these conditions. The soil under SF exhibited higher macroporosity (Table 3), resulting in increased soil volume loss and reduced clod volume for BD measurements by the Clod and JBM methods (Figure 2). There exists an inversely proportional relationship between soil pore size and the energy required to break up a soil volume. Soil macropores under SF serve as lines of weakness (Barreto et al., 2009), justifying the higher volume loss.

Lestariningsih, Widianto, and Hairiah (2013) confirmed the inverse correlation between sample volume and BD for the CrM by analyzing two types of soil collection, one with a 44 times larger volume than the other. They concluded that the larger volume sample had the lowest BD, as the smaller the volume of the volumetric ring, the more susceptible the sample is to soil compaction at the time of collection. Considering this, BD overvaluation by methods employing irregular and small-volume samples tends to be exacerbated.

Although the macroporosity of CT and MT resembled that of SF, soil volume loss was not significant in these management systems (Figure 2). A tension of -6 kPa delineates pores between macro and micropores, utilizing a diameter of 50 μm. However, it is impossible to identify the distribution of macropores concerning total porosity (Flint & Flint, 2002a).

In soils with higher SOC contents and minimal anthropic activity, edaphic macrofauna activity is elevated, leading to the formation of biopores. Since biopores are the largest pores of the macropore class and are thus dominant concerning total porosity (Alvarez, Poch, & Osterrieth, 2021), SF soil likely possesses high bioporosity (Borges et al., 2019). Larger pore diameters under SF may also facilitate the entry of paraffin into clods, resulting in elevated BD values (Pires et al., 2019a). This hypothesis may help elucidate the contrast between SF BD measurements by the Clod and JBM methods concerning the CrM (Table 2).

Regardless of the method, as a consequence of soil volume loss post-sample collection, clod volumes analyzed by the Clod and JBM methods were reduced, leading to higher BD values. Thus, BD measurements by the Clod and JBM methods are significantly impacted by sample volume, even under constant paraffin temperature (Figure 2b). Numerous studies have proposed this hypothesis (Pires, Rosa, & Timm, 2011; Uteau et al., 2013; Auler et al., 2017; Al-Shammary et al., 2018), yet none have investigated the determination of a minimum volume required for analyses by the CdM.

Effects of paraffin temperature on waterproofing in clod volume classes

Clod volume dependency was also observed when analyzing the effect of paraffin temperature on clod coating, for both Clod and JBM (Figure 4). Hence, it was imperative to segregate samples into two volume classes, considering the median volume. For the smallest volume class (31.9-69.9 cm^3), BD variations were attributed not to paraffin temperature variation, but to clod volume. Conversely, for the largest class (69.9-81.3 cm^3), although clod volume was no longer a concern, paraffin temperature influenced BD measurements in both methods, with JBM exhibiting greater susceptibility to analytical errors (Figure 4).

Depending on the irregularity and size of the sample, coating often requires multiple submersions in paraffin. With several submersions, paraffin layers accumulate on the clod, enhancing its coating (Pires, Roque, Rosa, & Mooney, 2019b). Hence, prior separation and selection of sample volume for BD determination by the Clod or JBM method is imperative.

At 65 ± 3°C, the paraffin melting temperature utilized here, air bubbles formed between paraffin layers. In soil mass-volume relationships, the air mass is practically negligible, but its volume is not (Reichardt & Timm, 2019). Consequently, these air bubbles interfere with determining the volume of paraffin used in sample coating. This relationship is challenging to measure in the Clod and JBM methods, as the negligible air mass does not elevate the paraffin (experimentally measured value) that coats a sample. However, upon submersion of the clod in water, the volume of paraffin and air bubbles impact the buoyant force, leading to errors in estimating sample volume. Thus, the overvaluation of BD values when the samples were paraffin-coated at 65 ± 3°C can be attributed to these experimental errors.

According to thermodynamic precepts, increasing fluid temperatures alters its viscosity (Reichardt & Timm, 2019). As a consequence, increasing paraffin temperature reduces its viscosity (Ferrer et al., 2017), facilitating clod coating and inhibiting the formation of air bubbles between paraffin layers. Nonetheless, this temperature rise does not adhere to a linear model (Figure 4). Therefore, deriving the fitted regressions, the optimal paraffin melting temperatures obtained, resulting in lower errors in BD measurements, were 91.7 and 92°C for the Clod and JBM methods, respectively. Beyond these temperatures, errors in BD measurements increase proportionally with rising temperature, peaking at 150°C (Figure 4). At the highest temperatures, 120 and 150°C, reductions in paraffin viscosity (Ferrer et al., 2017) may have facilitated its entry into macropores, inducing the largest BD measurement errors (Pires et al., 2019a). In this context, the lack of dependency on paraffin temperature for the smaller clod volume class may be linked to the lower macroporosity of these samples (Figure 2a), precluding paraffin entry. For situations where paraffin temperature control is unfeasible, preference should be given to samples of smaller volumes. Nevertheless, BD measurements would still be impacted by reduced clod volumes.

Representative elementary volume of clods for BD determination by the Clod method: analytical and field-sampling considerations

Experimental measurements were influenced by clod volume even when paraffin temperature was controlled. This underscores the need for a representative elementary volume (REV) to minimize experimental errors (Borges et al., 2018). The volume of samples used in BD determination by the CrM, also employed for REV simulation, ranged from 58.8 to 97.4 cm³, with a mean, median, and standard deviation of 89.8, 94.5, and 12.3 cm³, respectively. BD values ranged from 0.82 to 1.17 g cm^{-3}, with mean and standard deviation of 1.01 and 0.09 g cm^{-3}, respectively. Although sample volume varied, BD values measured by the CrM were minimally affected. These variations are attributed to structural differences between soil management systems (Tables 2 and 3).

The CdM yielded mean BD determination errors when compared to those of the CrM, ranging from 13.2 to 39.9%, and were proportional to clod volume (r = -0.56, p = 0.002). REV determination considered mean admissible errors ranging from 30 to 1%, seeking to bolster the results' confidence level. REV simulation considering mean and median clod volumes, based on the estimation of an infinite population (Miot, 2011), approximated mean and median sample volumes in volumetric rings with a 5% accuracy level (Table 5).

However, enhancing measurement accuracy to 1% resulted in an extensive REV (exceeding 150 cm³), complicating sample collection and analytical determinations. Clod REV, defined by mean and median (Table 5), equates to a volumetric ring with internal diameters of 6.6 and 6.3 cm and a standard height of 5 cm, measurements rarely used in the Core sampling. Similarly, the REV of 172.9 cm³ equates to a sphere with a diameter of 6.9 cm, underscoring the challenges of sample collection and analytical determinations.

The REV values of 99.0 and 89.2 cm³, set at the 5% admissible error, may be influenced by paraffin temperature (Figure 4). Hence, REV should be combined with optimal paraffin temperature to minimize experimental errors. When collecting large clods is infeasible, a REV with admissible error of 10 to 15% suffices. Volumes of 57.8 and 72.1 cm³ equate to sphere diameters of 4.8 and 5.2 cm, respectively, demonstrating the feasibility of collecting samples of this size.

The REV values estimated by linear regression of BD mean errors by the CdM regarding the CrM and clod volumes surpassed those based on infinite populations at all standardized confidence levels. Although this estimation was numerically simpler, it was unsatisfactory, as volumes exceeding 100 cm³ are required to achieve a minimum accuracy level of 15% (Table 5). REVs based on regression, adjusted for mean error levels below 10%, surpass the limits (13.2-39.9%) used for linear regression fit, and although significant (p = 0.002), the coefficient of determination was low (R^2 = 0.31), suggesting potential errors with this criterion.

The REVs considering maximum relative errors of 5%, accounting for sample volume increase from the smallest volume (31.9 cm³), and average volume increase from dataset increase (n+1), yielded comparable results (difference of 5.7 cm³), yet resulted in BD measurement errors around 30% (Figure 5). With these methods, although simulating mean errors below 26.9% and increasing result accuracy was unattainable, the trend of lower overvaluation of measurements proportional to sample volume increase persisted.

It is worth noting that adopting the 5% relative error criterion was purely empirical, based on interpreting the third law of thermodynamics (Borges et al., 2018), without considering any statistical distribution. Using the t-distribution, accuracy levels for REV can be established. This analysis revealed that even at the highest accuracy level ($t_{0.01,\,27} = 6.857$) of REV (47.8 cm³), the mean BD measurement error remains around 30%. This suggests that adopting the 5% maximum relative error criterion is no longer empirical but demonstrates that estimating REV by increasing sample volume, as presented by Borges et al. (2018), was unsatisfactory for this determination. One plausible reason for such inconsistency is the narrow range of volume additions, from the minimum volume of 31.9 cm³ (from 9.0 to 49.3 cm³) or the average volume (36.5 to 64.8 cm³), considering increased sample numbers (n+1).

Conclusion

The choice of method for determining bulk density (BD) significantly impacts result representativeness, which is influenced by the soil management system, sample volume, and paraffin temperature used in the Clod (CdM) and Jolly balance (JBM) methods, compared to the Core (CrM) method. The CrM exhibits fewer analytical issues compared to the CdM and JBM. These methods display a low correlation with the Core one but a high correlation with each other. Samples from secondary forest exhibit the greatest BD disparities among determination methods. While the CrM shows the lowest BD for the secondary forest, Clod and JBM yield the highest BD. This discrepancy correlates with volume loss in secondary forest samples due to higher soil macroporosity, resulting in reduced clod volumes analyzed by the CdM and JBM methods. Clod volume also influences paraffin temperature behavior during sample coating. In clods smaller than 69.9 cm³, temperature does not affect BD measurements. However, larger clods exhibit quadratic behavior concerning paraffin temperature and BD measurements. The optimal paraffin temperature for coating, resulting in reduced errors, is 92°C, contrary to the commonly reported 65°C (melting temperature) in the literature. JBM proves the most sensitive among methods to experimental errors induced by paraffin temperature. The representative elementary volume most effective in minimizing BD measurement error, feasible for collection and analytical procedures, is 99 cm³. With this volume, the maximum allowable error in estimating BD is 5%. Nonetheless, the sensitivity of this volume to coating procedures should be emphasized. In the future, the representative elementary volumes defined in this study should be considered and implemented for BD measurements in soils with significant texture and structure variations. Evaluations should also account for different coating temperature conditions and various coating agents and fluids, besides water. Specifically, novel studies should focus on optimizing buoyant force determination to enhance sample volume estimation.

Acknowledgements

To the CAPES (Coordination for Improvement of Higher Education Personnel) and the CNPq (National Council for Scientific and Technological Development) for the master's and PhD scholarships granted.

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