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# Alcohol concentrations for determining soil particle density using the volumetric glassware method

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**Abstract:** The objective of this study was to evaluate the efficiency of alcohol in concentrations of 0.0, 47.6, 70 and 99 °GL, to determine the soil particle density ($\rho_p$) with the Volumetric Glassware Method (*VG*), verifying the best time of analysis and the possibility of alternatives to 99 °GL alcohol concentration. The soil samples were collected in five experimental areas of the Fundação ABC (Arapoti, Castro, Itaberá, Ponta Grossa and Tibagi), at 0.0-0.20 m depth, stored in a plastic bag and forwarded to the Soil Physics laboratory of the Federal University of Paraná. Analyzes with alcohol concentrations were performed with five repetitions, for each location, totaling 100 samples. The comparison of the "$\rho_p$ obtained with *VG* and 99 °GL alcohol" ($\rho_{pVG.99}$) *vs* "$\rho_p$ with alternative methods" (water: $\rho_{pVG.w}$; 47.6 °GL alcohol: $\rho_{pVG.47.6}$; and 70 °GL alcohol: $\rho_{pVG.70}$) values were performed considering linear regression analysis, *Box Plot* diagram, coefficient of variation (*CV*) and determination ($R^2$), index of agreement (*d*) and mean absolute error (*MAE*). The 70 °GL concentration obtained good associations with the 99 °GL concentration ($R^2 > 0.82$), as well as low values of *CV* and *MAE*. The other concentrations analyzed had promising results. For the soils analyzed, the 70 °GL alcohol concentration can replace 99 °GL to obtain $\rho_p$ values with the Volumetric Glassware Modified Method, requiring data readings after 48 h.

**Keywords**: soil physical attribute, soil particle density, methodology.

## Concentrações de álcool para determinação da massa específica das partículas do solo com o método do balão volumétrico

**Resumo:** Teve-se por objetivo no presente estudo avaliar a eficiência do álcool nas concentrações 0.0, 47.6, 70 e 99 °GL, para determinar a massa específica das partículas do solo ($\rho_p$) com o Método do Balão Volumétrico (*BV*), verificando o melhor tempo de análise e a possibilidade de alternativas à concentração de álcool 99 °GL. As amostras de solo foram coletadas em cinco áreas experimentais da Fundação ABC (Arapoti, Castro, Itaberá, Ponta Grossa e Tibagi), na profundidade de 0.0-0.20 m, armazenadas em saco plástico e levadas ao laboratório de Física do Solo, da Universidade Federal do Paraná. As análises com as concentrações de álcool foram realizadas com cinco repetições, para cada localidade, totalizando 100 amostras. A comparação dos valores de "$\rho_p$ obtidas com *BV* e álcool 99 °GL" ($\rho_{pBV.99}$) *vs* "$\rho_p$ com métodos alternativos" (água: $\rho_{pBV.w}$; álcool 47.6 °GL: $\rho_{pBV.47.6}$; e álcool 70 °GL: $\rho_{pBV.70}$) foram realizadas considerando análise de regressão linear, diagrama *Box Plot*, coeficientes de variação (*CV*) e determinação ($R^2$), índice de concordância (*d*) e erro absoluto médio (*MAE*). A concentração 70 °GL obteve boas associações com a concentração 99 °GL ($R^2 > 0.82$), bem como baixos valores de *CV* e *MAE*.

As demais concentrações analisadas tiveram resultados promissores. Para os solos analisados, a concentração do álcool 70 ºGL pode substituir a 99 ºGL para obtenção de valores de $\rho_p$ com o Método do Balão Volumétrico Modificado, sendo necessário que as leituras dos dados sejam feitas após às 48 h.

**Palavras-chave:** atributo físico do solo, densidade de partícula, metodologia.

## Introduction

Usually the quality of agricultural soil is considered based on physical, chemical and biological aspects, being important to assess the extent of soil degradation or improvement, as well as to identify the sustainability of management systems. Some soil physical attributes, such as the soil density (particles and soil), total porosity, soil penetration resistance, aggregate stability, volumetric water content, available water and hydraulic conductivity, can be used as indicators of the soil quality (Assis et al., 2019; Bertollo and Levien, 2019; Bonfante et al., 2019).

The soil particle density ($\rho_p$) is one of the essential physical properties of the soil, being defined as the mass per unit volume of the solid components. An exact measurement of $\rho_p$ is required, as the parameter is used in most mathematical expressions in which the volume or weight of a soil sample is being considered (Rosa et al., 2018; Dourado et al., 2019).

The most common methods for $\rho_p$ determination are based on quantifying the displacement of the volume of a liquid or air, provided by a soil sample of known weight. Other possibilities are the $\rho_p$ determination with the relationship between organic matter (*OM*) and organic carbon (*OC*). However, the difficulty of the method is the requirement of previous *OM* and *OC* data (Gubiani et al., 2006; Braida et al., 2010).

Two methods based on volume displacement stand out in $\rho_p$ analysis in the laboratory: pycnometer and volumetric glassware method (*VG*), both of which are simple, direct and precise if done with criteria (Blake and Hartge, 1986). Gubiani et al. (2006) consider that the *VG*, described by the Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA, 1997) and updated by Teixeira et al. (2017), is the most commonly applied method in laboratory routines, using alcohol (99 °GL) to determine the $\rho_p$.

Alcohol is used to determine $\rho_p$ due to its surface tension lower than water, penetrating the soil's capillary pores more easily and expelling the air content (Gubiani et al., 2006). However, the 99 °GL alcohol commonly used as a standard in laboratory analyzes can become a problem, since the cost is high when the number of analyzes to be performed is large, in addition to increase in the incidence of experimental error, due to evaporation. Santos and Rodrigues (2009) indicate that the loss of alcohol by evaporation is always present in the $\rho_p$ determination with the volumetric glassware method, constituting the main source of error. The losses of alcohol by evaporation mask the volume of the sample solids, decreasing its value and, consequently, increasing the sample particle density. For each 0.1 mL of alcohol added in the volume measurement, it implies an increase of 30 kg $m^{-3}$ in $\rho_p$.

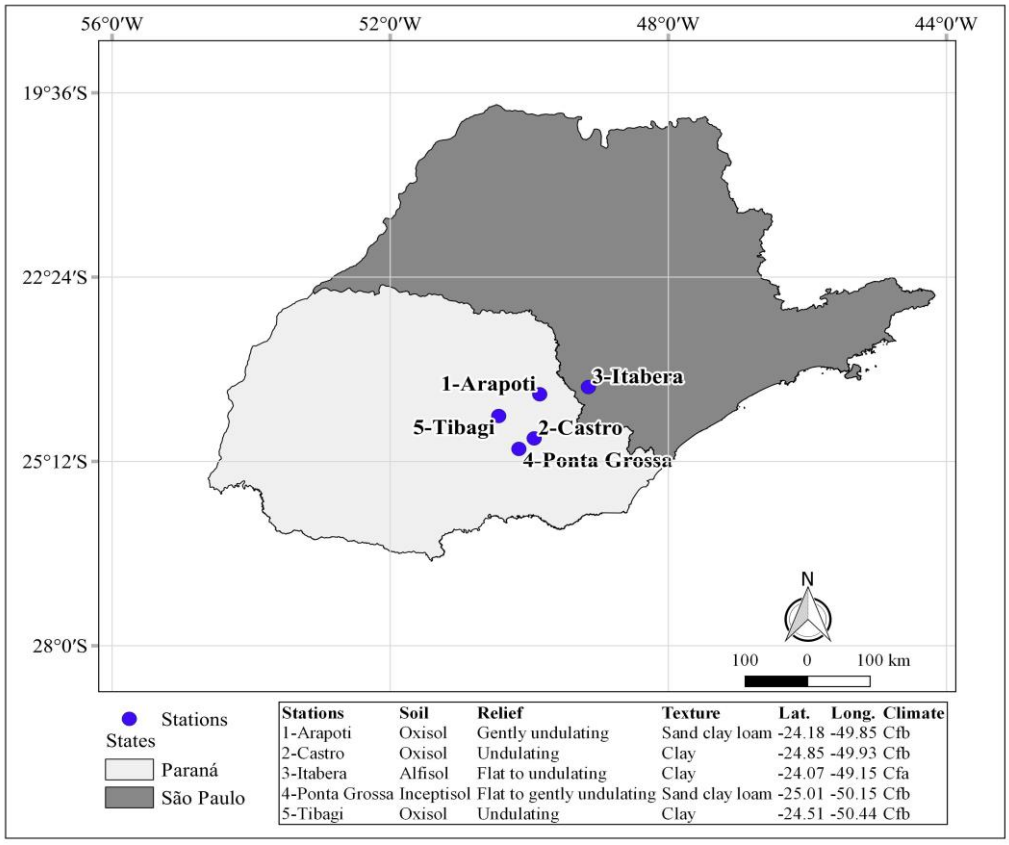
It becomes necessary to develop new methods and techniques for soil analysis, seeking to provide reliable results with reduced costs. Thus, analyzes aiming to determine $\rho_p$ with lower alcohol concentrations than that usually used in the laboratory (99 °GL) are of great use.

In this context, the objective this study was to evaluate the efficiency of alcohol in concentrations of 0.0, 47.6, 70.0 and 99 °GL, to determine the soil particle density ($\rho_p$) with the "Volumetric Glassware Method" (*VG*), checking the best analysis time and the possibility of alternatives to 99 °GL alcohol.

## Materials and methods

*Characterization of the study area*

The soil samples were collected in five experimental plots, containing 5000 m² each. The areas belong to the Agrometeorology sector of Fundação ABC, located in Arapoti, Castro, Ponta Grossa and Tibagi cities, in Paraná state, southern Brazil, and Itaberá, in São Paulo state, southeastern Brazil. Figure 1 shows the soil type, relief, texture, latitude, longitude and climate of the region, according to Alvares et al. (2013).




| Stations | Soil | Relief | Texture | Lat. | Long. | Climate |
|---|---|---|---|---|---|---|
| 1-Arapoti | Oxisol | Gently undulating | Sand clay loam | -24.18 | -49.85 | Cfb |
| 2-Castro | Oxisol | Undulating | Clay | -24.85 | -49.93 | Cfb |
| 3-Itabera | Alfisol | Flat to undulating | Clay | -24.07 | -49.15 | Cfa |
| 4-Ponta Grossa | Inceptisol | Flat to gently undulating | Sand clay loam | -25.01 | -50.15 | Cfb |
| 5-Tibagi | Oxisol | Undulating | Clay | -24.51 | -50.44 | Cfb |

**Figure 1**. Characterization of the Fundação ABC study area.

All experimental areas are managed in the no-tillage system in a long-term experiment, with crop rotation, being soybean and maize in summer crops and wheat and oats in winter crops.

The textural classification (Figure 2) was obtained with granulometric analysis using the densimeter method, according to Teixeira et al. (2017), and textural triangle with the "*Soiltexture*" package, Version 1.5.1 (Moeys et al., 2018), from RStudio *software*.

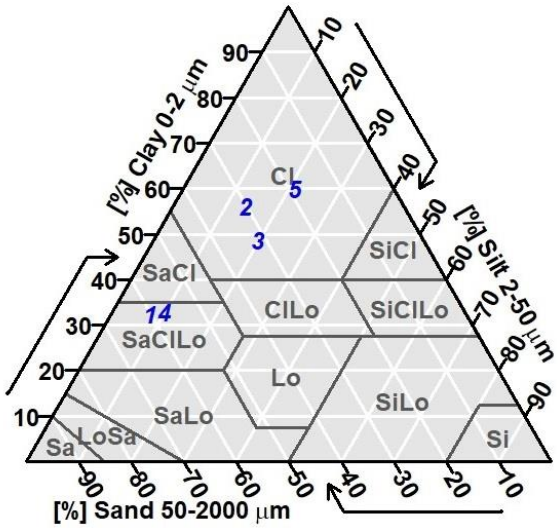



**Figure 2**. Soil granulometric distribution in the localities: 1) Arapoti; 2) Castro; 3) Itaberá; 4) Ponta Grossa; and 5) Tibagi, from the Fundação ABC Agrometeorology Sector.

*Sampling and sample preparation*

Sampling was carried out only in the topsoil (0.20 m), since the highest concentration of root hair, responsible for capturing water and nutrients, occurs in this range. Five samplings were made per area, collecting approximately 0.8 kg of soil at each sampling point.

The samples were stored in identified plastic bags and sent to the Soil Physics laboratory of the Federal University of Paraná. Subsequently, sample preparation procedures were carried out, including maceration, dried in 105 °C oven for a period of 24 h, and cooled in a desiccator.

*Procedures for $\rho_p$ analysis with the Volumetric Glassware Modified Method*

To determine the soil particle density ($\rho_p$) with the Volumetric Glassware Modified Method (*VG*), it was followed the Soil Analysis Methods manual according to Teixeira et al. (2017), which consists of the steps: *i*) Weighing volumetric glassware of 50 ml on a previously tared scale; *ii*) Transfer of approximately 20 g of soil from the sample already prepared to the volumetric glassware, noting the weight of the glassware + soil; and, *iii*) Adding of ethyl alcohol (99 °GL) up to half of glassware + soil volume, followed by manual agitation until the air between the soil particles is eliminated, subsequently completing the volume of the glassware until the measuring line (meniscus).

To verify the viability of reducing laboratory costs with alcohol, the same procedure with *VG* was repeated using water and alcohol at concentrations of 47.6 and 70 °GL. The process of analyzing the volumetric glassware occurred with the glassware weighing at the times: 0 (instantly), 24, 48 and 168 h after sample preparation. The analyses were performed considering five repetitions and five sample types for each alcohol concentration, totaling 100 samples.

For 70 °GL alcohol concentration, 729.16 mL of 96 °GL alcohol was diluted with 270.84 mL of deionized water. The values were obtained with the relation:

$$C_1 \cdot V_1 = C_2 \cdot V_2$$

Where: $C_1$– Initial concentration (°GL); $V_1$ – Initial volume (L); $C_2$– Final concentration (°GL); $V_2$ – Final volume (L).

The soil particle density was calculated with the expression:

$$\rho_p = \frac{M_s}{V_s}$$

The $\rho_p$ determination was performed using the Volumetric Glassware Method (Teixeira et al. 2017):

$$\rho_p = \frac{M_s}{(50 - V_s)}$$

Where: $\rho_p$ – Soil particle density (kg m$^{-3}$); $M_s$ – Weight of oven-dried soil (kg); $V_s$ – Volume of solid soil particles (m$^3$).

*Statistical analysis*

Associations between "$\rho_{p\ standard}$ *vs* $\rho_{p\ alternative}$" were verified, with linear regression analyzes between the values:

– Values of "$\rho_p$ obtained with *VG* and 99 °GL alcohol ($\rho_{pVG.99}$)" *vs* "$\rho_p$ obtained with *VG* with water ($\rho_{pVG.w}$) or alcohol at 47.6 °GL ($\rho_{pVG.47.6}$) or 70 °GL ($\rho_{pVG.70}$)":

"$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$"
"$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$"
"$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$"

The linear regression analyzes were performed with the "*ggplot2*" package, version 3.3.2 (Wickham et al., 2020), from RStudio *software*.

The verification of associations was performed considering indexes and errors recommended in the literature (Jacovides and Kontoyiannis, 1995; Gubiani, 2006), such as the trend of the $\rho_p$ values obtained, observing the mean and distribution in *Box Plot* diagram, average, maximum and minimum values data, coefficient of variation (*CV*) and determination ($R^2$), mean absolute error (*MAE*) and index of agreement "*d*", according to the following equations:

$$CV = \frac{\sigma}{\overline{Y}} \cdot 100$$

$$R^2 = \frac{\sum_{i=1}^{n}[(Y_{p_i} - \bar{Y}_p) \cdot (Y_{a_i} - \bar{Y}_a)]}{\sqrt{\sum_{i=1}^{n}(Y_{p_i} - \bar{Y}_p)^2 \cdot \sum_{i=1}^{n}(Y_{a_i} - \bar{Y}_a)^2}}$$

$$MAE = \frac{\sum_{i=1}^{n}\left|Y_{p_i} - Y_{a_i}\right|}{n}$$

$$d = 1 - \frac{\sum_{i=1}^{n}\left(Y_{a_i} - Y_{p_i}\right)^2}{\sqrt{\sum_{i=1}^{n}\left(\left|Y_{a_i} - \bar{Y}_p\right| \cdot \left|Y_{p_i} - \bar{Y}_p\right|\right)^2}}$$

Where: *CV* – coefficient of variation (%); $\sigma$ – standard deviation (kg m$^{-3}$); $\overline{Y}$ – average of the $\rho_p$ values obtained with the standard or alternative method (kg m$^{-3}$); *MAE* – mean absolute error (kg m$^{-3}$); $Y_{pi}$ – *i*-th value of soil particle density obtained with the Volumetric Glassware Modified Method using 99 °GL alcohol (kg m$^{-3}$); $Y_{ai}$ – *i*-th value of soil particle density obtained with alternative method (kg m$^{-3}$); *n* – number of repetitions; $R^2$ – coefficient of determination (unitless); $\overline{Y}_a$ – average of the $\rho_p$ values obtained with the alternative method (kg m$^{-3}$); $\overline{Y}_p$ – average of the $\rho_p$ values obtained with the standard method (kg m$^{-3}$); *d* – Willmott's index of agreement (Willmott, 1982) (unitless).

The indices and errors were performed with the "*hydroGOF*" package, version 0.04 (Zambrano-Bigiarini, 2020), from RStudio *software*.

## Results and discussion

*Soil particle density ($\rho_p$) with VG*

Linear regression analyzes between "$\rho_{p\ standard}$ *vs* $\rho_{p\ alternative}$" showed that "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at time zero, presented the best association. However, in the other hours analyzed, there were low associations, especially at hour 168 (Figure 3).

With exception of the time zero, the

"$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" ratio was the one with the best associations for all evaluated localities (Figure 3). The results stabilized after 24 h, with good results in the coefficient of determination values ($R^2 > 0.82$). The water provided the worst results, especially after 48 h of analysis ($R^2 < 0.42$), in which the highest *CV* values were observed (Tables 1 and 2).

**Table 1**. Real average of soil particle density ($\rho_p$; kg m$^{-3}$) and coefficient of variation (*CV*) obtained with the volumetric glassware method, using water ($\rho_{pVG.w}$), alcohol 47.6 ($\rho_{pVG.47.6}$), 70 ($\rho_{pVG.70}$) and 99 °GL ($\rho_{pVG.99}$), in 0, 24, 48 and 168 h periods.

| Localities | Hour | | | | *CV* |
|---|---|---|---|---|---|
| | 0 | 24 | 48 | 168 | % |
| | ---------- $\rho_{pVG.w}$ ---------- | | | | |
| Arapoti | 2359.11 | 2359.11 | 2565.26 | 2436.06 | 4.00 |
| Castro | 1893.56 | 1893.56 | 2447.99 | 2410.62 | 14.33 |
| Itaberá | 2349.03 | 2349.03 | 2687.56 | 2614.97 | 7.08 |
| Ponta Grossa | 2066.13 | 2066.13 | 2484.49 | 2724.78 | 13.96 |
| Tibagi | 2026.26 | 2026.26 | 2252.32 | 2336.41 | 7.34 |
| | ---------- $\rho_{pVG.47.6}$ ---------- | | | | |
| Arapoti | 2488.13 | 2566.94 | 2565.57 | 2426.06 | 2.70 |
| Castro | 2323.29 | 2442.97 | 2440.53 | 2467.94 | 2.68 |
| Itaberá | 2492.43 | 2572.60 | 2567.71 | 2584.40 | 1.64 |
| Ponta Grossa | 2371.46 | 2501.10 | 2497.22 | 2507.48 | 2.65 |
| Tibagi | 2376.90 | 2486.03 | 2477.35 | 2491.60 | 2.21 |
| | ---------- $\rho_{pVG.70}$ ---------- | | | | |
| Arapoti | 2420.62 | 2548.86 | 2536.70 | 2554.16 | 2.52 |
| Castro | 2234.55 | 2386.42 | 2388.27 | 2409.53 | 3.43 |
| Itaberá | 2490.99 | 2587.24 | 2577.13 | 2583.48 | 1.80 |
| Ponta Grossa | 2341.45 | 2506.43 | 2502.12 | 2515.22 | 3.38 |
| Tibagi | 2307.08 | 2505.37 | 2504.93 | 2518.55 | 4.13 |
| | ---------- $\rho_{pVG.99}$ ---------- | | | | |
| Arapoti | 2707.30 | 2508.61 | 2513.86 | 2554.61 | 3.62 |
| Castro | 2357.72 | 2409.53 | 2411.10 | 2433.84 | 1.34 |
| Itaberá | 2534.39 | 2598.98 | 2597.99 | 2602.59 | 1.27 |
| Ponta Grossa | 2470.41 | 2468.63 | 2495.44 | 2502.88 | 0.70 |
| Tibagi | 2438.29 | 2462.25 | 2483.50 | 2507.65 | 1.20 |

* CV – Coefficient of variation.

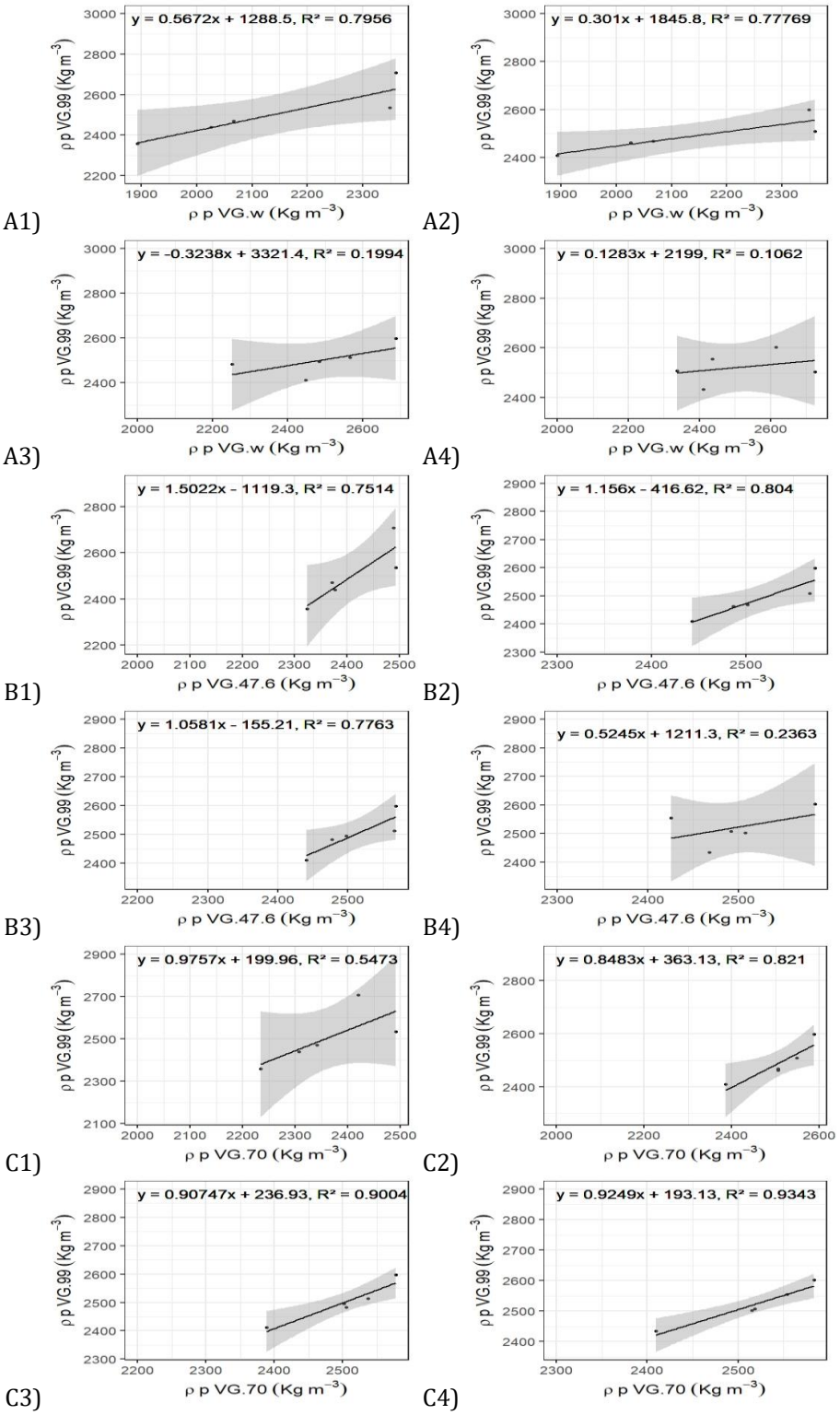



**Figure 3.** Linear regression analysis and respective coefficients of determination ($R^2$) at 0, 24, 48 e 168 h, considering the five analyzed areas and associations between "$\rho_{pVG.99}$ *vs* $\rho_{p\ \text{alternative methods}}$", being: A1) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at time 0; A2) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at 24 h; A3) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at 48 h; A4) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at 168 h; B1) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" at time 0; B2) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" at 24 h; B3) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" at 48 h; B4) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" at 168 h; C1) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" at time 0; C2) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" at 24 h; C3) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" at 48 h; C3) "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" at 168 h.

**Table 2**. Mean absolute error (*MAE*; kg m$^{-3}$) and index of agreement ("*d*"; unitless) obtained in the associations between "$\rho_{pVG.99}$ *vs* $\rho_{p\ \text{alternative methods}}$" at 0, 24, 48 and 168 h, considering the five areas analyzed.

| Association | Hour | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 0 | | 24 | | 48 | | 168 | |
| | *MAE* | *d* | *MAE* | *d* | *MAE* | *d* | *MAE* | *d* |
| "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" | 360.00 | 0.54 | 350.00 | 0.50 | 80.00 | 0.63 | 110.00 | 0.43 |
| "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" | 90.00 | 0.70 | 30.00 | 0.89 | 20.00 | 0.92 | 40.0 | 0.71 |
| "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" | 140.00 | 0.57 | 30.00 | 0.93 | 20.00 | 0.97 | 10.00 | 0.98 |

Linear regression analyzes between "$\rho_{p\ standard}$ *vs* $\rho_{p\ alternative}$" showed that "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" at time zero, present the best association. However, in the other hours analyzed, there were low associations, especially at 168 h (Figure 3).

In general, the lowest *CV* values between "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" were very similar (Table 1), a fact that is consistent with the results obtained in the linear regression, in which a good association was observed until the period of 48 h, with an evident reduction in the association afterwards (Figure 3). It was also found that the $\rho_{pVG.99}$ showed higher stability (lower *CV* values), making it possible to perform the analysis in 24 h, as well as the $\rho_{pVG.70}$. The $\rho_{pVG.47.6}$ was promising to replace $\rho_{pVG.99}$, as it showed a high reduction in the coefficient of determination after 48 h. Table 2 shows higher errors with the associations "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" for all analyzed hours, and a better association for "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$", proving that the $\rho_{pVG.70}$ is promising to replace $\rho_{pVG.99}$.

Considering the 70°GL alcohol concentrations, which were better associated with 99 °GL alcohol, an average $\rho_p$ value (Table 1) was obtained at 168 h (with higher possibility of results stability), with values of 2554.16 kg m$^{-3}$ in Arapoti, 2409.53 kg m$^{-3}$ in Castro, 2583.48 kg m$^{-3}$ in Itaberá, 2515.22 kg m$^{-3}$ in Ponta Grossa, and 2518.55 kg m$^{-3}$ in Tibagi. The $\rho_p$ values obtained in the present study were very close to those obtained by Souza et al. (2017), in a study analyzing the soil physical-water attributes for the same region and localities (with $\rho_p$ ranging between 2490.00 and 2630.00 kg m$^{-3}$). Libardi (2005) and Santana (2009) comment that $\rho_p$ has little variation between soil types, ranging from 2030.00 to 2900.00 kg m$^{-3}$. In case of mineral soils, the $\rho_p$ value is usually approximately 2650 kg m$^{-3}$ (Ruehlmann, 2020), showing the quality of the results obtained. However, the values obtained in the present study were not very close to those obtained by Rosa et al. (2018), studying the soil physical-water attributes in Campos Gerais. Considering the average between 0.0 to 0.10m and 0.10 to 0.25m depths, the referred authors obtained values of $\rho_p$ = 2789.59 kg m$^{-3}$ in Arapoti, $\rho_p$ = 2810.29 kg m$^{-3}$ in Itaberá and $\rho_p$ = 2740.55 kg m$^{-3}$ in Ponta Grossa. It is believed that the differences in values were due to the variability of the experimental areas used, or to the particularities of the methodological procedures of each study.

*Trends in $\rho_p$ values obtained with VG using alcohol at 99 °GL and alternative concentrations*

The results of $\rho_{pVG.99}$, for readings at 0, 24, 48 and 168 h, showed higher amplitudes in Arapoti and averages very close for the other localities (Figure 4). However, the measurements at the instantly time (zero hour) always indicated $\rho_p$ values below the averages performed in the other analyzed periods.

The 99 °GL alcohol shown to be efficient, performing good $\rho_s$ measurements after 24 h. Air bubbles were eliminated and the variability between samples was low. However, the instantly $\rho_s$ measurement should be avoided whenever possible, since the results indicate that air bubbles can resist for some time.

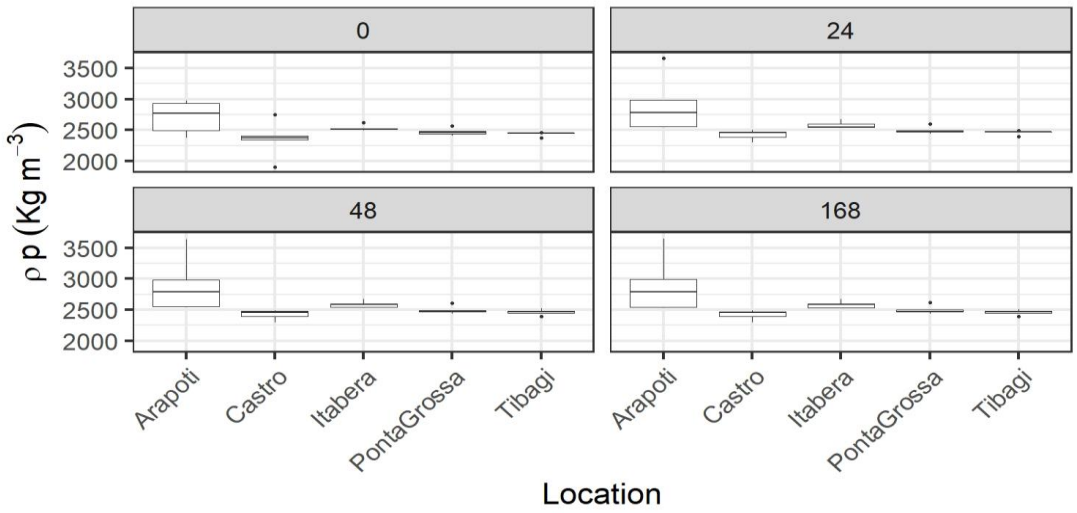



**Figure 4**. *Box Plot* of soil particle density ($\rho_p$; kg m$^{-3}$), obtained with the volumetric glassware method, using 99 °GL alcohol ($\rho_{pVG.99}$), at the instantly time (zero hour), 24, 48 and 168 h, in Arapoti, Castro, Itaberá, Ponta Grossa and Tibagi localities.

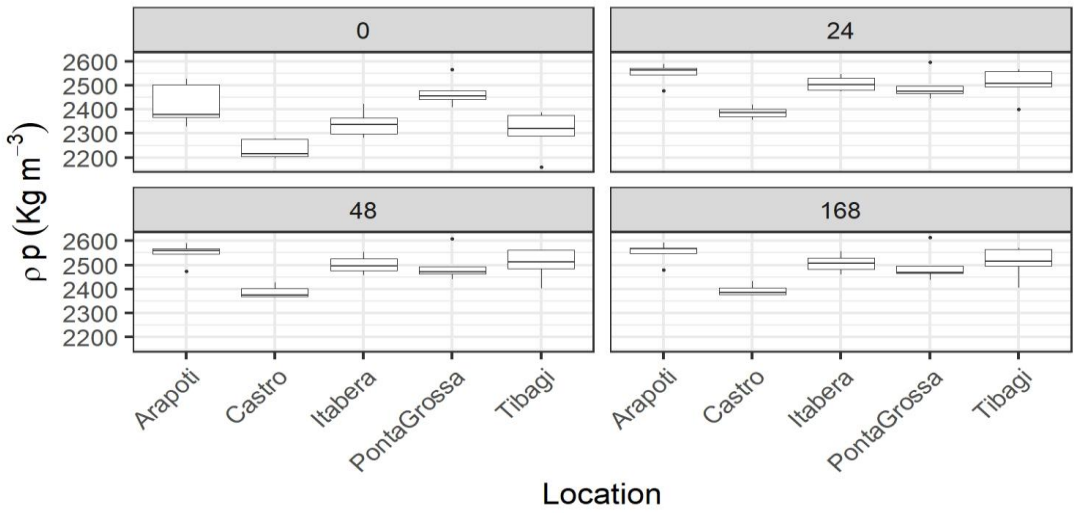



**Figure 5.** *Box Plot* of soil particle density ($\rho_p$; kg m$^{-3}$), obtained with the volumetric glassware method, using 70 °GL alcohol ($\rho_{pVG.70}$), at the instantly time (zero hour), 24, 48 and 168 h, in Arapoti, Castro, Itaberá, Ponta Grossa and Tibagi localities.

The $\rho_{pVG.70}$ results had a similar trend to those obtained with alcohol at 99 °GL (Figure 4), referring to the analysis periods. The instantly time was even more limited. However, it was found that the magnitudes of the

$\rho_{pVG.70}$ and $\rho_{pVG.99}$ values were different (Figure 5), with underestimation in practically all $\rho_{pVG.70}$ values, with mean absolute error between $\rho_{pVG.99}$ and $\rho_{pVG.70}$ readings from 140.00 kg $m^{-3}$ at zero hour to 10.00 kg $m^{-3}$ at 168 h (Table 2).

In general, the results obtained indicated that the 99 °GL alcohol provided less air bubble formation than the 70 °GL alcohol, probably due to the lower surface tension, being possible to obtain results in 24 h, while 70 °GL alcohol requires a minimum time period of 48 h for no bubbles occur.

The results of $\rho_{pVG.47.6}$ were stable for the periods at 24, 48 and 168 h. The instantly time (zero hour) also showed higher variability, underestimating the $\rho_p$ values when compared to the $\rho_{pVG.99}$ values (Figure 4 and 6).

The association of $\rho_p$ results was better for the "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$" than "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" analyzes (Figure 3). The reason was probably due to the following aspects: *i*) The soil samples used to perform the analyzes were not the same, which may have led to variations due to natural changes in their physical structure; and, *ii*) There were large variations in temperature between the analyzed days, in addition to the samples not being processed in the same period, which may have contributed to the difference in the results. The average absolute error between "$\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$" was higher than the readings in 70 °GL alcohol (Table 2), providing a smaller association between $\rho_{pVG.99}$ *vs* $\rho_{pVG.47.6}$ than que $\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$.

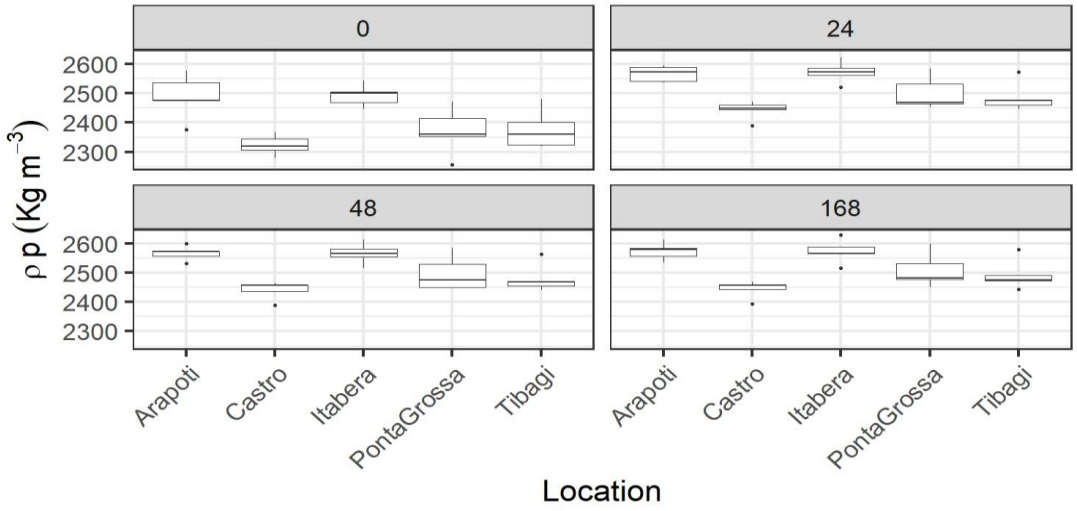



**Figure 6.** *Box Plot* of soil particle density ($\rho_p$; kg $m^{-3}$), obtained with the volumetric glassware method, using 47.6 °GL alcohol ($\rho_{pVG.47.6}$), at the instantly time (zero hour), 24 , 48 and 168 h, in Arapoti, Castro, Itaberá, Ponta Grossa and Tibagi localities.

The $\rho_p$ determination with *VG* method using only water showed interesting results in the trends of the samples, for the five evaluated localities. The instantly time (zero hour) and 24 h periods indicated the existence of sufficient air in the glassware in all samples. The analysis times did not show stability of the results, and there is variability between samples from the same place. This last aspect was evident in the *Box Plot* graphs, when is observed that the amplitude of the quartiles in relation to the mean represented by the

square, differs a lot as to the $\rho_{pVG.99}$ values (Figure 4 and 7).

The samples in Arapoti, Ponta Grossa and Tibagi, even after 168 h, still had bubbles, showing that the time of analysis with water needs an even longer period (Figure 7). This fact may be due to the strong interaction of the bubbles with soil particles, which cannot be broken using only water as a solvent, or due to the water surface tension be higher than the alcohol (Santos et al., 2010), making it difficult to the water to penetrate the soil pores.

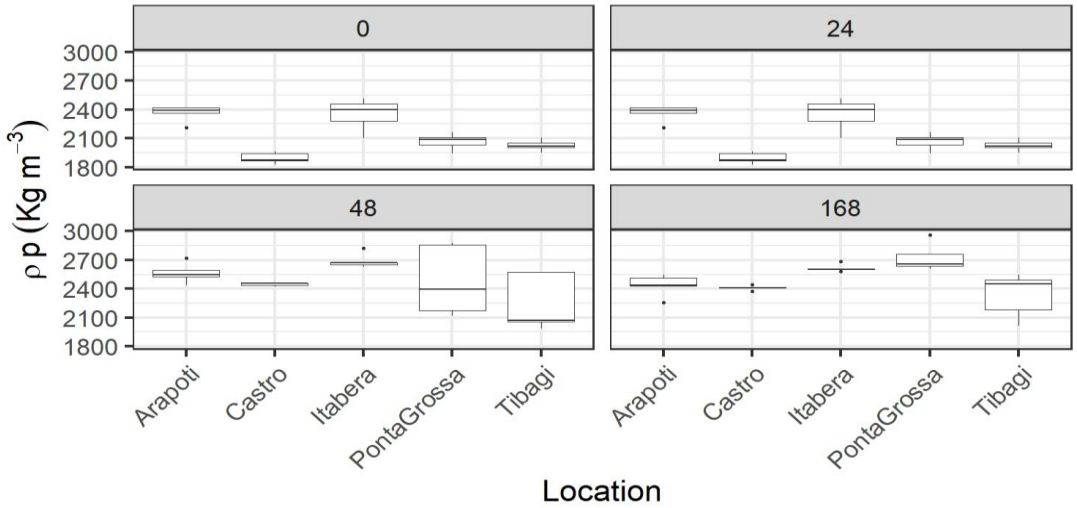



**Figure 7.** *Box Plot* of soil particle density ($\rho_p$; kg m$^{-3}$), obtained with the volumetric glassware method, using water ($\rho_{pVG.w}$), at the instantly time (zero hour), 24 , 48 and 168 h, in Arapoti, Castro, Itaberá, Ponta Grossa and Tibagi localities.

The analysis in Arapoti showed inconsistency in the results, since the soil particle density decreased in 168 h (Figure 7). The result characterized the difficulty in working with water at *VG* method. Some analyzes showed layers with particles suspended in the solution, characteristic of organic matter (Figure 8). In some cases the layer formed was so strongly adhered to the volumetric glassware walls that it was difficult to complete the water up to 50 ml height. Thus, when applying the complementary water drops on top of the formed layer, it was necessary to wait for their absorption. The layers occurred in different ways, some more adhering to the glassware walls, a fact that occurred in Arapoti samples, and others less adhering, as occurred in Itaberá. These aspects contributed to obtain high errors and low associations between "$\rho_{pVG.99}$ *vs* $\rho_{pVG.w}$" (Table 2 and Figure 3).

A)  B) 

**Figure 8.** Volumetric Glassware Method: occurrence of a layer formed by particles suspended, due to the presence of organic matter.

The water presented serious limitations to be used in the analysis of $\rho_p$ determination with the *VG* method, showing inefficiency in removing air in soil samples with clay texture, possibly due to its higher surface tension, as reported by Santos et al. (2010). Thus, the results obtained make its use in laboratory routines unfeasible to replace 99 °GL alcohol. In another way, the 70 °GL alcohol showed good results, indicating the possibility of sparing alcohol in laboratory analyzes, by reducing the concentration of 99 °GL alcohol. However, it is necessary to increase the analysis time (minimum of 48 h for 70 °GL alcohol) so that the results can be stable. The 47.6 °GL alcohol showed instability in the results, making its use unfeasible, despite the good results previously verified at 48 h.

## Conclusions

The association "$\rho_{pVG.99}$ *vs* $\rho_{pVG.70}$", after 24 h, showed an excellent result ($R^2 > 0.82$). The use of water in the analysis was unsatisfactory, especially after 48 h ($R^2 < 0.42$). The 47.6 °GL alcohol concentration was not promising to replace 99 °GL concentration, due to the high reduction on the coefficient of determination and correlation after 48 h;

The 99 °GL alcohol allows less variable $\rho_p$ analyzes after 24 h. The 70 °GL alcohol provides a better response only after 48 h;

For the analyzed soils, alcohol at a 70 °GL concentration can replace the 99 °GL to obtain $\rho_p$ values with the Modified Volumetric Glassware Method, in the period after 48 h. The 47.6 °GL alcohol concentration or water is not recommended to obtain $\rho_p$ values with *VG* modified method.

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