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ANALYTICAL METHODS COMPARISON FOR pH DETERMINATION OF COMPOSTING PROCESS FROM GREEN WASTES

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Abstract

Composting is the accelerated microbiological decomposition of organic waste. pH, temperature, and moisture are necessary to be monitored to ensure an adequate control of this process. pH can be a fundamental indicator of the degree of degradation, however, there are few specific analytical methods of its determination in samples originated from composting and, therefore, data can be difficult to interpret when consulting the literature. With the aim of evaluate extractant solutions (CaCl_2 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹) and the universal solvent (water) used for pH determination, a comparison of four analytical methods was performed in samples originated from composting of leaf and garden waste. A descriptive statistical analysis, normality tests (Shapiro-Wilk), comparison of medians (Kruskal-Wallis with post-hoc by Nemenyi) and linear regressions with robust variance were performed (software R 3.4.2). Statistical analysis showed significant differences, suggesting CaCl_2 and H_2O methods tend to be more indicated when is desired to apply the final compound directly on the soil. KCl tends to be the solution with the greatest extraction capacity of H⁺ ions. Therefore, the waste characteristics and the purpose of the monitoring parameters must be evaluated for the determination of the appropriated pH methodology.

Keywords: green waste, leaf and garden waste, microbiological degradation, organic waste, pH analytical methodologies

Received: March, 2020; Revised final: June, 2020; Accepted: September, 2020; Published in final edited form: January, 2021

1. Introduction

Composting is an accelerated decomposition and stabilization controlled process of organic wastes, resulting in a stable final product, called humus. Although, it may be only considered as a slow release fertilizer, the humus high C:N ratio can act to reduce the initially available N, possibly making its application to the soil beneficial thought the incorporation of nutrients (Haug, 2018; Mello-Peixoto et al., 2014; Pereira, 2007; Strom and Finstein, 2000). The development of decomposing organisms depends on the environment factors provided, especially by temperature range and the compound hydrogen potential (pH) between 40 and 70°C and 5.5 and 8.5, respectively. Values outside this range may negatively

interfere in the process (Haug, 2018; Pereira, 2007; Rodrigues et al., 2006; Vandecasteele et al., 2016; Xie et al., 2017). In general, the pH tends to increase gradually as the degradation process intensifies, being also an important parameter of the chemical properties of the composted material (Kiehl, 2004; Pereira, 2007; Strom and Finstein, 2000; Tedesco et al., 1995).

The phenomenon of pH self-regulation, performed by microorganisms in the composting process, was observed by Pereira (2007). However, there is no guarantee that the pH will be kept in the optimal range throughout the process, reinforcing the importance of monitoring this parameter. pH monitoring is also a fundamental indicator of the degree of compound degradation (Cerri et al., 2008; Jiménez and García, 1991; Kiehl, 1985; 2004).

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According to Kiehl (1985), the compost pile may have acidic pH values in the first days of composting due to the formation of inorganic acids that are later replaced, due to microbiological activity, by organic acids. Sesay et al. (1997) also highlighted the importance of pH monitoring as an indicator of biomass stabilization, noting its increase during the composting process and values close to neutrality at the end of the experiment.

In Brazil, and other many countries, there is a lack of specific analytical methods for the determination of compost pH. Often it is used established soil pH procedures for compost pH monitoring (Albuquerque et al., 2006; Cotta et al., 2015). In the UK the BSI, PAS100 (2011) standard, specifies the usage of the BS EN 13037 method (using water) which is also a European standard for pH compost analysis. Usually for the determination of pH it is recommended, among others, the use of water solvent or soluble salt extraction solutions, such as CaCl_2 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹, the use of soluble salts is convenient to minimize seasonal variation in soil pH (Burt et al., 2014).

The seasonal variation may influence the pH value of soils as the natural presence of soluble salts, accumulated during dry periods, such as sulphates and nitrates, reduces the pH value. On the other hand, in rainy periods, these salts are susceptible to leaching, causing the pH to return to its maximum value (Peech, 1965). The pH values obtained when using dissolved salt solutions are usually lower compared to purified water and may be similar or even higher in soils subjected to high rainfall, due to the large anion exchange capacity (Foth and Ellis, 1988). In addition, the use of these extraction solutions is based on empirical studies, hence, there is no scientific support

to choose one over the other (CEN/BT/Task Force 151, 2005).

Thus, the objective of the present work was to compare analytical methods for pH determination using purified water (type I) and extraction solutions CaCl_2 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹ as solvent in samples from the composting process of leaves and yard waste.

2. Methodology

2.1. Sample preparation and analytical methodologies

The samples were taken from a compost pile made with leaves and yard waste in a public institution in Belo Horizonte (Minas Gerais). The composting process was carried out between June and October 2017. Starting in the 67th day of composting, four samples from the center (about 500 grams) at different experimental days (67th, 68th, 84th and 85th) were collected. The samples were immediately packed, in plastic bags, and sent to the Ezequiel Dias Foundation Bromatological Chemistry Laboratory (FUNED) for pH determination.

For pH analytical assays, the methodologies indicated in Tedesco et al. (1995) and in the Soil Analysis Methods Manual of the Brazilian Agricultural Research Corporation (EMBRAPA, 1997) were used, in order to compare water type I (ultrapure), CaCl_2 0.01 mol L⁻¹ and KCl 1 mol L⁻¹. Thus, the analytical methods considered were: Tedesco et al. (1995) with CaCl_2 solution 0.01 mol L⁻¹ (TCaCl₂); EMBRAPA (1997) with type I water (EH₂O); EMBRAPA (1997) with CaCl_2 0.01 mol L⁻¹ (ECaCl₂); and EMBRAPA (1997) using KCl 1 mol L⁻¹ (EKCl).

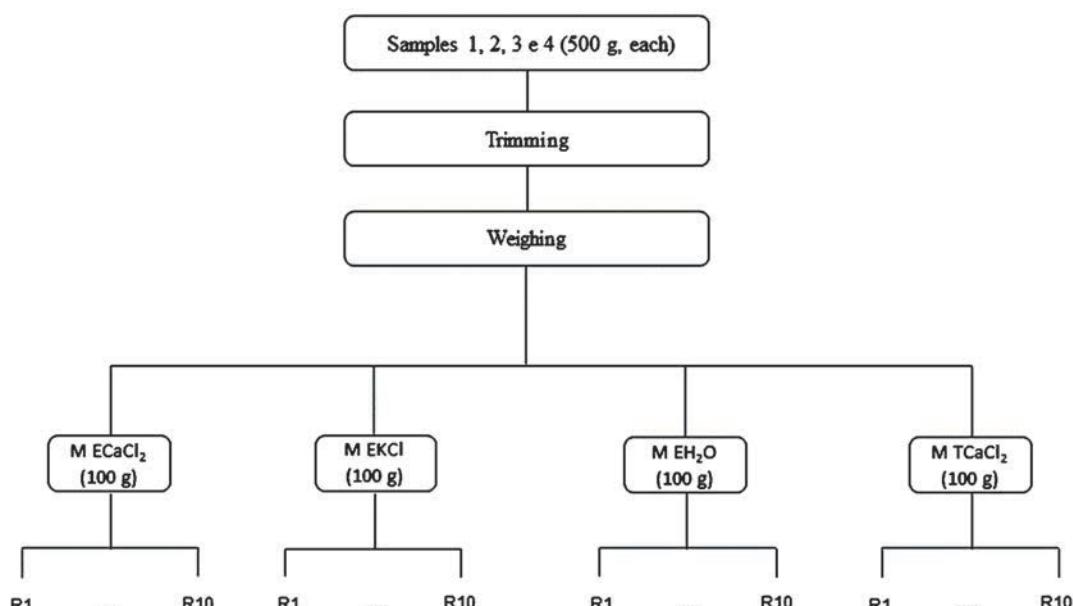


Fig. 1. Flowchart of sample preparation and experimental design for comparison of pH determination methods in compost residue [M = analytical method (ECaCl₂, EKCl, EH₂O and TCaCl₂); R = replicate (10 g each)]

The analytical assays were performed during August 2017, at four different periods and in ten independent replicates for each method. Initially, the entire collected sample was ground in a household multiprocessor for exclusive use. Samples were then weighed and distributed equally in 100 mL beakers. About 10 grams of compost was inserted to each beaker corresponding to each analytical method and to ten replicates (Fig. 1).

According to the method indicated by Tedesco et al. (1995), 50 ml of CaCl_2 0.01 mol.L⁻¹ were added to each replicate followed by intermittently shaking it for 30 minutes on a mechanical stirrer (IKA Labortechnik KS-501 Digital) at 140 revolutions per minute (rpm). While, EMBRAPA (1997) indicates three different methods where type I water, CaCl_2 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹, 50 ml of each solvent or solution were added to their respective replicates, homogenizing them with an individual glass rod and keeping them at rest for one hour.

Replicates of each method were filtered through a glass funnel containing cotton. The pH was measured in the resulting solutions using the pH meter (Digimed DM-20, precision ± 0.01 , calibrated in the measurement range 4.01 to 10.03 pH units).

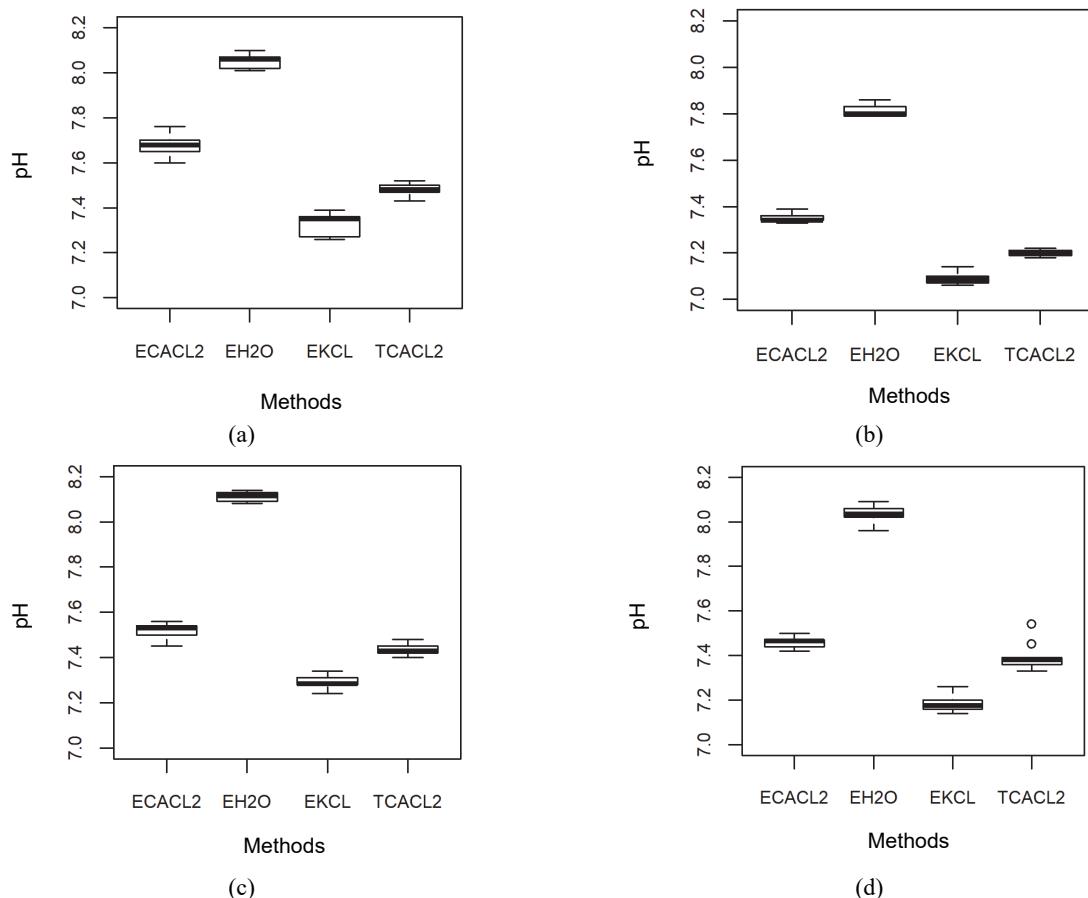


Fig. 2. Results of the descriptive analysis in relation to the analytical methods compared in the present study for each sample:

(a) Sample 1; (b) Sample 2; (c) Sample 3; (d) Sample 4 [Legend: EH₂O = EMBRAPA method (1997) using type I water; ECaCl₂ = EMBRAPA method (1997) using 0.01 M CaCl₂; EKCl = EMBRAPA method (1997) using 1 N KCl; TCaCl₂ = method Tedesco et al. (1995) using 0.01 M CaCl₂; n = sample number; SD = standard deviation; LL = lower limit calculated via bootstrap; UL = upper limit calculated via bootstrap; Min = minimum value; Max = maximum value; 1Q = first quartile; 2Q = second quartile (median); 3Q = third quartile]

2.2. Statistical analysis

We performed a descriptive statistical analysis that included number of samples, arithmetic mean, standard deviation, lower and upper limits calculated via bootstrap, variation coefficient, minimum value, first quartile, median, third quartile and maximum value (Ribeiro Junior, 2011).

The Shapiro-Wilk normality tests was used, the variance of homogeneity by Bartlett's test and, subsequently, the nonparametric Kruskal-Wallis median comparison test with post hoc by Nemenyi (Itano and Santos, 2006; Pohlert, 2018). The significance level adopted for all statistical analyses was 5% ($\alpha = 0.05$). Linear regressions were also calculated for each method, adopting linear regression with robust variance to assure the validity of the models (Itano and Santos, 2006). Software R (version 3.4.2) was used.

As the objective of the present study was to compare the four selected methodologies, it is noteworthy that the four samples collected at different periods were used and, therefore, the comparisons of results were performed only between the methods for each sample.

Table 1. Results of the comparison test of median pH values per sample

<i>Sample</i>	<i>Method</i>	<i>ECACL₂</i>	<i>EH₂O</i>	<i>EKCL</i>
1*	EH ₂ O	0.223	-	-
	EKCL	0.001	<0.001	-
	TCACL ₂	0.223	0.001	0.223
2*	EH ₂ O	0.223	-	-
	EKCL	0.001	<0.001	-
	TCACL ₂	0.223	0.001	0.223
3*	EH ₂ O	0.188	-	-
	EKCL	0.001	<0.001	-
	TCACL ₂	0.303	0.001	0.188
4*	EH ₂ O	0.129	-	-
	EKCL	0.002	<0.001	-
	TCACL ₂	0.514	0.002	0.129

Legend: * Kruskal-Wallis multiple comparison test with post hoc by Nemenyi

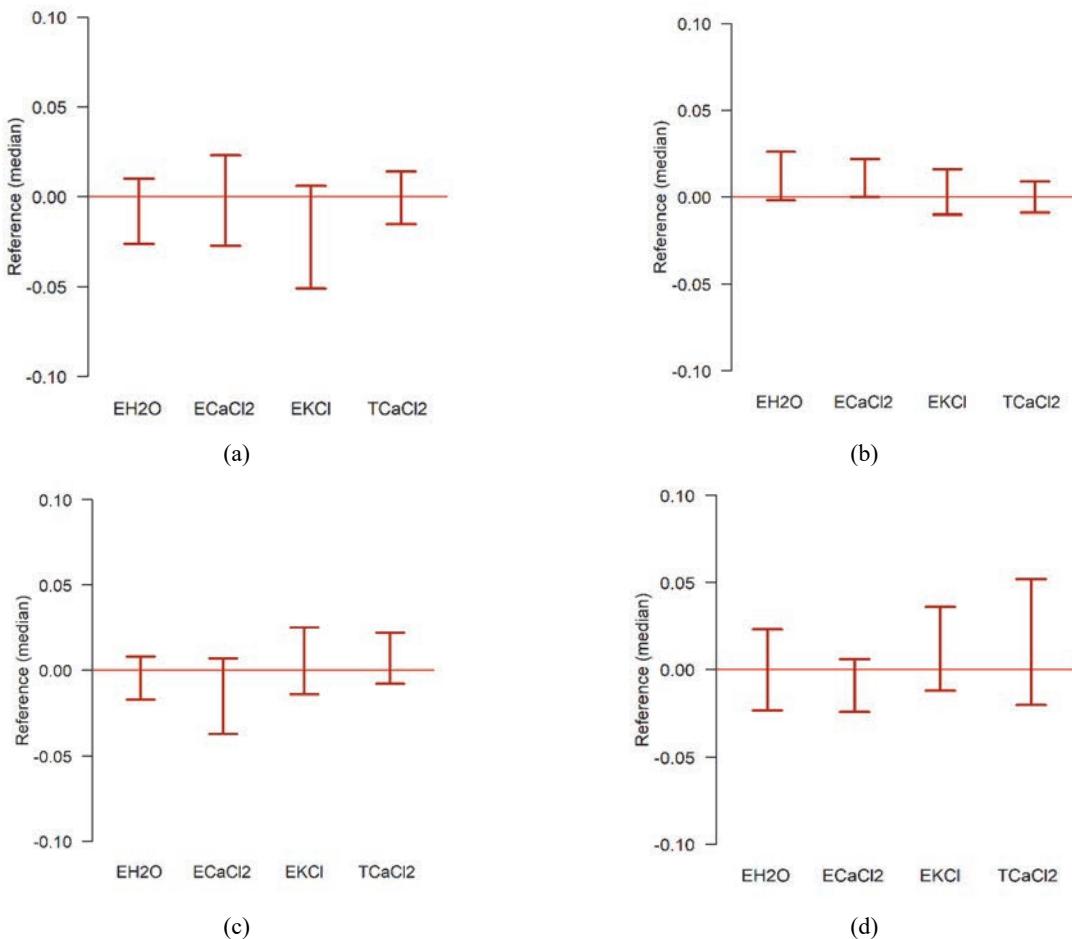


Fig. 3. Variation of standard pH values as a function of the median of each method for each sample:
 (a) Sample 1; (b) Sample 2; (c) Sample 3; (d) Sample 4

3. Results

Fig. 2 shows the results of the descriptive analysis considering the four methodologies for each sample. We found that the amplitudes obtained ranged between: 7.79 and 8.14 (EH₂O for samples 2 and 3, respectively); 7.33 and 7.76 (ECaCl₂ for samples 2 and 1, respectively); 7.06 and 7.39 (EKCl for samples 2 and 1, respectively) and 7.18 and 7.54 (TCaCl₂ for samples 2 and 4, respectively), thus being the largest and smallest amplitudes observed in the TCaCl₂

method (sample 4) and TCaCl₂ (sample 2), respectively.

Normality test indicated that the sample data are nonparametric, with all p-values less than 0.001 (Shapiro-Wilk test). The homogeneity tests of variances, performed by Bartlett's test, showed the existence of homogeneity, obtaining the values 0.1894, 0.4836, 0.4069 and 0.0898 for samples 1, 2, 3 and 4, respectively.

Median comparisons were made through Kruskal-Wallis test with post-hoc by Nemenyi (Table

1). We observed that the EH₂O methodology were significantly different ($p < 0.05$) from the others, except ECaCl₂. The ECaCl₂ also showed statistically significant differences ($p < 0.05$) when compared to the EKCl (Table 1).

Regarding the variation analysis of the standardized pH values around their own median, for each method and sample. There was no variability pattern, since different methods presented different amplitudes for each sample, and it was not possible to identify one that stood out (Fig. 3). The variations presented in Fig. 3 can be explained by the uncertainties of the methodologies.

Table 3 describes the results obtained by multiple regressions for each sample and methodology. We obtained a coefficient of determination (R^2) above 0.98 for linear regressions without robust variance in all analyzed samples, indicating an adequate adjustment of the statistical model used. The standard error of linear regression with robust variance, which best fit the model, was presented. It was found that regression performed for each method was statistically significant ($p < 0.05$) (Table 3). This regression analysis was done according to Miller and Kissel (2010).

We also observed that the results obtained using EH₂O presented higher values among all samples. It is noteworthy that, in all samples, the methods that presented the largest and smallest variation of results in relation to the intercept were EKCl and ECaCl₂, respectively (Table 3).

4. Discussion

The pH values from the different extraction methods in the present study showed adequate results in relation to the range suggested by Rodrigues et al. (2006) and Pereira (2007). According to the authors,

in composting processes, although the responsible microorganisms for degradation act in wide pH ranges, the recommended range for the development of this process is around 5.5 to 8.5, since most of the enzymes present in microorganisms are active in this pH range.

The largest variation of median values was verified between the EH₂O and EKCl for all the considered samples. This discrepancy of values was also identified in the statistical analysis, once, there is a significant difference in these same methods (Kruskal-Wallis posthoc test by Nemenyi, $p < 0.05$), as well as the results obtained by multiple linear regression.

The present work pointed divergences, mainly regarding the use of water as a pH extraction solution, presenting higher values when compared to the other methods. However, it was not possible to verify the best suitability of only one method, being necessary the evaluation, above all, of the material characteristics to determine the pH and the purpose of the referred analytical test.

Regarding pH determination in samples from the composting process, Melo et al. (2008) also used 0.01 mol.L⁻¹ H₂O and CaCl₂, it was found that the pH values in CaCl₂ were about 0.1 to 1.1 units lower than those measured in H₂O, obtaining variations similar to those observed in the present study.

For pH determination, the use of water as a solvent generally had a higher pH value than salt solutions such as CaCl₂ 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹. In addition, the proportion of water to be used in this analytical test should be observed, since the soil/water ratio is highly susceptible to variations, increasing the pH value proportionally to the sample dilution (CEN/BT/Task Force 151, 2005; Foth and Ellis, 1988; Hendershot and Lalande, 2010; Schierup and Jesen, 1979).

Table 3. Results of the multiple linear regressions performed for each sample, considering the EH₂O method as reference (intercept)

Sample	Method	Variation	Standard Error*	P Value	R ²
1	Intercept (EH ₂ O)	8.052	0.009143304	<0.001	0.981
	ECACL ₂	-0.374	0.015912259	<0.001	
	EKCL	-0.724	0.017355115	<0.001	
	TCACL ₂	-0.572	0.011815244	<0.001	
2	Intercept (EH ₂ O)	7.811	0.007273239	<0.001	0.995
	ECACL ₂	-0.461	0.009104944	<0.001	
	EKCL	-0.724	0.010049876	<0.001	
	TCACL ₂	-0.611	0.00853815	<0.001	
3	Intercept (EH ₂ O)	8.111	0.006700746	<0.001	0.991
	ECACL ₂	-0.595	0.013088163	<0.001	
	EKCL	-0.821	0.011954079	<0.001	
	TCACL ₂	-0.675	0.01006479	<0.001	
4	Intercept (EH ₂ O)	8.036	0.011679041	<0.001	0.982
	ECACL ₂	-0.58	0.013957077	<0.001	
	EKCL	-0.85	0.016994117	<0.001	
	TCACL ₂	-0.645	0.021892921	<0.001	

Legend: R² - adjusted value for linear regression without robust variance; * - standard error calculated for linear regression with robust variance.

According to Krishnamurthy (2005), a suitable resolution for the variation of pH values, due to the soil/water ratio, may be the usage of solutions such as CaCl₂ 0.01 mol.L⁻¹ and KCl 1 mol.L⁻¹ instead of water.

The 0.01 mol.L⁻¹ CaCl₂ solution is the most used in the determination of pH in soil samples, as it has advantages such as the noninterference of the pH value by the sample/solution ratio and the little influence of recent soil historic of fertilizers (Peech, 1965; Davey and Conyers, 1988). This solution still has an ionic strength of 0.03 mol.L⁻¹, which is close to the expected value in the soil solutions. Thus, its use is recommended for the determination of pH in soil samples, among other organic materials (CEN/BT/Task Force 151, 2005).

Furthermore, the use of CaCl₂ 0.01 mol.L⁻¹ in analytical pH assays in soils reduced its value by about 0.5 units compared to pH measured using water as solvent (Courchesne et al., 1995; Schofield and Taylor, 1955). A similar difference between the pH of the sample in water and CaCl₂ 0.01 mol.L⁻¹ solution was also observed in the present study, ranging from 0.37 to 0.68 units (Table 3).

The KCl 1 mol.L⁻¹ solution for pH determination is widely used in acid soils, however there are no advantages to its routine use in pH analytical tests in place of 0.01 mol.L⁻¹ CaCl₂ (Burt et al. et al., 2014). The 1 mol.L⁻¹ KCl solution has high ionic strength (1.0 mol.L⁻¹) and is not as suitable as CaCl₂ for pH measurements in soil samples (CEN/BT/Task Force 151, 2005).

It is also noteworthy that the pH determination using saline solutions results in a reduction of the cations concentration around the soil particles, and some H⁺ ions to be transferred to the solution, which approximates the pH value in the solution to the real soil and/or organic matter conditions (Burt et al., 2014; Schierup and Jesen, 1979).

However, when it is desired to apply the obtained compost to low electrical conductivity soils, water use may be an appropriate option, as pH measurement of soils in water represents the closest pH value of the field solution (Hendershot and Lalande, 2010).

Still considering the observed variations of results, small coefficients of variation were found among the results, and it is not possible to define the most appropriate method. It is also noteworthy that Annex III of Normative Instruction No. 25 of July 23, 2009 for the characterization of mixed and compound organic fertilizers (MAPA, 2009) recommends pH values to be above 6.0. Thus, it is suggested that each study should consider its specificities to determine the most appropriate extraction solution for each situation.

5. Conclusions

Results suggests the CaCl₂ 0.01 mol.L⁻¹ solution and the universal H₂O solvent as more appropriate when it is desired to apply the final compound directly to soil as it depicts the closest

conditions to the field conditions. It was also possible to infer that KCl 1 mol.L⁻¹ tends to be the solution with the highest extraction capacity of H⁺ ions.

We emphasize the importance to define specific methods for the determination of pH in compost samples, given the absence of specific methodologies and the numerous possibilities of combinations of organic material to be used. It is also recommended that the studies clearly indicate the analytical methods considered, as well as the justification of choice. This could avoid the results comparisons between methodologies that used different extractor solutions and, consequently, the finding of mistaken conclusions. This can provide further discussions about composting and allow better process control.

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