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Importance of Composing Representative Samples According to the Theory of Sampling (TOS) for the Reuse of Water Treatment Sludge

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Abstract. In Brazil, water treatment sludge (WTS) production reaches 1.9 million m^3/day[2], mostly discharged in rivers, and minorly destined to landfills or sludge treatment plants. WTS reuse as a geotechnical material, a sustainable alternative to reduce environmental impacts, demands knowledge about WTS behavior. However, WTS characteristics vary for each WTP and along time for the same WTP, due to local geology, quality of raw water, type and quantity of coagulant, dewatering process, among others. Sampling procedures may also cause significant variation of tests results. To separate sampling errors from "real" variation of characteristics, representative samples for laboratorial tests must be composed using concepts of the Theory of Sampling (TOS). This paper evaluates the effects of sampling method and number of increments to compose the representative sample on the variability of water content and specific gravity of WTS generated at Cubatão WTP in São Paulo, Brazil. Results, valuated by analysis of variance methodology (ANOVA), indicated that both control parameters are influenced by the sampling method and also by the number of increments. In conclusion, the application of TOS concepts to compose representative samples is mandatory to obtain reliable WTS parameters.

Keywords. Theory of sampling, water treatment sludge, representative sample, waste reuse, geotechnical applications, analysis of variance (ANOVA).

1. Introduction

Water treatment sludge (WTS) is generated from washing decanters and/or filters at water treatment plants (WTP). WTS is generally composed of 97% of water, and 3% of impurities removed from raw water, such as colloids, sand, organic matter and algae, and by chemical compounds added at the WTP for coagulation (coagulant), disinfection (chlorine), dental protection (fluorsilicic acid) and pH correction (lime). The high water content is a drawback for reuse as geotechnical material: even after dewatered, WTS has

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[2] Calculated by the authors from the correlation between Brazilian drinking water production [17] and the percentage of WTS generated from raw water treatment [18–20].

properties similar to fluids rather than to soils [1]. Water content is an essential parameter to specify additive (like lime or filler) content necessary to improve WTS workability.

WTS reuse should be based on the determination of reliable physical, chemical and mechanical parameters, which vary for each WTP and along time for the same WTP, and depend on characteristics of raw water, suspended solids, inserted chemicals, operation procedures (e.g., washing of decanters), dewatering process, and local geology [2,3]. Hence, variability and sampling representativity must be considered for WTS reuse [4]. The sampling procedure most often used, "*grab sampling*" (sample obtained by spooning from the top of the lot), may cause significant variation of tests results [6], particularly for high water content materials, which segregate in the containers. However, discussions about WTS sampling were not found in the literature.

The importance of sampling procedures is a known subject for construction and demolition waste [5], cement industry, mining, among others. Obtaining representative samples ("*sampling correctness*") and reliability of parameter determination mainly depend on homogenization and mass reduction procedures, and production of composite samples [6]. For waste, an additional step is cardinal: the definition of the sampling time period, since composition varies temporally.

This paper presents the methodology used to compose a representative sample of Cubatão WTS, according to the concepts of the Theory of Sampling (TOS) stated by Pierre Gy, aiming to separate sampling errors from real variability of WTS characteristics and to obtain reliable parameters. Additionally, the influence of sampling procedure and number of increments to compose a representative sample was investigated. The control parameters were: water content (w) and specific gravity of grains (G_s). Results were evaluated by analysis of variance methodology (ANOVA).

2. Definitions and concepts of the Theory of Sampling (TOS)

TOS covers all aspects of sampling, presents sampling errors related to heterogeneous materials, provides the tools to evaluate, eliminate or reduce errors, states the principles for sampling correctness, and defines the difference between "*correct sample*" (truly representative samples) and "*incorrect sample*" (not representative). TOS comprises practical aspects of sampling, e.g. the correct way to extract a sample, and statistics to characterize heterogeneities, to estimate errors and to generalize results [6,7].

Some basics terms are presented to improve the understanding of this paper: the *lot* is the sampling target or all the original material that will be sampled (a pile, a barrel, etc.); a *sample* is the amount of material correctly extracted; a *specimen* is the amount of material non-correctly extracted, thus, biased; *increment* is a partial sample, that combined with other partial samples provides the *sample* (composite sample).

Other important definition is the "*lot dimensionality*", valued from zero to three. The lot is 0-D if the whole lot is extracted as sample or if completely homogeneous. 1-D lot is not homogeneous and presents an elongated shape (e.g., when the lot is transported by a conveyor belt). For 1-D lot the sampling must cover the two transversal dimensions of the lot, i.e. height and width. 2-D lot is basically plane (height much lower than the dimensions of the plane area). 3-D lot is defined when the samples cannot fully cover any of the dimensions (e.g., a pile), and can be transformed in 2-D or 1-D lots to make sampling easier [6].

A sample is representative only when the sampling process is accurate and reproducible. Requisites are: definition of lot dimensionality, type of extraction equipment, and size of increments; elaboration of a sampling plan considering of heterogeneities of the material; random extraction of increments from the lot; complete homogenization before mass reduction; and adequate mass reduction procedures. Some recommendations: composite samples are preferred; the size of the increments must be as small as possible; the number of increments must be as high as possible; if possible, turn 2-D and 3-D lots into 1-D lot; if necessary, reduction of particle size [6,7].

Methods/equipment of mass reduction (to obtain a given mass of material from the lot) were compared by [6]. Results indicated that grab sampling is the worst method to ensure representativity, the best equipment is the riffle splitter, and the "*spoon method*" lies between grab sampling and splitter. The spoon method consists of spreading the material in a flat container in a "S" pattern layer by layer till obtaining a final layer of constant thickness; then 5 sub-samples are extracted throughout the layer thickness with a spatula, and the 5 sub-samples are combined to produce one composite sample.

3. Materials

Samples of dewatered WTS generated at Cubatão WTP, located in São Paulo, Brazil, were collected directly from the centrifuges. Cubatão WTS is composed of quartz, goethite, muscovite, kaolinite, and amorphous; and contains a large amount of ferric chloride (coagulant used at the WTP), attested by the high concentration of iron (47.5%) detected by FRX (that also showed 18.6% silicon and 10.1% aluminum). Cubatão WTS presents pH 7. The particle size distribution indicated 65-70% clay, 18-25% silt, and 5-14% sand. Specific gravity of grains is 2.9-3.2 g/cm^3 [1,8]. The solids content is low, 20-30%, corresponding to water content of 160-239%. Consistency index is between -0.08 and -1,15, presents undrained shear strength (S_u) of 1.3 kN/m^2, thixotropy [8], and shear thinning behavior at low shear rates [1]. Increase of particle size was observed during air drying (formation of grains with size of sand and gravel), such as formerly observed by [3,9–11], probably due to agglomeration of clay particles by ferric chloride. Dry-to-wet compaction curves showed parabolic shape (γ_d=1.4 g/cm^3; w_{opt}=37%), while wet-to-dry curves did not present a peak: dry unit weight increased with water content decrease [12], as observed for other WTS [3].

4. Methodology and experimental procedure

4.1. Composing the WTS representative sample (Sampling Plan)

Figure 1 depicts the methodology adopted for obtaining the representative sample of Cubatão WTS. First, the lot was defined as the total amount produced by the centrifuges during a month at the WTP (60 tons per day, 20 days a month). Second, the target sample was established as 200 kg, to attend to the ongoing investigations on WTS reuse by the research group. Third, the lot dimensionality was defined as 1-D: Cubatão WTS is delivered from the centrifuges to the trucks by conveyor belt. This definition was important to design the strategy to extract increments from the lot. Fourth: collection of 20 increments of 10 kg each, i.e., one daily increment for 20 days. Each increment was collected using a 20 L plastic bag.

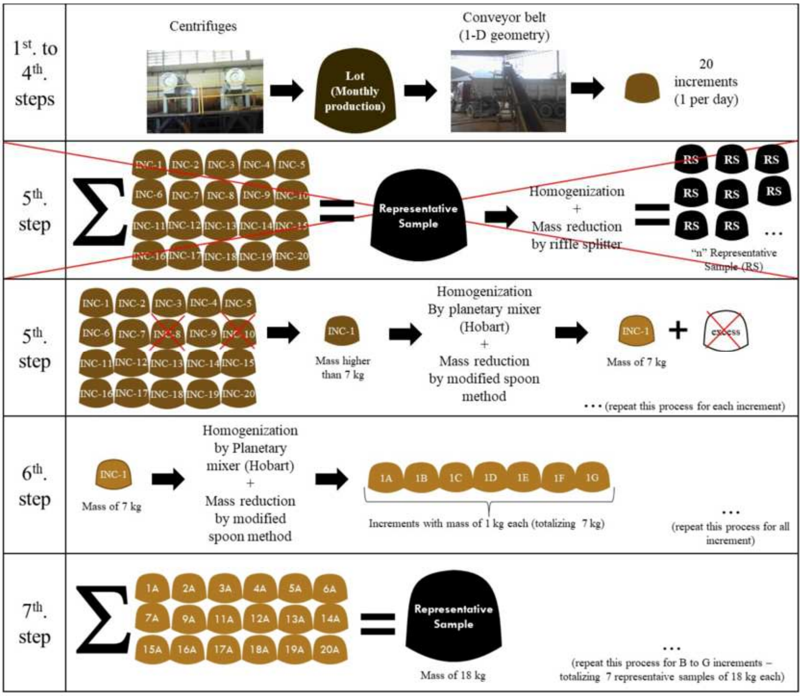

Figure 1. Schematic methodology to compose the representative sample of Cubatão WTS.

According to TOS, the fifth step would be the homogenization of the target sample and mass reduction by riffle splitter to the desired quantity for laboratory tests. In other words, all increments would be added to a container to reach the target sample (200 kg) and homogenized to obtain a composite sample. Yet, WTS is thixotropic [8] and becomes very hard to homogenize by hand in great quantities, while the available planetary mixer was limited to 20 kg. The traditional riffle splitter provides the most reliable representativity [6] for grains and powders, but it proved to be unsuitable for mass reduction of WTS (consistency of paste). Thus, homogenization and mass reduction procedures had to be adapted to the WTS, as follows (adopted 5th step).

The mass of each collected increment varied from 4.5 to 16 kg. As constancy of mass increment is important for sample representativity, a new 7-kg target increment was defined, and two increments (INC-8 and 10) with lower weight were discarded. Increments with mass higher than 7 kg were homogenized using the planetary mixer for 5 minutes (generating a 0-D lot) and reduced to 7 kg by the "*modified spoon method*". The modified spoon method consisted of extracting samples of equal volume necessary to reach 7 kg by spoon, directly from the bowl of the planetary mixer (WTS is very sticky and difficult to spread in a "S" pattern layer by layer). Excess material was discarded.

In the sixth step, each increment of 7 kg was homogenized again using the planetary mixer for 5 minutes, and the mass reduction was performed using the modified spoon method to obtain 7 samples of 1 kg. Therefore, each 1-day increment was homogenized and divided in equal 7 1-kg samples (A to G). Seventh, all 1-kg samples with the same letter, each from one day of the month, were mixed in a mixing bowl and homogenized by planetary mixer for 5 minutes to obtain one representative sample of Cubatão WTS. Considering that sample collection comprised 20 days, but 2 increments were discarded due to insufficient weight, 18 1-kg samples with the same letter were mixed, producing one representative sample of 18 kg. In total, 7 bags (A to G) of 18 kg of representative sample of Cubatão WTS (126 kg) were obtained.

4.2. Influence of sampling method

To understand the difference of grab sampling against sampling correctness according to TOS concepts, a new random 1-day increment was collected at Cubatão WTP and stored in a bag. At the laboratory, "grab samples" were collected directly from the bag without homogenization and mass reduction procedures. Six w and 6 G_s tests (ASTM methods [13] and [14]) were carried out for the grab samples and the TOS samples.

4.3. Influence of the number of increments

Here the purpose was to investigate whether the number of increments to compose the representative sample could be reduced without changing the average value of the control parameters. Therefore, 3 different samples were composed, with 18, 12 and 6 increments. The increments to compose each sample were chosen randomly by sortition. The sample composed of 18 increments was prepared using all the increments, as explained in 4.1. The 12-increments sample was composed of INC-2-5, 7, 11-13, 15,16, 18, and 19. The 6-increments sample was prepared with INC-1, 4, 5, 13, 14 and 20. Six w and 6 G_s tests ([13] and [14]) were carried out for each composite sample.

4.4. Statistical analysis

Analysis of variance (ANOVA) of results at a level of significance of 5% was performed to evaluate significant differences between the groups, using MINITAB 17 software. The independent variables are the sampling method (grab and TOS sampling) and the number of increments (18, 12 or 6), while the dependent variables are w and G_s. The adopted null hypothesis was $H_0=\mu_1=\mu_2=\mu_3=\mu$ (i.e., the average of populations is equal) and the alternative hypothesis: H_A= at least one average is different. The P-value (probability value) was calculated to evaluate the significance of H_0 hypothesis. When P-value is lower than the adopted value of significance (5% or 0.05 in this research), the H_0 is rejected, i.e., can conclude that at least one average is significantly different.

5. Results and discussions

5.1. Influence of sampling method

Results indicate that w and G_s is higher for grab than for TOS sampling (Table 1). The box plot (Figure 2) shows that w values obtained by grab sampling are more dispersed than by TOS sampling.

ANOVA analysis yielded P-value = 0.000 (<0.05, H0 rejected) for w and P-value = 0.001 (<0.05, H0 rejected) for Gs. This means that the obtained values are different and, therefore, the sampling method had a significant effect on both control parameters. P-values indicate that w is more affected by the sampling method than Gs. The higher w values obtained by grab sampling is coherent with observations at the laboratory: WTS segregates in two phases (water and solids) when at rest, and it is noticeable that water moves to the borders and top of the container. As grab sampling only extracts material from the top of the lot, higher values of water content are to be expected. Considering that the coagulant ferric chloride may be dissolved in the pore water of WTS, the accumulation of water at the top of the lot could also explain the higher values of Gs.

Table 1. Results of control parameters collected by "grab sampling" and "TOS sampling".

Sampling method	w (%)	G_s	Sampling method	w (%)	G_s
Grab sampling	348.4	3.23	TOS	343.3	3.06
	350.1	3.26		343.0	3.16
	348.5	3.29		342.5	3.20
	348.5	3.35		343.1	3.22
	349.9	3.28		342.6	3.16
	350.6	3.31		343.7	3.17

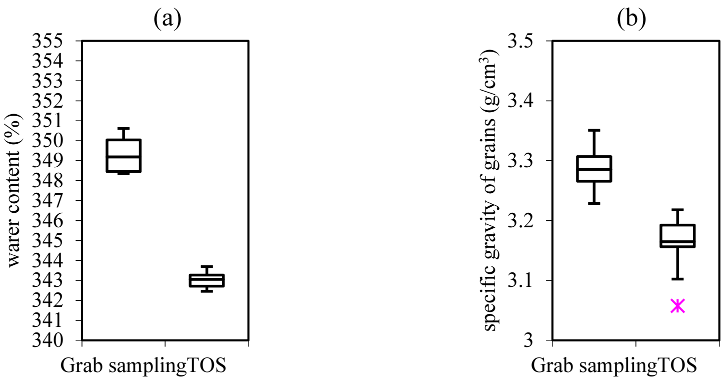

Figure 2. Influence of sampling procedures: (a) water content and (b) specific gravity of grains.

5.2. Influence of the number of increments

Water content values are higher for 12-increments sample than for 18 and 6 increments (Table 2), while specific gravity of grains is lower for 6 increments than for 18 and 12 increments. The box plot in Figure 3 shows that G_s values for the 6-increments sample are more dispersed and the average is lower than for the samples of 18 and 12 increments.

ANOVA analysis yielded P-value = 0.000 (<0.05, H_0 rejected) for w and P-value = 0.010 (<0.05, H_0 rejected) for G_s. Therefore, the number of increments had a significant effect on both control parameters, and w was more affected than G_s by the number of increments. In fact, w is very sensitive to WTP operational procedures. The 12-increments sample included an increment with very high w (~497%), probably collected in a day with problems with the centrifuges. The results indicate that the composed sample will be more representative the higher the number of increments.

The Tukey method revealed that G_s is statistically equal for 18 and 12 increments (indicating that reduction to 12 increments would not significantly change the average G_s), but different when the number of increments was reduced to 6. However, index parameters (void ratio, porosity, saturation degree etc.) calculated from the three averages yield values not different for practical applications (at the level of significant digits). Furthermore, calculated experimental errors, considering meniscus, temperature and balance, were ~0.02 g/cm^3, whereas standard deviations were ~0.03 g/cm^3.

This means that, despite G_s varying with the number of increments, the variation is not much more influential than the experimental errors, and it can be concluded that Gs is not a good control parameter. On the other hand, experimental errors for water content are very low (~0.07%) and water content is a sensitive control parameter.

Table 2. Results of control parameters for samples composed by 18, 12 and 06 increments.

Control parameters	18 Increments	12 Increments	06 Increments
W (%)	204.8	222.6	207.6
	202.9	223.3	206.3
	203.7	223.3	207.7
	204.0	222.8	207.1
	204.6	223.6	206.7
	204.4	222.6	207.2
Specific gravity of grains (g/cm^3)	3.16	3.16	3.08
	3.14	3.17	3.16
	3.14	3.18	3.12
	3.15	3.17	3.14
	3.21	3.13	3.07
	3.19	3.23	3.11

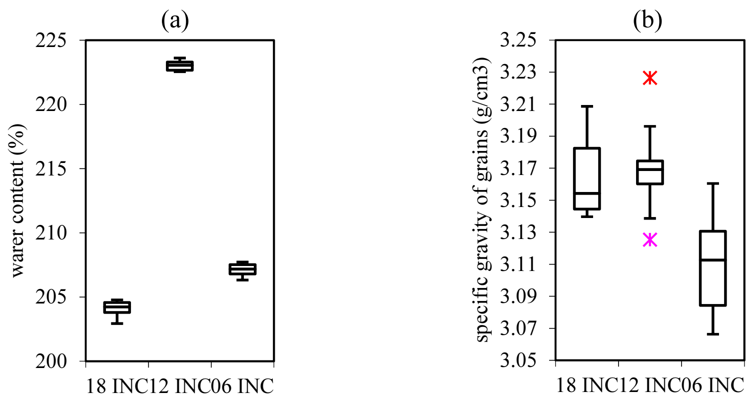

Figure 3. influence of number of increments: (a) water content and (b) specific gravity of grains.

6. Conclusions

The methodology conceived to compose a 1-month representative sample of Cubatão WTS, based on TOS concepts, furthers obtaining more reliable geotechnical parameters, while grab sampling may induce biased results. Water content and G_s varied with the sampling method at a significant level of 5%. The number of increments to compose the representative sample was also significant for w and G_s results, and a higher number of increments (the highest investigated number was 18) yields more trustworthy results. According to ANOVA, water content is more affected by the investigated variables than G_s. Water content proved to be an interesting control parameter to investigate sample representativity, while Gs is less sensitive because of experimental errors.

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