Influence of laboratory techniques on the geotechnical characterization of mining and industrial wastes

Influence de la technique de laboratoire utilisée sur des résultats géotechniques de caractérisation du mien et des pertes industrielles

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ABSTRACT
The design of fine-grained tailing reservoirs requires the determination of their physical, mechanical and hydraulic characteristics to predict both the reservoir filling operation and its reclamation. Once these characteristics are properly determined, moderately realistic tailings behavior prediction, such as its self-weight consolidation, can be obtained from numerical analysis. However, if a simple grain size analysis is not adequately carried out, one can find substantially large discrepancies between the analytical predictions and the actual field behavior. This paper presents results of an extensive laboratory program executed to obtain geotechnical parameters of mining and industrial wastes. Different residues from mining and industrial processing of bauxite were studied. The results of geotechnical characterization tests carried out on all materials are presented and compared. The effects of the testing technique utilized on the geotechnical characterization results are discussed. It is shown that the prediction of the sedimentation/consolidation behavior of these tailings when discharged as slurry using parameters obtained from characterization tests must be done with care.

RÉSUMÉ
La conception des réservoirs à grain fin de produits de queue exigent de la détermination de leurs caractéristiques géotechniques et de geomechanics de prévoir l'opération remplissante de réservoir et sa récupération. Une fois que ces caractéristiques sont correctement déterminées, la prévision modérément réaliste de comportement de produits de queue, telle que sa consolidation d'individu-poids, peut être obtenue à partir de l'analyse numérique. Cependant, si une analyse granulométrique simple de grain n'est pas exécutée bonne, on peut trouver des anomalies énormes entre les prévisions analytiques et le comportement réel de champ. Cet article présente les résultats d'un grand programme de laboratoire exécuté pour obtenir des paramètres géotechniques du mien et des pertes industrielles. Des résidus du mien et du traitement industriel de la bauxite ont été étudiés. Les résultats des essais de caractérisation ont exécuté sur tous les matériaux sont présentés et comparés. Les effets de la méthode d'essai utilisée sur les résultats géotechniques de caractérisation sont discutés. Par conséquent, la sédimentation/la prévision comportement de consolidation de ces produits de queue une fois déchargées comme boue employant des paramètres obtenus des essais de caractérisation doit être faite avec soin.

Keywords: mine tailings, geotechnical characterization, laboratory tests, influence of methodology

1 INTRODUCTION

Many models employed in the design of the disposal of fine grained tailings discharged as slurries inside reservoirs use results of characterization tests to predict both the filling operations and its reclamation (e.g Carrier et al., 1983; Carrier & Beckman, 1984). The adequateness of the design is, therefore, dependent on the quality and methodology used to perform the characterization tests. However, such tests were developed to characterize soils and standard procedures are employed worldwide. Mine tailings and industrial wastes do not behave as typical soils. For example, it is very usual that they have a pore fluid other than water. As these residues generally present low consolidation rates, several techniques are used in an attempt to speed up the deposition process. It is common to add chemical flocculants and sand to the wastes and such procedures will affect the final behavior of the reservoir. Nevertheless the wastes normally are still characterized using the soil standards procedures and the results of such tests are used to predict the field behavior where the wastes are in different conditions than those during laboratory characterization. This paper presents and discusses results of characterization tests performed on residues from mining and industrial processing of bauxites. Different methodologies were used to check the effects caused by others fluids than water on the grain size distribution curves and Atterberg limits data. The dependence of the results on the type of methodology was confirmed. As a consequence, the prediction of sedimentation and consolidation of these tailings when discharged as slurry using parameters obtained from characterization tests must be done with care.

2 TESTED MATERIALS

In this research, all residues tested came from bauxite exploration and its industrial processing to extract alumina, which is the basic product to obtain aluminium. The waste coming from mining operations herein considered is a result of bauxite ore washing using water to obtain a given grain sized product, especially to eliminate clay sized particles. It came from a mine located within a Brazilian rain forest and this residue is referred to in this paper as RF-MUD (rainforest mud). The other residues, usually referred to as “red mud”, came from the industrial plants. The majority of testing results were obtained using a red mud from the state of Minas Gerais, southeast of Brazil. Two wastes from this region were tested. One of them had the pH around 14 because its pore fluid was caustic soda. The caustic soda is the fluid used in the Bayer System to digest the bauxite to extract alumina and, then, to obtain the aluminium. It is cited here as NON-NEUT-MUD-01.

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The other red mud was formed by addition of sulfuric acid to the NON-NEUT-MUD-01 to reduce its pH. It is referred in this paper as NEUT-MUD-01 (neutralized red mud 02). Another red mud tested came from the industrial process of the RF-MUD and is cited here in as NON-NEUT-MUD-02 (non neutralized red mud 02). It has also the pH around 14.

2.1 *Chemical and mineralogical analysis*

Bauxite is not defined as a mineralogical specimen. This term refers to a mineral or to a mixture of minerals essentially constituted by alumina hydrate, iron oxide, aluminium silicate and titanium oxide. According to Gomes (1988), bauxite is a rock rich in aluminium oxide (Al_2O_3) as well laterite is rich in iron oxide (Fe_2O_3). The term "bauxite" is used to designate a substance that is a mixture of several hydrated aluminium oxides and its main constituents are gibbisite ($Al_2O_33H_2O$), bohemite ($Al_2O_3H_2O$) and diaspore ($HAlO_2$). All Brazilian bauxites are mostly red in color and rich in iron oxide and iron hydroxide and poor in silica (Santos, 1989). The waste from the washery process probably will have all the chemical components originally present in the ore. However, the tailings from industrial process will have their mineralogical composition strongly dependent upon the ore uniformity and the applied technology used to beneficiate it (Bulkai, 1983).

Li & Rutherford (1996) analyzed the chemical and mineralogical composition of bauxites from the southeast region of Brazil and their corresponding red mud. They concluded that these two types of tailings have very similar mineralogy but very different amounts of each mineralogical and chemical element. All red mud they tested had presented one specific product as a consequence of the industrial process: the Bayer sodalite. It influences the red mud sedimentation rate, reducing it in caustic soda, and has high ability to exchange sodium ions (Li, 2001). Li & Rutherford (1996) also concluded that both composition and content of amorphous material present in red mud can also influence its sedimentation rate.

Table 1 summarizes the chemical analysis results obtained for the four wastes studied here. Almost no differences in the chemical and mineralogical composition between the NEUT-RED-MUD 01 and the NON-NEUT-RED-MUD-01 could be observed. Because of this and because they have almost the same grain sizes, as it will be shown later, all differences in their behavior was assumed to be only due to the influence of pore fluid type.

All the wastes were also submitted to electronic microscopy analysis. Basically, it was found the same elements previously identified by the chemical and mineralogical analysis using X-ray diffraction. The images showed that all wastes could be considered homogeneous. A large amount of amorphous material was noticed. The tailings particles showed a clear tendency to flocculate. Images of the NEUT-RED-MUD 01 and the NON-NEUT-RED-MUD-01 are demonstrated in Figures 1(a) and (b) respectively. It can be observed that there is a larger tendency of particle flocculation in the latter.

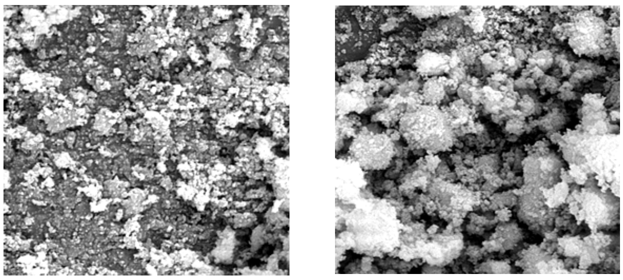
3 TESTS RESULTS

3.1 *Grain size analysis*

The particle size of mining and industrial wastes is closely related to the ore mineralogy and to the industrial technique used to extract and to process them. As these features vary from region to region and even from plant to plant, it is almost impossible to have a standard granulometric curve for this type of material. In the industrial process, there are no standards regulating the amount of chemical substances that can be used, like flocculants, which can be applied to accelerate the deposition rate of the wastes. Therefore, it is common to find grain sizes differences even among mine and industrial residues obtained from the same ore source and same industrial process. Even more, it is probable to have large particles segregation inside the reservoirs where they are launched usually in a mud form. Some places of the reservoir may concentrate the coarse fractions whereas another will receive the finer fraction. All these aspects indicate the impossibility to have just one grain size distribution curve as a guide to predict the geotechnical behavior of these wastes. Somogyi & Gray (1977) already had realized that the grain size distribution was influenced by the industrial process and could not be adequate to estimate sedimentation rates of wastes in the field.

Table 1 Chemical analysis of the mining and industrial bauxite wastes

Chemical element	rf-mud	non-neut-mud-02	non-neut-mud-01	neut-mud-01
SiO_2	21.0	11.9	5.7	6.2
Al_2O_3	31.2	27.4	17.2	17
Fe_2O_3	28.0	31.6	52.8	50.3
FeO	0.28	0.14	0.56	0.42
CaO	0.17	2.3	3.1	4
MgO	<0.10	0.10	0.15	0.21
TiO_2	3.8	3.4	5.5	5.5
P_2O_5	<0.05	<0.05	0.37	0.35
Na_2O	0.51	7.9	1.9	2.4
K_2O	0.02	0.01	0.15	0.12
MnO	<0.01	<0.01	0.06	0.05
Cr_2O_3	0.04	0.051	0.097	0.088
NiO	<0.003	<0.003	<0.003	<0.003
F	0.047	0.05	0.086	0.10
Cl	0.008	0.39	0.031	0.043
Fire lost	14.84	14.56	11.37	12.67
TOTAL	100.08	99.79	99.06	99.45



(a) (b)

Figure 1. Images from electronic microscopy increased 5.000 times: (a) NEUT-RED-MUD 01 and (b) NON-NEUT-RED-MUD-01

The grain size distribution analysis conducted throughout this research clearly show how the granulometric curve is dependent on the used lab technique. All the grain size analyses done in this research were conducted after the residues had been first air dried. The sedimentation tests were performed with and without sodium hexametaphosphate, which was the substance used as deflocculant. Both water and the industrial fluid of each waste were used in the tests to check in which case the fine fraction tended to flocculate more. In order to study the influence of the finer fraction in the whole behaviour of the residues, separate tests were also conducted using only the material finer than the # 200 sieve. In all sedimentation tests carried out using the industrial fluid instead of water, no matter if this fluid was neutralized or caustic, it was noticed the formation of very thin crystal plates, which were deposited at the bottom of the sedimentation burette and its amount tended to increase with time. Therefore, the density of the fluids used in the sedimentation tests changed. This was monitored within the 24 hours testing period and the obtained results were utilized to get the grain size distribution curves.

In general, it was observed that the addition of deflocculant (sodium hexametaphosphate) was less efficient when using caustic fluid as sedimentation media than when using neutralized fluid. The results obtained from tests using non neutralized fluid showed lower clay sizes than when using the neutralized industrial fluid. This shows that the caustic fluid tends to flocculate the particles, as already was concluded by Solymár et al. (1992). The results of the NEUT-RED-MUD 01 were more influenced not by the type of fluid (water or the neutralized industrial fluid) but by the use or not of deflocculant (sodium hexametaphosphate). The neutralized industrial fluid combined with the sodium hexametaphosphate also tended to flocculate grains coarser than the #200 mesh. However, just the opposite behavior was observed when the fraction finer than the # 200 mesh was tested separately, indicating that this mixture acted as a dispersant to this range of particle size. The grain size curves of the NON-NEUT-MUD-01 obtained using water during all tests were very similar to those obtained for the NEUT-MUD-01. This result was expected since these two materials only differ in pore fluid from each other.

Table 2 summarizes the grain size range found from all tests performed in the NEUT-RED-MUD 01 and NON-NEUT-RED-MUD 01. Figure 02 shows the area that includes all the grain size distribution curves of these two materials

In Table 2 "Upper Bound" represents the curve showing smallest grain sizes and "Lower Bound" the curve with the coarser grain sizes. It can be seen that, depending on the testing methodology, one can find 0% to more than 50% of sand or 0% to more than 60% of clay sizes in these wastes. It is quite clear, therefore, that any prediction of the behavior of this type of waste based on their grain size distribution curves will be strongly dependent on the technique used to obtain them.

Table 2. Grain size range found analysing the NEUT-RED-MUD 01 and the NON-NEUT-MUD-01

Material	Upper Bound	Lower Bound
NEUT-RED-MUD 01	0% of sand	63% of sand
	49% of silt	37% of silt
	51% of clay	0% of clay
NON-NEUT-RED-MUD 01	0% of sand	67% of sand
	48% of silt	37% of silt
	32% of clay	6% of clay

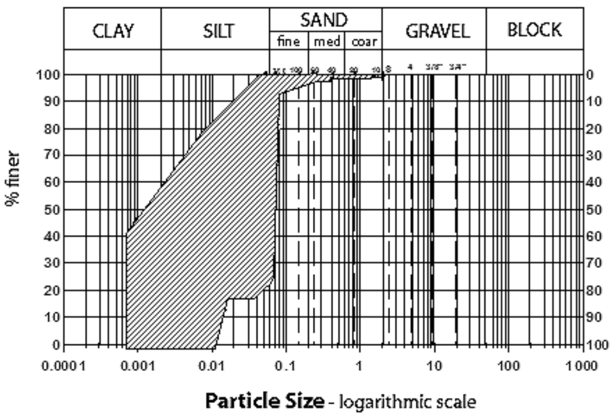


Figure 2. Area that includes all grain size distribution curves obtained for the NEUT-RED-MUD 01 and NON-NEUT-RED-MUD-01

Furthermore, the use of standard techniques may provide predictions that may be fairly distinct from the actual field behavior owing to differences in pore fluid.

3.2 *Consistency Limits of the residues*

Wastes from bauxite ore, no matter if from the industrial or the mining phase, generally have low plasticity index. However, they behave like clays if compared to natural soils (*Li & Rutherford, 1996).* Alves (1992) also studied the NEUT-RED-MUD 01, obtaining its consistency limits. The samples were dried and not dried before the tests. It was observed that without previous drying, the plasticity index was higher. This procedure influenced more the values of the liquidity limit.

Other consistency limits values were obtained during this research, using both the standard procedures and also using the industrial fluid of each material instead of water. Tests were also carried out without drying the wastes previously. Grain sizes finer than the # 200 mesh and finer than the # 40 mesh, which corresponds to 90% of all wastes grain sizes, were studied separately. The findings are summarized in Table 3. It can be seen that the caustic fluid increased the plasticity of the fraction finer than the # 40 mesh of both red muds, changing its plasticity limit when compared to the value obtained using water. On the other hand, it is also possible to observe that the neutralized fluid reduced the plasticity of the NEUT-RED-MUD-01 compared to the values obtained when water was used. Considering the samples with grains coarser than the #40 mesh, the plasticity was higher when it was followed a drying path instead of the standard wetting path when were used caustic or neutralized fluids. So, it is quite clear that the re-hydration capacity of this residue is limited. The plasticity limit changed very little, but the plasticity index increased by almost five times in the case of the NON-NEUT-RED-MUD-01.

The shrinkage limit was also determined following the methods specified by the ASTM D427 using water and the industrial fluid. In this case, all the samples were formed by grains finer than the #40 mesh. The shrinkage limit of the NEUT-RED-MUD-01 varied between 30 and 35, values that are close to the plasticity limit. The NON-NEUT-RED-MUD-01 values ranged between 34 and 39. Since they are above the obtained plasticity limit value, it is obvious that one of the tests was wrong. Because of this, the curve that relates void ratio and the gravimetric moisture content was also used to estimate the shrinkage limit (Villar, 2002). They were obtained according to the BS1377 standard for the NEUT-RED-MUD-01 and NON-NEUT-RED-MUD-01 using each proper industrial fluid. It was observed that the shrinkage limit of the NEUT-RED-MUD-01 found using this technique was around 32, almost the same as that obtained using the ASTM D427. However, for the NON-NEUT-RED-MUD-01 it was found a shrinkage limit of 19.5, quite different from previously obtained value. The caustic mud contracted more than the neutralized one, reaching a final void ratio of approximately 0.7, while the neutralized mud reached 1.2.

3.3 *Grains density or specific gravity*

The grain density of mining wastes is highly dependent on the mineralogy of the deposit rock and can vary substantially. Li & Rutherford (1996) found values of relative density varying between 2.70 to 2.87 for most of the bauxite from the southeast of Brazil. These values were justified due the high presence of gibbsite and low levels of iron ore.

Some waste samples were tested during this research to determine their grain density using international standards and the industrial fluid instead of water. The determination also was made using both integral samples and samples that were finer and coarser than the #200 mesh. The results are shown in Table 4. It can be noticed that almost all the tests carried out using the industrial fluid instead of water provided the lowest values of grain density regardless if the fluid was neutralized or caustic or if the sample was integral or partial. If only the results obtained using the industrial fluids are compared, the grain density values measured in the tests performed using the caustic fluid were higher than those obtained using the neutralized one.

One possible explanation to justify the difference between grain densities values obtained using industrial fluid instead of water may lie on adopted testing standards.

One of the standard procedures of this test is to boil the pycnometer to facilitate elimination of air trapped within soil grains. But, when the industrial fluids are heated, especially caustic ones, new chemical bonds between the minerals present are formed. Indeed, this technique can be used in industrial processes to change the chemical and mineralogical constitution of the red mud. The objective is to provide a higher waste sedimentation rate and to facilitate the separation between the red mud and alumina (Solymár et al., 1992).

4 CONCLUSIONS

The design of fine-grained tailing reservoirs requires an as full as possible knowledge of physical-chemical-mineralogical, mechanical and hydraulic characteristics of the waste. Once these characteristics are properly determined, reasonable predictions of the tailings behavior, such as its self-weight consolidation, can be obtained from appropriated numerical analysis.

There are cases were design is based upon results of simple characterization tests. Under this condition, if a simple grain size distribution analysis is not adequately carried out, one can find large discrepancies between analytical predictions and actual field behavior.

Wastes are usually characterized using soil standards procedures. Results of such tests are or may guide predictions of field behavior.

This paper presents partial results of an extensive laboratory program executed to obtain geotechnical parameters of mining and industrial wastes. Different residues from mining and industrial processing of bauxite were studied. The results of the characterization tests carried out on all materials were presented and compared.

It was shown that, depending on the used testing technique, large variation on grain size distribution can be found, being possible to classify the residue either as a sandy or a clayey material. As a consequence of that, it can be expected that the prediction of the sedimentation/consolidation behavior of the waste can be quite variable.

It was verified that the major factor affecting the results of the grain size analysis, consistency limits and specific gravity of grains tests was the fluid used in the experiments. Owing to that, it is recommended to characterize mining and industrial wastes using not the soil standards procedures but modified methods. Data obtained with modified procedures using the industrial fluid instead of water might better represent the actual field behavior of the waste.

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Table 3 Consistency limits of the studied wastes.

MATERIAL		Finer than #200			Finer than #40			Finer than #40 – drying path		
		Liquid limit	Plastic limit	Plasticity index	Liquid limit	Plastic limit	Plasticity index	Liquid limit	Plastic limit	Plasticity index
RF-MUD	water	49.4	47.6	1.8	48.1	23.8	24.3	53.8	25	28.8
NON-NEUT-RED-MUD-02	Industrial fluid	42.8	16.1	26.7	40.8	20.1	20.7	-	-	-
	water	-	-	-	43	30	13	-	-	-
NEUT-RED-MUD-01	Industrial fluid	35.8	28.6	7.2	33.4	27	6.3	48.8	34	14.8
	water	38.5	27.1	11.4	41.4	32.7	8.7	-	-	-
NON-NEUT-RED-MUD-01	Industrial fluid	36.5	28.2	8.4	33.9	26.8	7	63.6	29.3	34.3
	water	41.2	27.4	13.8	33.5	27.2	6.3	-	-	-

Table 4 Relative density of the studied wastes grains

MATERIAL		Coarser than #60	Coarser than #100	Coarser than #200	Finer than #200	INTEGRAL
NEUT-RED-MUD-01	water	3.66	3.65	3.65	3.57	3.68
	Industrial fluid	3.49	3.51	3.49	3.54	3.39
NON-NEUT-RED-MUD-01	water	3.56	3.58	3.59	3.62	3.59
	Industrial fluid	3.53	3.54	3.60	3.54	3.55
NON-NEUT-RED-MUD-02	water	-	-	-	-	3.16
	Industrial fluid	-	-	-	-	2.86
RF-MUD	water	2.96	2.96	2.92	2.92	2.96

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