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Using density to determine the solids content of construction slurries

Le rôle de la densité pour déterminer la teneur en matières solides des boues de construction

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ABSTRACT: Bentonite slurries, polymer fluids, cement-bentonite slurries and cementitious grouts are used in a wide variety of geotechnical works including tunnelling, diaphragm walls, bored piling, cut-off walls and ground improvement. The density of these fluids is often the key parameter used in their preparation and control and yet over the years very little has been done to develop test procedures or to improve the precision of on-site measurements. The most commonly used tool for on-site density measurement is the mud balance, a device ‘borrowed’ from the oil industry decades ago. It is currently specified in many national and industry standards despite its relatively poor resolution. This paper sets out some of the problems associated with density measurement in the laboratory and in the field and the confounders that may limit the precision with which density can be measured and the tests results interpreted.

RÉSUMÉ : Les boues de bentonite, les fluides polymères, les boues de ciment-bentonite et les coulis de ciment sont utilisés pour de nombreux travaux géotechniques, y compris les tunnels, les parois moulées, les pieux forés, les parois étanches et l'amélioration du sol. La densité de ces fluides est souvent le paramètre clé utilisé pour déterminer les proportions du mélange de contrôle. Cependant jusqu'à présent il y a eu peu de recherche pour développer des méthodes d'essai ou pour améliorer la précision des mesures en chantier. A l'heure actuelle, une balance de boue empruntée à l'industrie pétrolière est utilisée, conformément aux nombreuses normes nationales et industrielles malgré une précision relativement médiocre. Notre étude présente les problèmes associés avec l'utilisation de cet appareil et recommande des alternatives.

KEYWORDS: Density measurement, bentonite slurries, cement-bentonite slurries, polymer fluids, grouts.

1 INTRODUCTION

Density is a key parameter for the preparation and control of many fluids used in geotechnical engineering including excavation-support fluids based on bentonite clays and polymers, cementitious and chemical grouts and slurries used in cut-off walls. For all these fluids, density is typically used as a surrogate for the solids content of the fluid. For a fluid containing a mass S of solids per cubic metre of fluid, the density of the fluid (mixture) ρ_m can be calculated from Eq. 1.

$$\rho_m = S + (1 - \frac{S}{\rho_s})\rho_w \quad (1)$$

where ρ_w and ρ_s are respectively the density of the suspending fluid (water for most construction fluids) and the solids used to form the suspension. It follows from Eq. 1 that the solids content in the fluid is given by:

$$S = \rho_s \frac{(\rho_m - \rho_w)}{(\rho_s - \rho_w)} \quad (2)$$

Differentiating Eq. 2 gives the sensitivity of the solids content to density change:

$$\frac{dS}{d\rho_m} = \frac{\rho_s}{(\rho_s - \rho_w)} = \frac{G_s}{(G_s - 1)} \quad (3)$$

where G_s is the grain specific gravity of the solids. As an example of the application of Eq. 3, bentonite slurries for excavation support are typically prepared at concentration of 25 to 50 kg/m³ (≈ 2.5 to 5% w/w). The grain specific gravity of a bentonite powder may be about 2.28 (calculated from a rule of mixtures assuming that the grain specific gravity of dry sodium bentonite powder is 2.74 and that the moisture content of the powder as supplied is 13% by dry weight), so that:

$$\frac{dS}{d\rho_m} = 1.78 \quad (4)$$

Now the smallest scale division on the mud balance, the instrument most often used for on-site density measurements is 0.01 g/ml (10 kg/m³) and the results from the test are usually reported to 0.01 g/ml which is effectively the best repeatability of the instrument. From Eq. 4, 0.01 g/ml corresponds to 17.8 kg of bentonite powder per cubic metre of slurry or 44% of the bentonite concentration in a slurry prepared at 40 kg/m³. It is therefore clear that the mud balance can identify only gross errors in the batching of fresh bentonite slurries. Despite this, mud balance tests continue to be made on construction sites presumably because those specifying or undertaking the tests believe that they provide a useful measure of control for the as-mixed concentration of fresh bentonite slurries.

For cementitious fluids, the resolution for cement content is slightly better as the grain density of cement is higher, so that 0.01 g/ml corresponds to ≈ 15 kg/m³ or perhaps 10% of the total concentration of cementitious material in a cement-bentonite slurry. For a cement grout at a water/cement ratio of 1 (a cement content of ≈ 760 kg/m³), the resolution is a useful 2%.

Lam (2016) found that under laboratory conditions, slurry density measurements with a pycnometer procedure (weighing a known volume of liquid) can achieve an accuracy of 0.001 g/ml. Such an accuracy would give a more useful resolution of bentonite concentration at 1.8 kg/m³ or for example, 4.4% of the concentration for a 40 kg/m³ slurry. Table 1 presents the concentration resolution that can be achieved for some common materials dispersed in water with density resolutions of 0.001 and 0.01 g/ml together with the density resolution required to achieve a solids content resolution of 1 kg/m³.

Table 1. Density and concentration resolution data.

Material	G_s	Density change for 1 kg/m ³ change in concentration	Resolvable concentration, kg/m ³ for density resolution of:	
		g/ml	0.001 g/ml	0.01 g/ml
PHPA polymer	≈1	-	Density not useable for assessment of concentration	
Bentonite at 13% moisture content	2.28	0.00056	1.8	17.8
Silica sand	2.65	0.00062	1.6	16.1
Portland cement	3.15	0.00068	1.5	14.7

2 COMPLICATING FACTORS

A number of complicating factors can affect the accuracy of the density measured using a pycnometer. These include:

- dissolved air;
- dissolved salts;
- air bubbles trapped in the fluid;
- temperature;
- the accuracy and repeatability of the equipment used.

2.1 Dissolved air

If water is exposed to air, then the gases in the air will tend to dissolve into the water. Construction slurries under normal conditions therefore can be expected to contain dissolved air and indeed the water phase can be expected to be in equilibrium with air. Bignell (1983) investigated the effect of dissolved air on the density of water and presented the following equation for its effect on density as a function of temperature:

$$\Delta\rho \text{ (g/ml)} = -0.000004612 + 0.000000106 T(^{\circ}\text{C}) \quad (5)$$

where $\Delta\rho$ is the increment in density over that for gas-free water at temperature T in $^{\circ}\text{C}$. As the solubility of gases decreases with increasing temperature, $\Delta\rho$ reduces with increasing temperature. From Eq. 5, at 0°C the change of density on air saturation is -0.000004612 g/ml and at 20°C it is -0.0000025 g/ml, both of which increments are trivial in the context of this paper. At 43°C the effect of equilibration with air is effectively nil.

2.2 Dissolved salts

Typically civil engineering fluids such as clay slurries and cementitious grouts will be prepared with water drawn from a mains supply or a fresh-water well or water body. For waters containing minor amounts of salts, the density of the solution (ρ_{sol}) can be estimated from an equation of the form:

$$\rho_{\text{sol}} = \rho_w + \alpha \text{ TDS} \times 10^{-6} \quad (6)$$

where ρ_w is the density of pure water at the temperature of the solution and TDS is the total dissolved solids content of the solution in mg/l. The value of the parameter α will depend on the species in solution but a value of 0.8 may give a reasonable estimate. Thus, for example, a water with a TDS of 500 mg/l will have a density about 0.0004 g/ml above that of pure water at the same temperature.

2.3 Air bubbles

Bentonite and cementitious slurries are typically rather viscous gelling fluids that can readily trap small air bubbles which will reduce the measured density of the fluid if the measurement is

not made in a pressurised device. Note: there is a pressurised version of the mud balance but as for the standard instrument it has a minimum scale division of only 0.01 g/ml.

It is difficult to give any indication of typical gas bubble contents of construction fluids but contents of 0.01 to 0.1% by volume are realistic noting that sources of bubbles include:

- air trapped in the pores of the powders (bentonite, cement, etc.) as added to the slurry mixer;
- gases sorbed onto the surface of the powders and released on wetting during mixing;
- air introduced into the mix during the mixing process.

Bubbles therefore can be expected to be present in and reduce the measured density of construction slurries. Allowance may have to be made for this either in the control values used for slurries or by using a density measurement procedure that either compresses the gas or encourages its release.

Bubble release can be encouraged by diluting the test fluid. With a volume calibrated bottle, the procedure could be:

(a) weigh bottle empty, (b) add the test fluid to fill perhaps half the bottle and weigh, (c) fill bottle to about 80% with water, gently swirl and tap the bottle to mix the fluids and encourage bubble release, (d) fill to brim with water and re-weigh. The amount of test fluid to be added at step (b) and water at step (c) to be adjusted depending on the test fluid properties.

2.4 Temperature

Water has a non-linear coefficient of expansion with a maximum density at $\approx 4^{\circ}\text{C}$. Gupta (2002) gives the densities shown in Table 2 for air-free water at temperatures from 0 to 40°C , which covers the likely range of temperatures for samples tested on site, though higher temperatures may be possible, for example, for cement grouts on hot days once hydration is underway. From Table 2, it is clear that assuming a water density of 1 is not appropriate if the temperature of the sample is significantly different from 4°C , and a precision of better than 0.01 g/ml is required.

Table 2. Density of air-free water from 0 to 40°C .

Temperature $^{\circ}\text{C}$	Density g/ml	Temperature $^{\circ}\text{C}$	Density g/ml
0	0.9998	20	0.9982
4	1.0000	25	0.9970
5	1.0000	30	0.9956
10	0.9997	35	0.9940
20	0.9991	40	0.9922

3 DENSITY MEASUREMENT TECHNIQUES

Many construction slurries exhibit a yield stress and thus techniques such as the use of a hydrometer or hydrostatic weighing (weighing of an object of known volume in the fluid) cannot be used as the yield stress will prevent a true reading.

Lam (2016) used a fixed-volume container and an electronic balance (i.e. a pycnometer method) for his tests. His reported accuracy for density measurement of 0.001 g/ml was with a 2-litre fixed-volume container such as a stoppered volumetric flask in a temperature controlled laboratory. This accuracy was relative to calculated densities using assumed densities for the components of the slurries.

In an attempt to find a readily available class of containers that can be used on site, the first author has experimented with a number of container types and has found that 1-litre spring-top bottles (see Figure 1) have advantages over volumetric flasks of: readily repeatable sample volume as the cap is snapped on, wide availability in hardware stores (and as table water bottles in many offices), low cost, and as the stopper is retained on the

bottle there is no risk of using a wrong or poorly fitting stopper.

It was found that under isothermal conditions the weight of a water-filled bottle was repeatable to ± 10 mg between repeat fillings offering a notional repeatability of $\pm 1 \times 10^{-5}$ g/ml.



Figure 1. A typical spring-top bottle

4 DENSITY TRIALS WITH SPRING-TOP BOTTLES

A number of 1-litre spring-top bottles were purchased from a hardware store and small file marks were made on the base of each bottle so that individual bottles could be identified. The bottles were weighed empty and when filled with distilled water. Weighings with water were carried out over a range of temperatures from 4 to over 40°C. It was necessary to undertake measurements over a range of temperatures as both fluid and container will expand on increase in temperature – and for the mass of fluid in the container, the expansion of the container will partially compensate that of the water.

At this point, it is appropriate to add a word of caution in relation to fully-filled bottles. A small change in temperature can lead to a substantial change in pressure in a liquid-filled bottle with no air space. One of the first author's fluid-filled bottles cracked when left full and sealed. Round section bottles may be more robust than square section bottles but be cautious.

The density of the water in the bottle at each temperature was calculated using the volume of the bottle determined from the weight of water filling the bottle at 4°C and the published density of water at this temperature (see Table 2). Note that the weight at 4°C was chosen for convenience, a weight at any of the test temperatures could have been used as the baseline.

The results of these density calculations are shown in Table 3 together with the errors in the measured densities relative to the density calculated using the fifth order polynomial given in Gupta (2002) which matches data from the International Union of Pure and Applied Chemistry published in Lide and Frederiske (1994) to within $\pm 1 \times 10^{-5}$ g/ml for temperatures up to 40°C. Both these data sets are for gas-free water. However, the effect of saturation with air is very small as shown in Section 2.1 above.

Table 3 also shows the calculated densities corrected for the volumetric expansion of the bottle assuming a coefficient of volumetric expansion of $2.9 \times 10^{-5} \text{°C}^{-1}$ (i.e. assuming that the bottle was soda glass with a coefficient of linear expansion $9.8 \times 10^{-6} \text{°C}^{-1}$ and that the coefficient of volumetric expansion is three times that for linear expansion). This coefficient is almost an order of magnitude less than the average coefficient of expansion of water over the range 4 to 40°C, $2.2 \times 10^{-4} \text{°C}^{-1}$ (though noting that the coefficient is strongly non-linear ranging from near zero around 4°C to $3.8 \times 10^{-4} \text{°C}^{-1}$ at 40°C). A coefficient of $2.9 \times 10^{-5} \text{°C}^{-1}$ corresponds to a very minor Table 3. Results of water density measurements with a spring-top bottle.

Temperature °C	Calculated density g/ml	Density error g/ml	Density Corrected for expansion of bottle g/ml	Density error after correction for bottle expansion g/ml
4.0	1.0000	0.0000	1.0000	0.0000
14.1	0.9992	-0.0001	0.9989	-0.0004
20.9	0.9982	0.0002	0.9977	-0.0003
21.2	0.9980	0.0001	0.9975	-0.0004
21.6	0.9981	0.0002	0.9976	-0.0003
26.7	0.9969	0.0003	0.9962	-0.0003
34.7	0.9947	0.0006	0.9938	-0.0003
35.0	0.9945	0.0005	0.9936	-0.0004
41.6	0.9919	0.0003	0.9913	-0.0003
Standard deviation		0.00022		0.00013

volume change of 0.029 ml °C^{-1} for a 1-litre bottle.

The density measurements also will be influenced by thermal changes in the volume of the solid particles (clay, cement, etc.) in the fluid. However, as for glass, mineral solids typically have markedly lower expansion coefficients than liquids and so effects are expected to be trivial in the context of this paper – confirmatory data would be useful.

From Table 3 it can be seen that in principle the combination of a spring-top bottle and an electronic balance can achieve a standard deviation, for water density under laboratory conditions, of 0.00022 g/ml over the temperature range 4 to 42°C. This standard deviation can be somewhat further reduced to 0.00013 g/ml if the correction for the expansion of the bottle is applied. However, these results were achieved for a simple fluid, water under laboratory conditions. Furthermore, there were individual errors up to 0.0006 g/ml .

In a second series of tests with a different bottle, a standard deviation of 0.0002 g/ml was again achieved over the same temperature range but the correction for expansion of the glass had minimal effect. For both bottles the standard deviation could be further reduced by using a lower value of the coefficient of volumetric expansion for the glass than $2.9 \times 10^{-5} \text{°C}^{-1}$.

The conclusion from the tests is that water density measurements can be achieved to of the order of $\pm 0.0005 \text{ g/ml}$ under laboratory conditions but for this it is necessary to apply temperature corrections to the measured values, though corrections for the bottle expansion are probably not justified.

Consideration of thermal expansion of solids invites the use of a ferritic stainless steel container in the place of glass, as the coefficient of volumetric expansion of this steel may be closer to that of water over the temperature range of interest and so provide better volume compensation. However, the cost of such equipment could be substantial.

5 TESTS WITH BENTONITE SLURRIES

The same bottles that were used for the water tests were used for the assessment of the concentration of four bentonite slurries. Bentonites from two suppliers were used and for each, slurries were prepared at concentrations of 40 and 50 kg bentonite per cubic metre of slurry. Weighing of mix components was to an accuracy of $\pm 0.02\%$. The estimated bentonite concentrations from the density measurements are shown in Table 4. For the estimation of these concentrations, water densities appropriate to the test temperature were used in the calculations which followed Eq. 2 and for both bentonites the grain density was assumed to be 2.28 g/ml . Despite the temperature correction it can be seen from Table 4 that concentration errors were in the range -0.8 to $+1.6 \text{ kg/m}^3$.

Table 4. As-mixed and estimated bentonite concentrations in kg/m^3

Bentonite	As-mixed bentonite concentration kg/m ³	Concentration estimated from measured density	
		Bottle 1	Bottle 2
A	40	41.0	41.6
A	50	51.4	50.8
B	40	40.3	41.4
B	50	50.3	49.2

Curiously seven of the eight assessed concentrations were above the as-mixed concentration suggesting that air bubbles were not significantly affecting the slurry density and/or significantly too low a density had been used for the bentonite powder – more research is needed on this.

If it is assumed from the water trials that a density resolution of ±0.0005 g/ml can be achieved (with temperature compensation for volume change of the water), then from Table 1 the bentonite concentration resolution should have been ±0.9 kg/m³. The slurry results were slightly outside this range.

Realistically from the results in Table 4, bentonite concentrations derived from such density measurements should not be assumed to better than ±2 kg/m³ under laboratory conditions and this in itself requires that an appropriately accurate value is available for the density of the bentonite powder as received (or can be calculated from a known dry grain density G_s and moisture content for the bentonite as received) and also that the volume of the bottle has been determined at a temperature comparable to that of slurry tests – though the error is small as the coefficient of expansion of glass is small in this context.

5.1 Immediate availability of concentration results

A problem with density measurements by weighing a fixed volume of fluid is that a calculation is required to obtain the result and a site laboratory is not an environment conducive to accurate calculations. In contrast, the mud balance which although of poorer resolution, does give a direct reading.

The problem can be avoided by preparing a look-up table showing density by weight of the fluid-filled container. This table prepared using the volume of the bottle determined from its weight empty, its weight full of water at a known temperature and the density of water at this temperature (the volume need be determined only once but checked regularly, say, weekly to ensure there has been no damage, especially to the stopper). Full container weights can be presented for density increments of, say, 0.005 g/ml.

6 DETERMINATION OF CONCENTRATION BY MOISTURE CONTENT MEASUREMENT

As demonstrated above, the determination of bentonite concentration in fresh bentonite slurries by density measurement requires careful measurements best undertaken under laboratory conditions. For checks on mixer batching accuracy, it is suggested that bentonite concentration should be determined by moisture content determinations carried out according to standard soils laboratory test procedures. However, when converting moisture content to bentonite concentration, it is necessary to allow for the moisture in the bentonite as received and the contribution of this water to the evaporable water content of the resulting slurry. If the moisture content of a bentonite powder as received is m_b and that of the resulting slurry is m_{sl} , then the concentration C_w of bentonite at its as-received moisture content by weight of mix water added is:

$$C_w = \frac{(1 + m_b)}{(m_{sl} - m_b)} \quad (7)$$

and if C is the concentration of bentonite at its as received

moisture content as mass per unit volume of slurry, then:

$$C = \frac{C_w G_s \rho_w}{C_w + G_s} \quad (8)$$

To test the concentration resolution that could be achieved by moisture content measurement, slurries were prepared at three concentrations with two duplicates of each slurry. These tests gave concentrations repeatable to ±1 kg/m³.

7 CONCLUSIONS

This paper has shown that accurate determination of the concentration of solids in slurries is challenging. For bentonite, density measurement with a spring-top glass bottle can achieve a realistic concentration resolution about ±2 kg/m³ in the laboratory. Determinations by moisture content can give better repeatability of ±1 kg/m³ but this requires an oven, drying for at least 24 hrs and possibly a correction for the dissolved solids content of the mix water. However, usefully it does not require any assumption about the grain density of the bentonite.

For day-to-day control of the as-mixed solids concentration of fresh bentonite slurries, it is suggested that density is checked with a fixed-volume container such as a spring-top bottle. This should give a readily achievable on-site resolution of 0.0025 g/ml or for bentonite about 4.4 kg per cubic metre of slurry. This will detect significant batching errors. However, if the actual as-mixed concentration is required, laboratory moisture content determinations can give better accuracy.

Little purpose is served by using the mud balance for the day-to-day management of fresh bentonite slurry preparation. This device has a smallest scale division of 0.01 g/ml and hence a resolution of no better than 17.8 kg/m³. Note that the above discussion relates to the assessment of the concentration of fresh slurries. The mud balance has sufficient resolution for the control of used bentonite slurry density for which specifications may set a limit of perhaps 1.08 g/ml for a slurry for re-use.

The mud balance is not suitable for the control of polymer support fluids either freshly prepared or when being checked for re-use. Density is not a suitable parameter for fresh polymer fluid concentration control as polymer powder is of near water density and dissolves to form a solution which, at the concentrations typically used for excavation-support fluids (≈1 kg/m³), produces a solution of density so close to that of water that concentration cannot be determined by on-site density measurement. For polymer fluids for re-use, the authors recommend a maximum density of 1.02 g/ml and so again the mud balance is not suitable. A spring-top bottle is well suited for the control of these fluids as the spring-top also can prevent the self-siphoning problem which is commonly observed with polymer fluids prepared using partially hydrolysed acrylamides (PHPA). If a container is slightly overfilled with a PHPA fluid, as it overflows the long-chain polymer molecules draw fluid from the container partially emptying it. With a spring-top, the bottle can be filled to near the brim and the top then snapped on displacing the last air from the top of the bottle before any polymer fluid can self-siphon out.

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