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BULK VOLUME DETERMINATION USING THE SAND PYCNOMETRY METHOD WITH OTHER GRANULAR MATERIALS

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EXTENDED ABSTRACT

Il peso di unità di volume di un materiale solido esprime, come è noto, il rapporto tra il suo peso e il suo volume totale. Di norma la determinazione di questo rapporto si ottiene tramite metodologie che utilizzano, per la misura indiretta del volume, la bilancia idrostatica o il picnometro ad acqua. Queste tecniche di indagine non sono però applicabili nel caso in cui si debbano analizzare dei solidi porosi e permeabili e/o che possano reagire fisicamente con l'acqua (es.: fenomeni di rigonfiamento, perdita di coesione, solubilizzazione, ecc.). Per ovviare a questo problema sono state proposte procedure di impermeabilizzazione tramite rivestimento dei campioni con paraffina (es.: *wax shrink-wrap immersion method* o *wax-immersion method*); tuttavia anche ciò comporta un certo grado di imprecisione legata a fenomeni di non perfetta aderenza della paraffina alle superfici del provino o alla formazione di bolle d'aria.

In alternativa, per la determinazione del peso di unità di volume può essere utilizzata la tecnica del picnometro a sabbia, un sistema rapido e di facile esecuzione che non richiede l'uso di attrezzature costose o particolarmente complesse.

Il metodo, pur trovando applicazione soprattutto nella determinazione delle proprietà indice di rocce o materiali lapidei naturali e artificiali, è anche particolarmente indicato nell'esame di fossili, reperti archeologici o altri oggetti che necessitino di essere preservati, in quanto si tratta di una prova non distruttiva e che non disturba o altera il materiale esaminato.

Anche l'utilizzo del picnometro a sabbia, tuttavia, può comportare imprecisione nei risultati in quanto, a causa delle dimensioni, del *sorting* e della forma irregolare delle particelle di cui la sabbia è costituita, non è facile ottenere un valore costante nel grado di addensamento di questo materiale granulare.

Con questo lavoro si sono voluti sperimentare materiali granulari alternativi alla sabbia, quali microsfere di acciaio e vetro, di differenti diametri, il cui impiego potesse eliminare tale problema o almeno contenere l'errore entro valori accettabili. Inoltre, per una più completa valutazione di come il grado di addensamento possa influenzare i risultati, le prove sono state condotte utilizzando ciascuno dei materiali granulari testati in tre differenti condizioni: senza costipamento, con costipamento ottenuto tramite vibrazione e con costipamento ottenuto tramite vibrazione e contemporanea applicazione statica di un sovraccarico verticale.

Per l'esecuzione della prova si è utilizzato come recipiente a volume costante un contenitore di alluminio di forma cilindrica, con un'apertura nella parte superiore tale da permettere l'applicazione del sovraccarico per il costipamento del materiale. Il volume di questo contenitore è stato determinato con estrema precisione pesando il contenuto d'acqua necessario al suo riempimento. È stato quindi determinato il peso di unità di volume di ciascun materiale granulare scelto in ciascuna delle tre condizioni descritte, riempiendo il contenitore e livellandolo accuratamente con una spatola metallica.

Per poter valutare quale fosse il materiale granulare più idoneo a raggiungere l'obiettivo prefissato si è proceduto a determinare il peso specifico o il peso di unità di volume rispettivamente di 10 cristalli o aggregati di cristalli privi di porosità e di due campioni di minerali e rocce di forma geometrica semplice (cubo). Tale scelta è stata fatta in quanto tutti i cristalli hanno peso specifico ben noto in letteratura, mentre per i campioni cubici il peso per unità di volume è stato determinato con precisione attraverso una bilancia analitica e l'accurata misura delle loro dimensioni tramite calibro digitale. Ciò permette di avere un riscontro immediato sulla precisione dei risultati ottenuti.

Per ciascuno dei campioni selezionati è stato determinato prima il peso proprio quindi i pesi complessivi del contenitore di alluminio contenente il campione e riempito con il materiale granulare in ciascuno dei tre stati di addensamento, per un totale di oltre 1000 pesate. È stato così possibile calcolare i pesi specifici/pesi di unità di volume dei campioni analizzati per ciascun materiale granulare e per ciascuna metodologia di addensamento.

Dai risultati è emerso che la migliore soluzione per determinare il volume dei campioni con il metodo del picnometro, superando nello stesso tempo le limitazioni connesse all'impiego dell'acqua, è quella di utilizzare le sfere d'acciaio in assenza di costipamento. L'utilizzo di questo materiale, che rappresenta quello a più alto peso specifico tra quelli testati, ha permesso di ottenere valori di peso specifico/peso per unità di volume decisamente più attendibili rispetto a quelli determinabili con l'impiego della sabbia.

ABSTRACT

It has always been difficult to determine precisely the bulk volume of irregularly shaped natural and artificial solid objects such as rocks, bricks, concrete and mortar, considering their porosity, permeability and potential interaction with water.

The sand pycnometry method could fix these issues, but it can also lead to imprecision due to the irregular shape and the degree of compaction wide variability span of sand grains.

For this reason, we tested granular materials other than sand, in particular steel and glass beads, in order to identify the most suitable procedure in conducting laboratory bulk volume determinations. The effect of packing on the fine granular materials was investigated using three different compaction conditions: no compaction, compacted by vibration alone, and compacted by vibration and simultaneous plugging.

According to the results, the heaviest, uncompacted steel bead granular material was identified as the best solution among those studied.

***Keywords**: bulk volume, irregularly shaped porous solid objects, non-destructive test, sand pycnometry method, granular materials*

INTRODUCTION

Unit weight γ is one of the fundamental physical properties of matter and represents the weight per unit volume of a material. It is well established that, depending on the context and the parameters considered, different types of unit weight of geological materials can be determined using geotechnical tests in the laboratory. In particular, if the material is dry, the dry unit weight is:

$$\gamma_d = P/V$$

where P corresponds to the weight of the solid phase and V is the bulk volume, i.e. the geometric volume occupied by the whole rock, including both the volume of the solid phase and the volume occupied by pore spaces and cracks.

Knowledge of the bulk volume of rock and consolidated sediment samples is important because it is one of the parameters necessary for calculating some fundamental properties of interest to geoscientists as, for example, porosity.

The common way to determine the dry unit weight of an object is through an accurate measure of its bulk volume, which can be performed using different methods.

In non-consolidated soils characterised by poor cohesive strength, the determination of this parameter can be easily achieved in undisturbed samples obtained by means of a thin-walled sampling tube, where the specimen volume perfectly matches the inner volume of the sampler.

However, this method cannot be applied to more competent materials such as rocks. In this case, the linear dimension of the specimen, modelled in a simple geometric shape (e.g., cubic or parallelepiped or a cylindrical rock core), can be measured with a calliper (Crawford, 2013). Nevertheless, achieving an intact and/or perfectly geometrical specimen cannot be guaranteed.

For irregularly shaped samples, measurement of bulk volume poses numerous challenges. Cutting an irregular rock can produce a shape whose volume can be easily calculated geometrically, but it has always been desirable to measure the volume of a sample without altering or destroying it during the process.

Alternative procedures for the volume definition involve the measure of the amount of fluid displaced by a specimen immersed therein (Kushelevski, 1975; ASTM, 2015a).

In the past mercury was used as the fluid (ASTM D427-04): it is no wetting so it does not enter pores of less than 200 μm in the sample or cause loosely consolidated samples to disaggregate, and its high specific gravity allows small differences in volume to be distinguished by weighing. However, due to the high toxicity of mercury, this procedure is not used anymore.

Another fluid used in similar methods is water. Its use, though, could cause detectable problems, especially if it is applied to samples that are porous, permeable or reactive to water; hence precautions must be taken.

For instance, waterproofing techniques are mandatory (e.g., the wax shrink-wrap immersion method and the wax-immersion method ASTM, 2015b, 2018). The coating prevents the water from entering pores or disaggregating the samples, but variations in the thickness of the coating and the volume of the coating itself can produce significant errors in determination of the bulk volume of small samples. Therefore, the standard of this method requires at least five representative samples or a relatively large mass of material to be coated. When only a single, small sample is available, the errors in determination of bulk volume are unacceptably large and contamination by the wax can make the sample useless for later chemical analyses.

As it is well known, the pycnometer method (ASTM, 2014) is the most commonly used laboratory technique to define the specific gravity of a disaggregated specimen, which can be achieved by determining the volume of the specimen through the weight difference of a constant-volume flask (the pycnometer) filled with water only, and with water and the specimen inside:

$$V = (P_1+P_2-P_3) / \gamma_w$$

where P_1 is the weight of the pycnometer with water; P_2 is the weight of the specimen; P_3 is the weight of the pycnometer filled with water and the specimen and γ_w is the water density.

As water has a constant, specific weight up to 30°C (1.00 g/cm^3), it can be disregarded.

The same method can be applied using sand in place of water to define the volume of irregularly shaped porous/permeable specimens or reactive to water itself (Yeager & Slowey, 1996). However, if the filler is sand, its unit weight, being different from 1.00 g/cm^3, must be determined and considered. It should

be also take into account that sand cannot be featured through univocal values, due to not only the various specific gravity values of the grains of which it is made up, but also because of the variability of sorting and the irregular shape of sand grains; this affects its degree of compaction (porosity) and, consequently, the accuracy of the results.

In order to minimise the variables affecting the sand filler, like slight differences in the composition of the grains as well as irregular shape and sizes, a new method developed by Consolmagno & Britt (1998), Consolmagno *et alii* (1998) and Balco & Stone (2003) based on an idea of Sheldrick (1984), and subsequently rechecked and confirmed by Macke, Britt & Consolmagno (2010), was proposed. In this method, the bulk volume is measured using small (250-425 μm size) glass beads as the displacement medium in place of the fluid and characterised by the same regular shape and dimension. The glass beads are nonreactive, and clearly do not penetrate into the cracks and pores. This method of bulk volume measurement can have accuracy as high as 1% and a precision of 1.2% (Wilkison & Robinson, 2000).

The Archimedean glass bead method for determining bulk density was tested using well characterized, zero-porosity quartz and topaz samples to determine the systematic error (Macke *et alii*, 2010). Systematic error varies according to bead size, container size and settling method, but in all cases is less than 3%, and generally less than 2%. While measurements using larger containers (above 150 cm^3) exhibit no discernible systematic error but much reduced precision, higher precision measurements with smaller containers exhibit systematic error. For all methods, reliability of measurement is severely reduced for samples below 5 cm^3, providing a lower-limit selection criterion for measurement of samples.

It is worth noticing that this method is not commonly described in any relevant manuals; thus, this research sought to analyse granular materials other than the sand filler, with the aim of identifying the most suitable for determining γ_d in natural or artificial samples of different shapes and natures.

METHODOLOGY

As discussed above, the sand pycnometry method is based on the same principles of the water pycnometer approach, but it uses a cylindrical container instead of a glass-specific gravity bottle. The determination of the unknown volume (incognita) is obtained with a reciprocal γ-formula, $V=P/\gamma$, where P is the weight of a volume of filler equivalent to the volume of the specimen (Fig. 1).

The unit weight $\gamma=P/V$ is therefore obtained using the following equation:

$$\gamma=P_2/[(P_1+P_2-P_3)/\gamma_{fil}]$$

Since in this case the filler is sand, the term γ_{fil} is different from 1.00 g/cm^3 and it must be retained in the formula. The unit weight of the granular material used to fill the volume of the container is not identified by its specific gravity, but through its dry unit weight. This was obtained by weighing the material inside the container before the test. Nevertheless, as it is difficult to realise the same degree of sand compaction every time, a correct determination may not always be achieved. Accordingly, we tested granular materials other than sand, such as steel and glass beads (Tab. 1). The choice of testing such materials arose from the need to optimise the sorting and homogenise the sphericity and roundness of the grains.

Type of material	*Particle diameter (μm)*	*Label*
Steel beads	500 ÷ 700	Steel
Glass beads	200 ÷ 300	Glass 23
Glass beads	400 ÷ 800	Glass 48

Tab. 1 - Granular materials other than sand chosen for tests

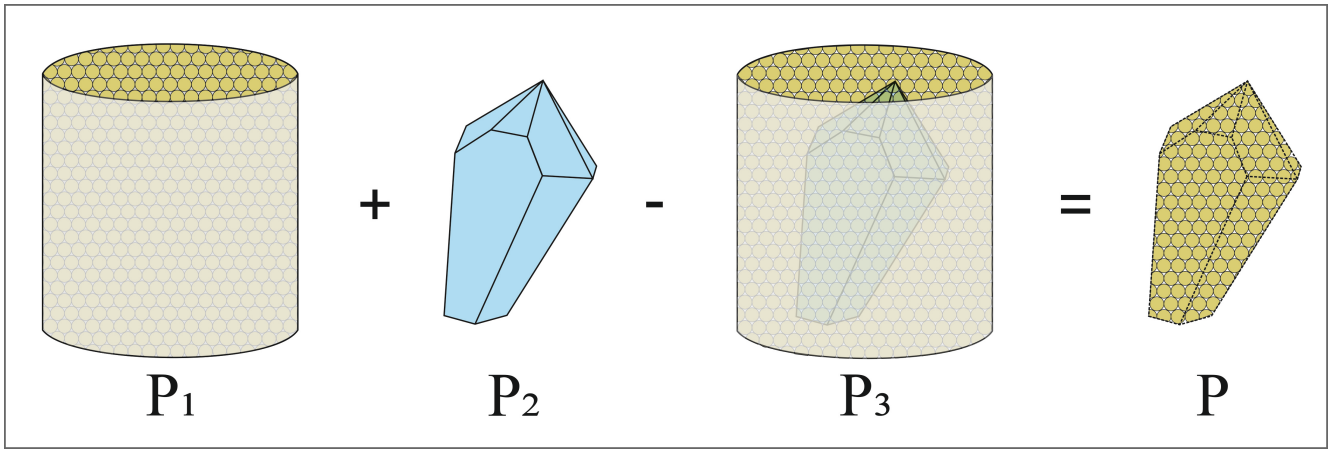

Fig. 1 - The sand pycnometer method: determination of the weight of filler volume (P) that is equivalent to the volume of a specimen. P1: weight of the pycnometer (in this case, a cylindrical container) with filler only; P2: weight of the specimen; and P3: weight of the cylindrical container with filler and the specimen

The research also examined how the grains of the materials can arrange in the space around the sample inside the containers. The effect of such a packing was therefore investigated using three different compaction conditions for the granular materials:

- no compaction;
- compacted by vibration alone;
- compacted by vibration and simultaneous plugging.

Tests were carried out on: compact mono-mineral crystals, where the lack of porosity makes the unit weight equal to their specific gravity, and crystal or rock specimens with a simple geometrical shape.

To this end, we chose different samples with a well-known, specific gravity (or with a dry unit weight that could be accurately calculated with a precision balance and a calliper), and with sufficient heterogeneity in their shape, weight and size (Tab. 2).

The laboratory tools (vibrometer, spatula, scale, etc.) were carefully chosen, and the volume of the aluminium container used for all the weighing was thoroughly pre-determined by weighing its equivalent volume of water.

The temperature in the laboratory was constantly kept close to 23°C in order to avoid possible water-density fluctuations or further inaccuracies on other measures.

Single crystals	***Crystals Lump***	***Geometric specimens***
Quartz	Galena	Sphalerite / Regular cube
Calcite	Pyrite	
Beryl		Calcareous rock / Regular cube
Apophyllite		
Tourmaline		
Fluorite		
Gypsum		
Icelandic Spar		

Tab. 2 - Geo-materials of a well-known, specific gravity/dry unit weight selected for testing

RESULTS

The first step was to calculate the granular materials' γ_{fil} values, which were obtained by filling up and levelling the aluminium container at the rim and dividing the obtained weight of each material (in its various compaction degrees) by the container volume, determined as explained above. The average results are shown in Table 3.

An important limit recognised during the pouring of the materials was the difficulty in filling every void between the container and the specimen (Fig. 2). The friction angle of the

	Net weight (g)			γ_d (g/cm^3) *(for a volume of 194.08 cm^3)*		
	uncompacted	vibrated	vibrated & plugged	uncompacted	vibrated	vibrated & plugged
SAND	288.93	327.14	320.87	1.49	1.69	1.65
STEEL	876.32	937.00	944.34	4.52	4.83	4.87
GLASS48	294.41	316.28	314.64	1.52	1.63	1.62
GLASS23	286.03	303.11	303.96	1.47	1.56	1.57

Tab. 3 - Dry unit weight mean values (γ_d) of the granular materials

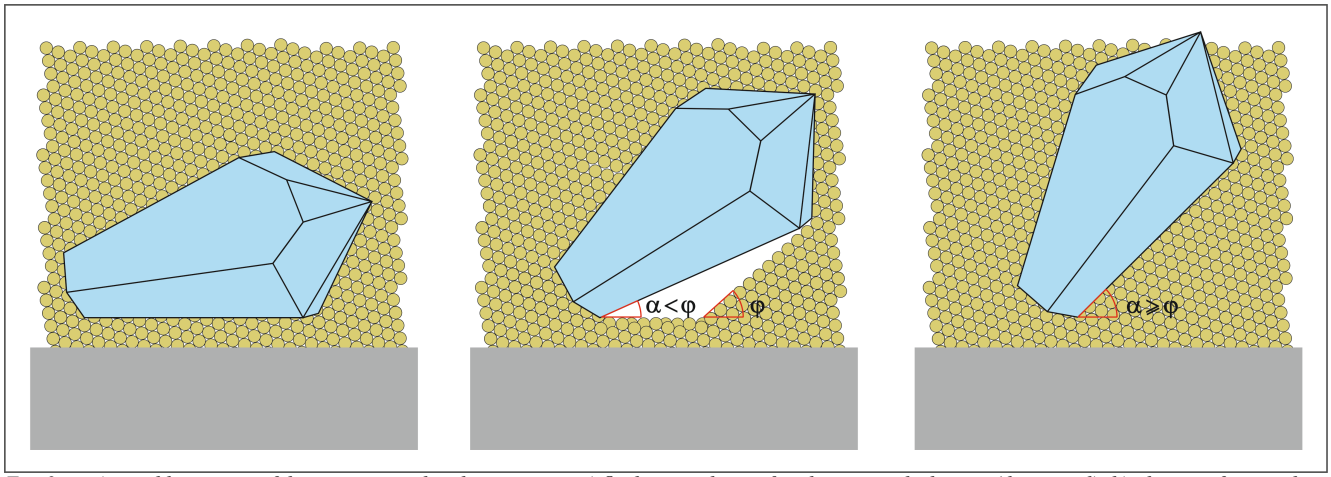

Fig. 2 - A possible position of the specimen within the container: a) flat base, with a surface leaning to the bottom (theoretical); b) tilting surfaces with an angle less than the friction angle (possible voids under the sample); and c) tilting surfaces with an angle of approximately 45° (optimum)

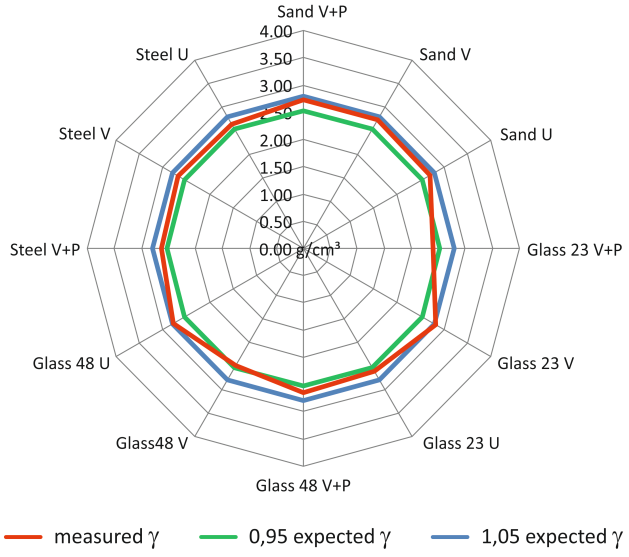

Fig. 3 - Distribution of the quartz-measured specific gravity (γ) (red line) compared with the ±5% expected γ range (blue and green lines)

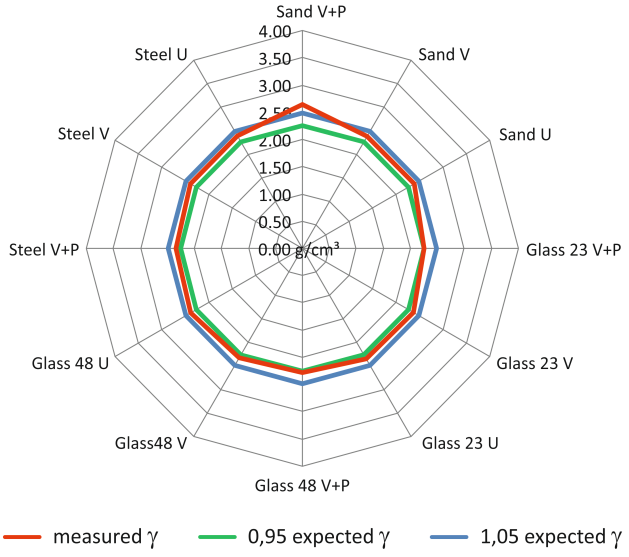

Fig. 4 - Distribution of the Apophyllite-measured specific gravity (γ) (red line) compared with the ±5% expected γ range (blue and green lines)

Granular material	Friction angle (°)
SAND	27
GLASS 48	27
STEEL	26
GLASS 23	24

Tab. 4 - Friction angle of the granular materials

various granular materials was therefore identified experimentally (Tab. 4). The higher the friction angle is, the greater the risk of failing to achieve satisfactory filling around the sample. The solution of placing the sample above a first layer of granular material, thereby tilting its surfaces close to 45° (or at least more than the friction angle of the material), was identified as the best approach; in this way, the presence of hidden sectors not reached by the filling material was avoided.

As shown in Table 4, the steel beads and "Glass 23" were the granular materials with lower friction angle.

After these preliminary steps, a multiple series of weights with a precision balance were performed on the 12 selected samples and the mean value was calculated for each of them.

In view of the purpose of this research, a series of weights were performed also with the container filled with the various granular materials, compacted through three different procedures: no compaction (U); compacted by vibration (V); and compacted by vibration and plugging (V+P). A high number of tests were carried out and the mean values were calculated for each specimen and for every type of granular material and compaction method (Tab. 5).

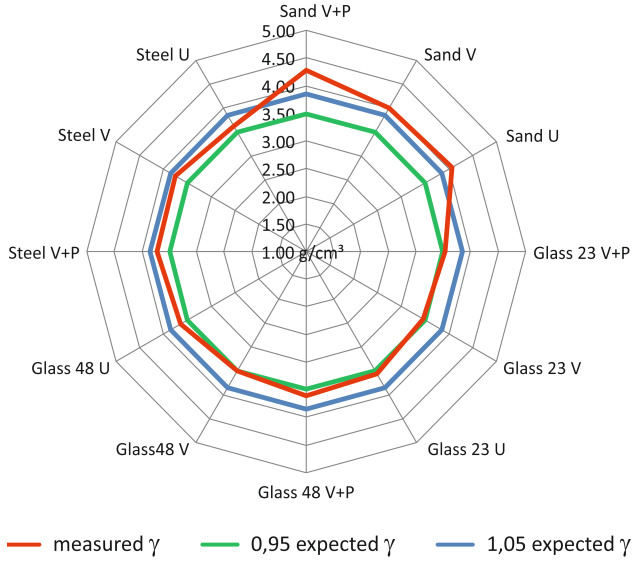

Fig. 5 - Distribution of the Sphalerite-measured specific gravity (γ) (red line) compared with the ±5% expected γ range (blue and green lines)

Several average values are shown in figures 3, 4 and 5 through spider diagrams. Data from quartz, apophyllite and sphalerite were evaluated as the most representative. According to the results, sand is not particularly reliable for γ values comparable with literature data; and there are similar unreliable outcomes with vibrated (V) and vibrated and plugged (V+P) "Glass 23" and with vibrated (V) "Glass 48".

The next step was the verification of the best granular material

	Expected γ g/cm³	Sand V+P	Sand V	Sand U	Glass23 V+P	Glass23 V	Glass23 U	Glass48 V+P	Glass48 V	Glass48 U	Steel V+P	Steel V	Steel U
Quartz	**2.66**	2.73	2.73	2.71	2.40	2.81	2.61	2.64	2.48	2.77	2.63	2.67	2.64
Calcite	**2.71**	3.94	3.85	3.05	2.04	3.01	2.65	2.70	2.50	2.85	2.65	2.97	2.78
Beryl	**2.78**	3.30	2.60	2.80	2.42	2.77	2.64	2.57	2.57	2.79	2.61	2.72	2.76
Apophyllite	**2.37**	2.63	2.38	2.39	2.24	2.38	2.36	2.30	2.33	2.38	2.34	2.40	2.39
Tourmaline	**3.00**	7.03	4.86	3.47	2.69	3.20	3.07	2.57	2.82	3.25	3.21	3.22	3.19
Galena	**7.57**	17.26	18.85	2.57	5.90	8.02	6.79	6.76	6.02	7.11	8.59	9.51	7.56
Pyrite	**4.85**	5.39	5.40	1.76	5.05	5.04	4.94	4.86	4.94	4.97	5.15	5.22	4.94
Fluorite	**3.37**	3.43	3.33	3.20	3.00	3.20	3.14	3.19	3.06	3.17	3.20	3.21	3.14
C.9 (Calcareous rock. regular cube)	**2.66**	2.81	2.72	2.66	2.63	2.63	2.64	2.65	2.59	2.66	2.64	2.68	2.63
Gypsum	**2.32**	3.67	3.24	2.66	2.20	2.54	2.14	2.02	2.09	2.35	2.13	2.34	2.39
Sphalerite (regular cube)	**3.67**	4.28	4.00	4.06	3.52	3.45	3.56	3.61	3.50	3.64	3.72	3.74	3.61
Iceland Spar	**2.71**	4.10	3.43	4.00	2.03	2.46	2.48	2.39	2.23	2.55	3.02	3.46	2.65

Tab. 5 - Specific gravity/dry unit weight expected values of the analysed samples and the mean of the values obtained with the sand pycnometry method. Results with a ±3% deviation from the expected γ values are shown on the green background; results with a ±5% deviation from the expected γ values are portrayed on the yellow background

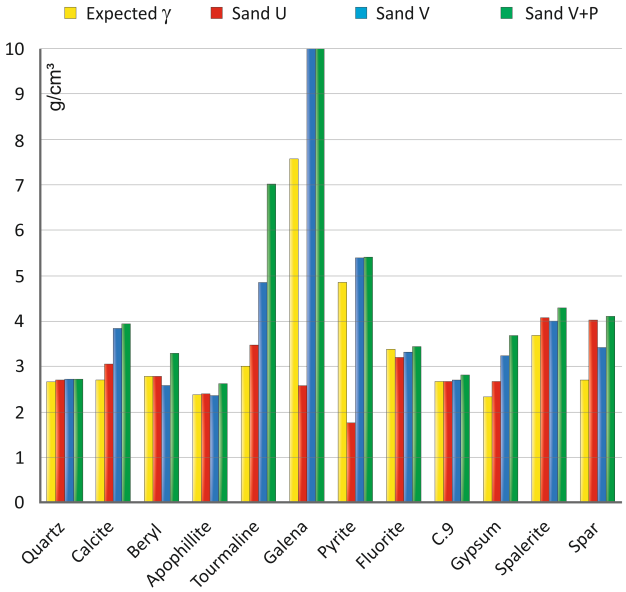

Fig. 6 - Specific gravity/dry unit weight values determined using sand grains (U for "no compaction", V for "vibrated", and V+P for "vibrated and plugged")

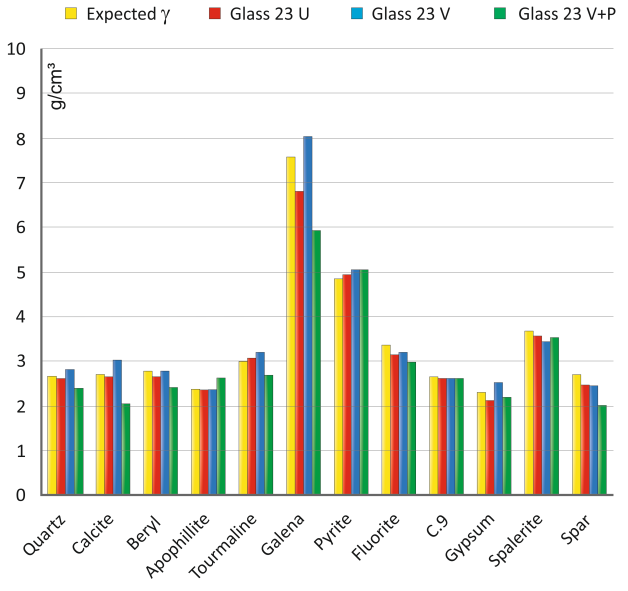

Fig. 7 - Specific gravity/dry unit weight values determined using Glass23 beads (U for "no compaction", V for "vibrated", and V+P for "vibrated and plugged")

and compaction method able to reproduce specific weights of the tested mineral samples closest to the literature values or resulting from weight measures and volumes determined using a calliper.

The obtained γ values are shown in figures 6, 7, 8 and 9.. The four diagrams are differentiated by the granular material used and the three compaction methods. The results are compared with the γ values expected from the literature: the yellow bars refer to the expected γ; the red bars record the γ values obtained with no compaction (U); the blue bars are the γ values achieved after vibration alone (V); and the green bars indicate the γ values obtained by vibration and plugging (V+P).

The γ values with the best fit are those obtained with the steel beads, with only the exceptions of Galena and Spar. This point is probably related to the high specific weight of steel beads, which enhances the compaction process.

Changing perspective, the three compaction methods are set out in figures 10, 11 and 12. In this case, the expected γ values are recorded as yellow bars; those obtained with the steel beads are the grey bars, those with the "Glass 48" spheres are the green bars, those with the "Glass 23" beads are the blue bars, and those with the sand are the red bars. As can be noted, the no compaction method (U) produces the best values (i.e., the values closest to the expected γ).

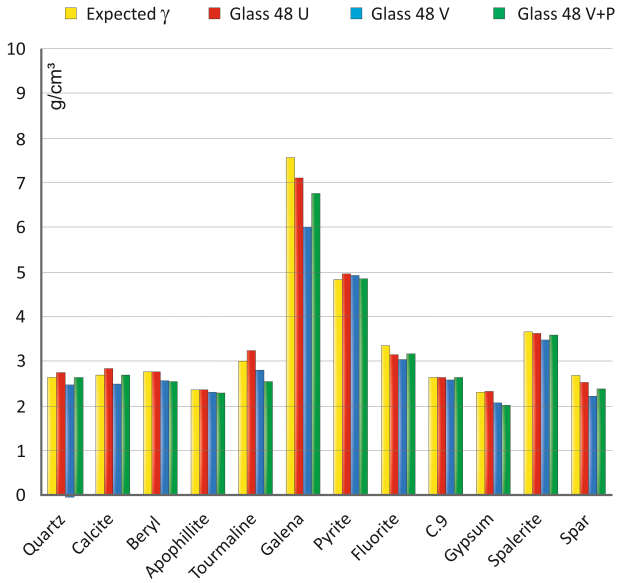

Fig. 8 - Specific gravity/dry unit weight values determined using Glass48 beads (U for "no compaction", V for "vibrated", and V+P for "vibrated and plugged")

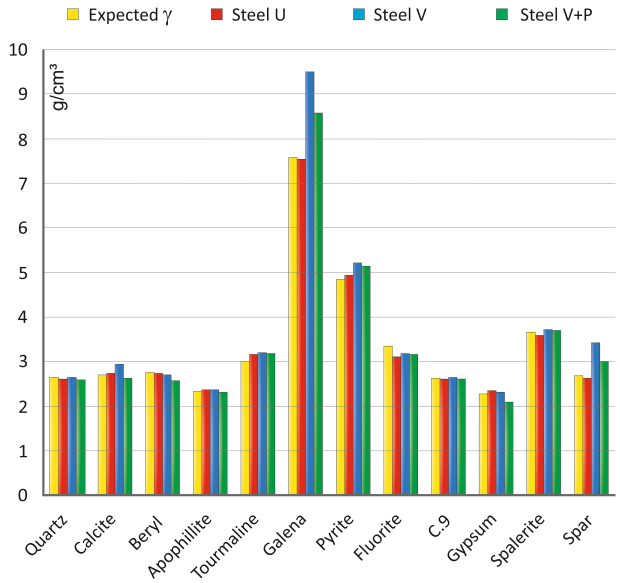

Fig. 9 - Specific gravity/dry unit weight values determined using Steel beads (U for "no compaction", V for "vibrated", and V+P for "vibrated and plugged")

DISCUSSION

The reported data clearly highlight an enhanced accuracy of the measurements and, at the same time, point out the numerous flaws of the sand when it is used as a filler. The irregularity of the shape of the sand grains, their various natural composition and the different dimensions do not allow the filler to reach a sufficient compaction level.

Among the tested compaction methods, the "No compaction" one showed the best results both with the glass and steel beads. This point can be explained by the fact that, without vibration or plugging, but just pouring the displacement medium, a more comparable compacting condition between the first phase of determining the γ_{fil} of the filler and the second one, when the sample unit weight is determined, can be achieved. If the displacement of the beads during the two phases is similar, there are better chances of having a comparable compacting condition before and after inserting the sample and, therefore, better chances to obtain reliable results. This finding is important, because the compaction process is time consuming: each specimen needs to be vibrated for some minutes to achieve the correct degree of compaction. If this time shall be multiplied by the high number of tests required to calculate a reliable mean value, the long duration of the process in relation to each sample is evident. This amount of work is, however, reduced if there is no need to compact the granular material.

Nevertheless, it must be noted that the use of uncompacted granular material requires very high precision and accuracy. Specifically, any sudden vibration may compact the granular material in an unexpected manner, thereby affecting the following results.

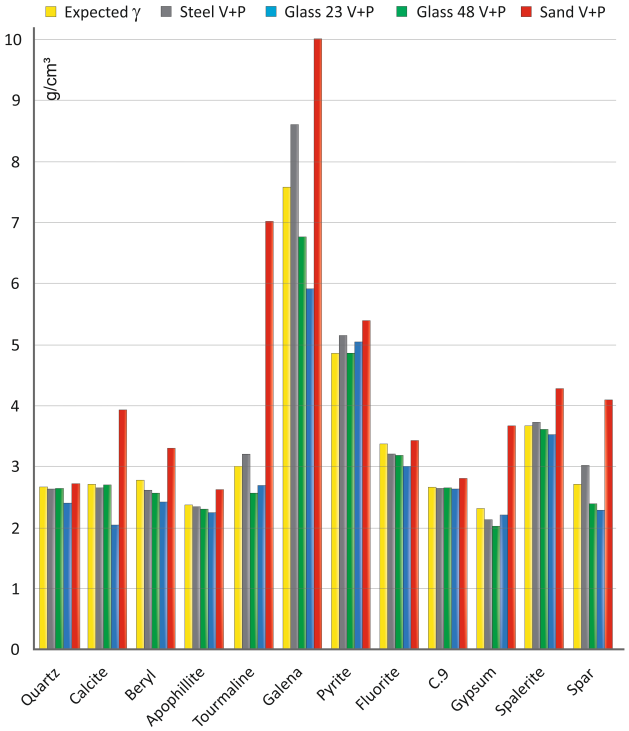

Fig. 10 - Specific gravity/dry unit weight determined with no compaction of the filler (U)

As described in figures 10, 11 and 12, the steel beads showed the best results. The high level of accuracy is probably caused by the material of which the beads are made, rather than the different

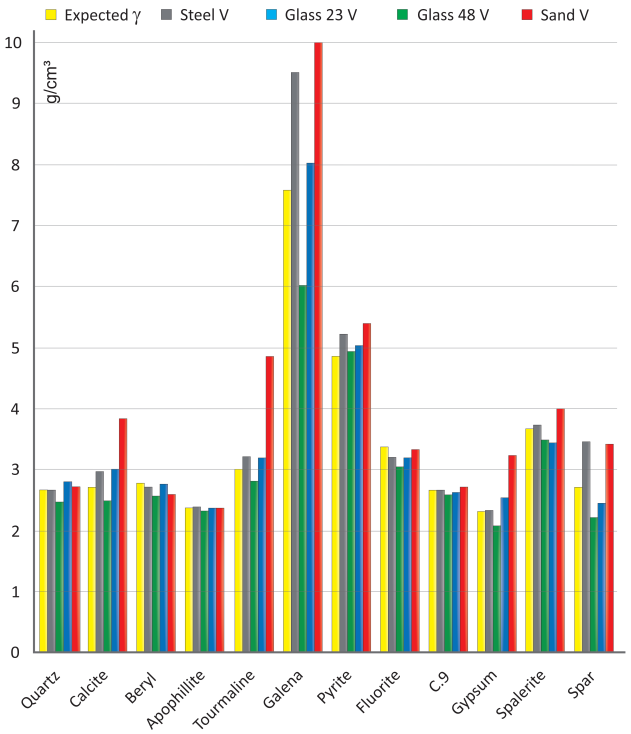

Fig. 11 - Specific gravity/dry unit weight determined compacting filler through vibration only (V)

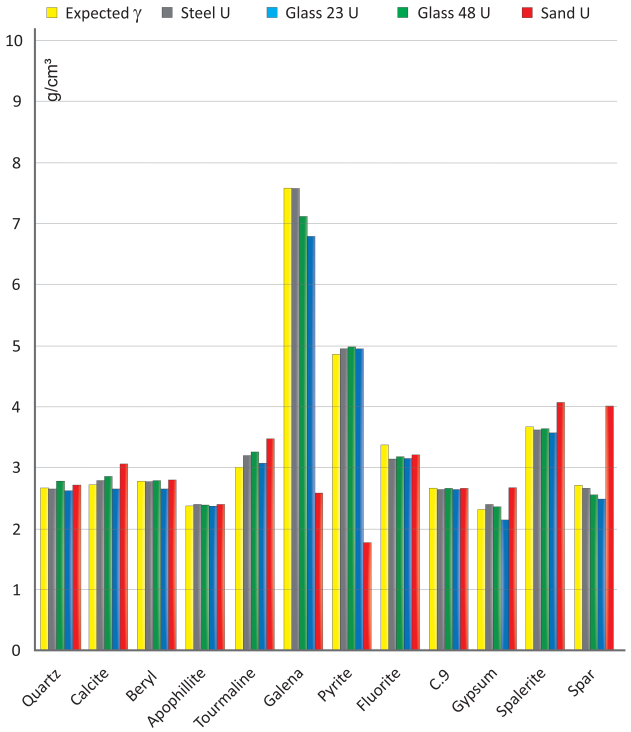

Fig. 12 - Specific gravity/dry unit weight determined with no compaction of the filler (U)

dimensions with respect to the glass ones. The higher weight of the steel beads is likely the reason why it is possible to obtain a more uniform compaction, which leads to more accurate bulk density values.

Another important parameter was the percentage of the total volume occupied by the sample compared with the displacement medium (filler) and the container volume. In this respect, it should be also noted that if the sample volume percentage is too low, it is difficult to accurately measure a difference between the beads volume with and without sample. On the other hand, if the sample volume percentage is too high, there may not be enough granular material (beads) to completely surround the sample.

CONCLUSIONS

The aims of this work were to: i) use the sand pycnometry method to calculate the unit weight of selected samples; ii) test alternative granular materials; iii) identify the most suitable procedure for laboratory tests on natural samples.

Specifically, the sand pycnometry method was assessed by comparing sand to three other granular materials (steel and glass beads of different sizes) used as fillers, and by evaluating three different compaction methods during the weighing of samples, having different compositions and shapes.

We selected 12 natural samples for the practical tests. Proper laboratory conditions and tools (e.g., vibrometer, precision balance and aluminium cylindrical container) were, respectively, maintained and selected.

Considering all the variables described above, the samples were weighed to obtain their γ values, and the following findings were inferred:

the container must be regularly cylindrical and as close as possible to the sample size;

the sample must be placed in the container over a first layer of granular material, with an approximately 45° tilting angle (or with an angle that is at least higher than the granular material friction angle);

the most suitable granular material is 500-700 μm steel beads;

the closest values were obtained when there was no compaction.

Although the proposed procedure requires very high precision and sensitivity, it can be considered an improvement of the sand pycnometry method, both in terms of result accuracy and procedure time reduction.

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