Original Article

Relationship between solids density and the PFD80 transportable moisture limit of hematite-rich and goethite-rich iron ore fines

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Abstract

The Transportable Moisture Limit (TML) is a relevant regulatory parameter for the mining industry, as it prevents the occurrence of mineral cargoes liquefaction aboard vessels, and represents a regulatory/technical restriction on shipments, requiring accurate moisture content control by the shipper. Determined through laboratory tests, the TML value varies with ore characteristics. This study investigated experimentally the influence of solids density on the TML of hematitic iron ore fines, using the Modified Proctor/Fagerberg Test for Iron Ore Fines, which defines TML as the moisture content at 80% saturation after compacting the test material applying a compaction energy of 27.59 kJ/m^3. The results showed that, for the same void ratio at 80% saturation, which is governed by a number of ore characteristics as particle size distribution, the lower the solids density, the higher the TML, which implies that ores with the same volumetric relationship between solids, water and voids, but with different mineralogical compositions, will have different TML values simply due to their different solids densities.

Keywords: Iron ore fines; Transportable Moisture Limit; Solids density.

Relação entre densidade dos sólidos e o TML PFD80 de finos de minério de ferro hematíticos e goethíticos

Resumo

O Limite de Umidade Transportável (TML) é um parâmetro regulatório relevante para a indústria de mineração, pois previne a ocorrência de liquefação de cargas minerais a bordo de navios, representando uma restrição regulatória/técnica para embarques, exigindo controle preciso da umidade pelo embarcador. Determinado por meio de ensaios laboratoriais, o valor do TML varia de acordo com as características do minério. Este estudo investigou experimentalmente a influência da densidade dos sólidos no TML de finos de minério de ferro hematíticos e goethíticos. O Ensaio de Proctor/Fagerberg Modificado para Finos de Minério de Ferro foi utilizado, o qual define o TML como a umidade correspondente a 80% de saturação após a compactação do material aplicando-se uma energia de compactação de 27,59 kJ/m^3. Os resultados mostraram que, para um mesmo índice de vazios em 80% de saturação, o qual é governado por diversas características do minério, como a distribuição granulométrica, quanto menor a densidade dos sólidos, maior o TML. Isso implica que minérios com a mesma relação volumétrica entre sólidos, água e vazios, mas com diferentes composições mineralógicas, terão valores de TML diferentes, simplesmente devido às diferentes densidades de sólidos.

Palavras-chave: Finos de minério de ferro; Limite de Umidade Transportável; Densidade dos sólidos.

1 Introduction

1.1 The Transportable Moisture Limit

Established in 1948, the International Maritime Organization (IMO) is the specialized agency of the United Nations responsible for maritime safety, and oversees international regulations for marine cargo transport. One of its main legislations is the International Maritime Solid Bulk Cargoes (IMSBC) Code, adopted in 1965, which ensures safe practices for solid bulk cargoes carriage on board vessels, including ores [1].

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The IMSBC Code contains individual schedules for various types of solid bulk cargoes, listing their characteristics, safety guidance and handling procedures to be followed, and became mandatory for SOLAS Convention signatory nations in 2011. The document categorizes solid bulk cargoes into three groups concerning the risk associated with handling and transporting by sea:

- Group A: Cargoes which may liquefy.
- Group B: Cargoes which possess a chemical hazard.
- Group C: Cargoes which are neither liable to liquefy nor to possess a chemical hazard.

According to the Code, solid bulk cargoes which may liquefy are cargoes that contain a certain proportion of fine particles and moisture, which can liquefy if loaded with moisture content above the Transportable Moisture Limit (TML) [1]. In summary, liquefaction is the phenomenon in which a wet granular material changes from a solid state to a fluid state due to an increase in pore water pressure and a reduction in the effective stress between the solid particles. In the context of the IMO regulatory framework, liquefaction of a solid bulk cargo is any moisture-related process that results in undesired cargo movement (shifting of the cargo within the hold), with the potential for loss of ship stability.

The TML corresponds to the maximum moisture content considered safe for the transportation of Group A mineral cargoes onboard vessels [1], a concept that dates back to the 1960s [2]. International regulations require, among other actions, that the cargo's moisture content remains below its TML during loading and during the voyage to ensure its safe transportation, preventing the occurrence of liquefaction. If the ore's moisture content is above the TML, the cargo cannot be loaded. The TML value is not fixed; it varies depending on the test method employed and on the characteristics of the tested material. According to the IMSBC Code, iron ore fines (IOF) with less than 35% goethite are categorized as Group A cargoes. The TML is determined using the Modified Proctor/Fagerberg Test for Iron Ore Fines (PFD80 test). It is defined as the moisture content in mass on a wet basis, corresponding to 80% saturation after compacting the test material under an energy of 27.59 kJ/m^3.

The moisture content at which 80% saturation is reached in a solid bulk material varies due to various factors, rendering TML a multi dependent complex variable [3,4]. Considering that the accurate moisture content control required by the legislation to the shipper is based on the TML value, it is crucial to comprehend the tested cargo characteristics that influence the variability of TML and to what extent the parameter may vary.

1.2 The solids density in the context of TML

The PFD80 method determines the TML through a dynamic compaction test. This method is based on the classic Proctor test but is calibrated to represent the compaction conditions of an ore heap in a vessel's hold, applying a compaction energy of 27.59 kJ/m^3. The moisture limit is defined at 80% saturation under this compaction condition.

The PFD80 TML represents the percentage relationship between the mass of water required to fill 80% of the void volume in the compacted ore and the total mass (ore+water). Intuitively, the TML shall be proportional to the void volume.

Ferreira et al. [3] demonstrated the positive relationship between TML and void ratio, developing a simple regression model for estimating TML based on this correlation. The authors also briefly addressed the theoretical relationship between TML and solids density. Ferreira and Lima [4] investigated the relationship between particle size distribution and TML, concluding that different size distributions imply different packing patterns of solid particles after the compaction test due to structural and interaction effects occurring between grains of different sizes, and each packing pattern will result in a different void volume. Therefore, TML is highly dependent on the particle size distribution of the tested material. According to the authors, the coefficient of uniformity can be used for representing numerically the particle size distribution for establishing a relationship with the TML. Considering the same type of material, the lower the coefficient of uniformity, the higher the TML.

When adapting the Proctor test to determine a moisture content limit for the maritime transport of mineral concentrates, Fagerberg and his team [5-7] opted to report the result in mass on a wet basis, as the common practice in the mining industry. Additionally, the authors presented the compaction curve in terms of void ratio (e) and net water content in volume (e_v) (Figure 1), which differs from the usual practice in Geotechnical Engineering of presenting the results using dry density and moisture content in mass on a dry basis. They justified this choice, stating that comparisons between test materials are facilitated if all pertinent data are referred to volume instead of weight [7]. With this configuration, the iso-saturation lines remain fixed on the graph, and only the compaction curve is variable. The TML is calculated using Equation 1, where $e_{v\,crit}$ corresponds to the e_v value for 80% saturation, which is the intersection point between the compaction curve and the 80% saturation line.

$$\boldsymbol{TML} = \frac{100\boldsymbol{e_{v\ crit}}}{100\boldsymbol{d} + \boldsymbol{e_{v\ crit}}} \qquad (1)$$

Since the variables on the graph are all volumetric, and TML is expressed in moisture content by mass, it is necessary to use the solids density (d) for this conversion in Equation 1. Several authors have highlighted the importance of accurately determining solids density for TML calculation [8-12], as it significantly impacts the test result.

Studies have investigated the sensitivity of the TML test result to solids density variations of the same sample, including the effects of measurement errors and variations in measurement methods [10]. However, to the best of the authors' knowledge, no published research has comprehensively

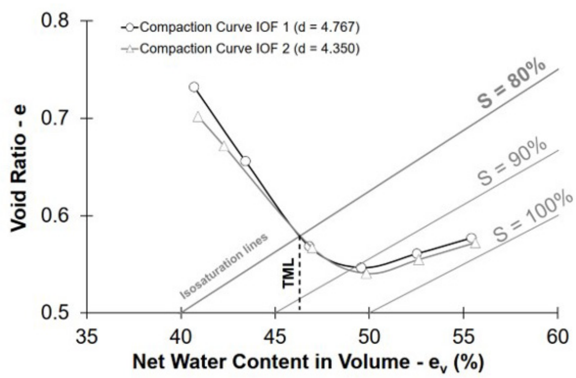

Figure 1. Examples of IOF Proctor/Fagerberg compaction curves.

evaluated the influence of the test material's solids density on the TML result. While extensive research in geotechnical literature explores relationships between soil characteristics and compaction test results, particularly optimal moisture content and maximum density, these findings are not readily transferable to TML determination. This is because TML, despite being a moisture content value, is by convention calculated differently than in soil mechanics applications, being also determined from a compaction curve defined by distinct parameters. Since mining personnel dealing with TML and moisture control might not have a background in Geotechnical Engineering, existing research on soil compaction, often written in different technical terms, may not be fully utilized. Therefore, research specifically tailored to TML applications is both needed and valuable.

The present study aimed to investigate experimentally the impact of the test material solids density on the TML of hematite-rich and goethite-rich iron ore fines. The IMSBC Code [1] outlines various methods for determining solids density to calculate TML, but this paper does not delve into the differences between the results obtained from these different methods.

2 Materials and methods

Eighty-six samples of various types of IOF were collected or generated in laboratory by blending or screening different ore materials. The size distribution of the samples was determined through wet sieving and laser diffraction of the material finer than 0.045 mm using a Malvern® Mastersizer diffractometer. The modal mineralogical composition was determined using reflected light microscopy. Fe content analyses were conducted via X-ray fluorescence using a Rigaku Simultix 12 spectrometer. Loss on Ignition (LOI) was determined by the weight loss after calcination at 1,000 °C for 1 hour in a muffle furnace.

Solids density (d) measurements were conducted using Helium pycnometry following the procedures outlined in the ASTM standard D5550-14 [13], employing a Quantachrome® Pentapycnometer. The test material was initially ground using a pulveriser ring mill to approximately 95% passing 0.075 mm. The specimen was then dried in an oven until a constant mass was achieved. After removal from the oven, the test sample was placed in a desiccator for the necessary time to cool to ambient temperature. The sample mass was determined using a precision balance, and the material was subsequently transferred to the gas pycnometer for volume measurement. For He pycnometer analyses, a cell with a volume of 131.7 cm^3 was utilized, filling approximately 3/4 of the cell with the sample. A pressure of 17 psi was applied to the equipment, with a purge time of 5 minutes, and three analyses were consistently conducted automatically, with the average volume taken as the final result.

The samples were submitted to TML determination through the PFD80 test method, as stablished by the IMSBC Code for testing iron ore fines. The procedures described in the IMSBC Code [1] have been followed. The test apparatus consists of a 1,000 cm^3 cylindrical iron mould with a removable extension piece, and a 150 g compaction hammer of 50 mm tamper head diameter and drop height of 150 mm, guided by a pipe open at its lower end. Initially the samples were divided through a rotary divider, according to NBR ISO 3082:2011 [14], and 5 to 10 subsamples of approximately 2 litres in volume were taken. To establish each point of the compaction curve, one subsample was placed on a tray, a suitable amount of water was added using a spray bottle, and the sample material was gently mixed for 5 minutes. Approximately one fifth of the mixed sample was filled into the mould and levelled, and then the increment was tamped uniformly over its surface. Tamping was executed by dropping the hammer 25 times.

The performance was repeated for five sample layers. When the last layer had been tamped, the extension piece of the mould was removed and the sample was levelled off along the brim of the mould, resulting in a moulded sample of approximately 1,000 cm^3. The weight of the cylinder with the tamped sample was determined, the cylinder was emptied, and the moisture content was determined according to NBR ISO 3087:2012 [15]. To establish a complete compaction curve, this procedure was repeated for another 4 to 9 subsamples with different moisture contents, without sample reuse. Using the measured solids density, the parameters net water content in volume (e_v), void ratio (e) and degree of saturation (S) were calculated using Equations 2, 3 and 4 respectively.

$$e_v = \frac{Water\ Volume\left(cm^3\right)}{Solid\ Volume\left(cm^3\right)} \times 100\% \tag{2}$$

$$e = \frac{Void\ Volume\left(cm^3\right)}{Solid\ Volume\left(cm^3\right)} \tag{3}$$

$$S = \frac{e_v}{e} \tag{4}$$

From the results, the graph e versus e_v was plotted, including iso-saturation lines. The e_v value that corresponds to the intersection between the compaction curve and the 80% iso-saturation line was taken from the graph e versus e_v, and the TML was calculated using Equation 1, being expressed as gross water content by mass percent.

Data processing was conducted in Python 3 using Google Colaboratory, employing the following libraries: Pandas version 1.5.3 [16], Numpy version 1.23.5 [17], Matplotlib version 3.7.1 [18], and Seaborn version 0.12.2 [19].

3 Results and discussion

The TML is the percentage of water mass needed to fill 80% of the void volume in the compacted ore, relative to the total mass (ore + water). As the IMSBC Code requires, the TML is converted from volumetric moisture content on a dry basis to moisture content in mass on a wet basis through Equation 1, which utilizes the solids density (d). Equation 1 can be rewritten for 80% saturation (S = 80%) as follows: using Equation 4 from the IMSBC Code [1] and considering the void ratio corresponding to 80% saturation (e_{TML}), we can replace e_v with 80 x e_{TML}, thus obtaining Equation 5.

$$\boldsymbol{TML} = \frac{100 \times 80\boldsymbol{e_{TML}}}{100\boldsymbol{d} + 80\boldsymbol{e_{TML}}} \tag{5}$$

Equation 5 allows us to plot a surface representing the TML function of the PFD80 method, which relates TML to solids density and void ratio at the compaction condition corresponding to the TML. This surface is generated assuming the same degree of saturation, in this case, 80% (Figure 2).

Figure 2 shows the TML function as a curved surface, with a steeper gradient for lower solids density and higher void ratio. This means that for the same void ratio, higher solids density leads to lower TML, and this effect becomes stronger as void ratio increases. This behavior is related to TML being based on the ratio of water mass to total mass (water + ore). For example, imagine a fixed volume of ore and an equal volume of water. The mass ratio between solids and water depends on the density. Water has an approximate density of 1 g/cm³, so its mass virtually equals its volume. On the other hand, the mass of the solids depends on their density. Higher solids density for the same ore volume means more mass. As a result, the ratio of water mass to total mass (TML) decreases because the denominator (total mass) increases while the numerator (water mass) remains constant.

To illustrate how solids density affects TML, let's consider two iron ore fines: a hematitic sample (IOF 1) and a goethitic sample (IOF 2). Their PFD80 compaction curves (Figure 1) and characteristics (Table 1) are presented for reference. While IOF 2 (goethite content 51%) qualifies as Group C cargo under the IMSBC Code, it is included to highlight the density difference. Figure 1 shows that IOF 1 and IOF 2 have very similar compaction curves. After compaction in a standard 1,000 cm³ Proctor/Fagerberg mould at 80% saturation, both samples essentially hold the same volumes of solids, water, and voids. The TML for both samples is at e_v = 46.3%. However, due to its lower density, the goethitic ore packs less mass into the same volume. As both samples hold nearly the same amount of water by mass, the goethitic sample has a higher moisture content in percent on a wet basis. Since the moisture content in this example corresponds to 80% saturation, it represents the TML. This highlights how TML reflects the interplay between water and solids under specific volumetric conditions. Importantly, a higher TML doesn't necessarily imply an ore can hold more water than another if their densities differ. This practical example is illustrated in Figure 3

To delve deeper into the relationship between TML and solids density, the dataset of 86 IOF samples was evaluated. Table 2 summarizes key descriptive statistics for the 86 samples, including TML, solids density, void ratio at 80% saturation, some chemical and particle size distribution parameters, and the dominant mineral phases identified.

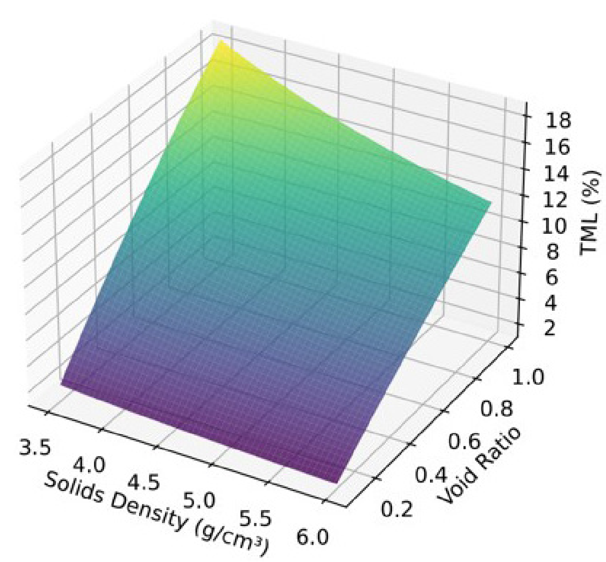

Figure 2. PFD80 TML function graph.

Table 1. IOF 1 and IOF 2 characteristics

Sample	TML	d (g/cm³)	e_{TML}	%Fe	%LOI	D_{50} (mm)	Coef. Unif.	% Hematite	% Magnetite	% Goethite	% Quartz
IOF 1	8.86	4.767	0.580	65.4	2.9	1.0	184	72.8	3.3	20.0	2.0
IOF 2	9.62	4.350	0.581	62.5	5.6	2.8	218	47.0	1.7	51.0	0.3

Table 2. Descriptive statistics for the samples characteristics

Parameter	TML	d (g/cm³)	e_{TML}	%Fe	%LOI	%<1.00 mm	Coef. Unif.	% Hematite	% Magnetite	% Goethite	% Quartz
Max	14.95	5.19	1.07	68.6	5.6	100.0	1137	96.5	13.9	51.0	29.3
Q3	13.02	4.92	0.90	65.4	2.4	87.4	279	82.1	3.3	15.3	5.6
Mean	11.22	4.80	0.76	63.7	2.1	62.8	190	77.2	2.0	13.4	4.8
Q1	9.47	4.72	0.63	63.1	1.6	49.1	10	73.0	0.4	9.5	2.6
Min	8.46	3.95	0.58	45.7	0.3	8.7	3	47.0	0.0	0.7	0.3
Std Dev	1.90	0.20	0.15	3.1	0.9	24.1	258	8.7	2.2	7.4	4.1

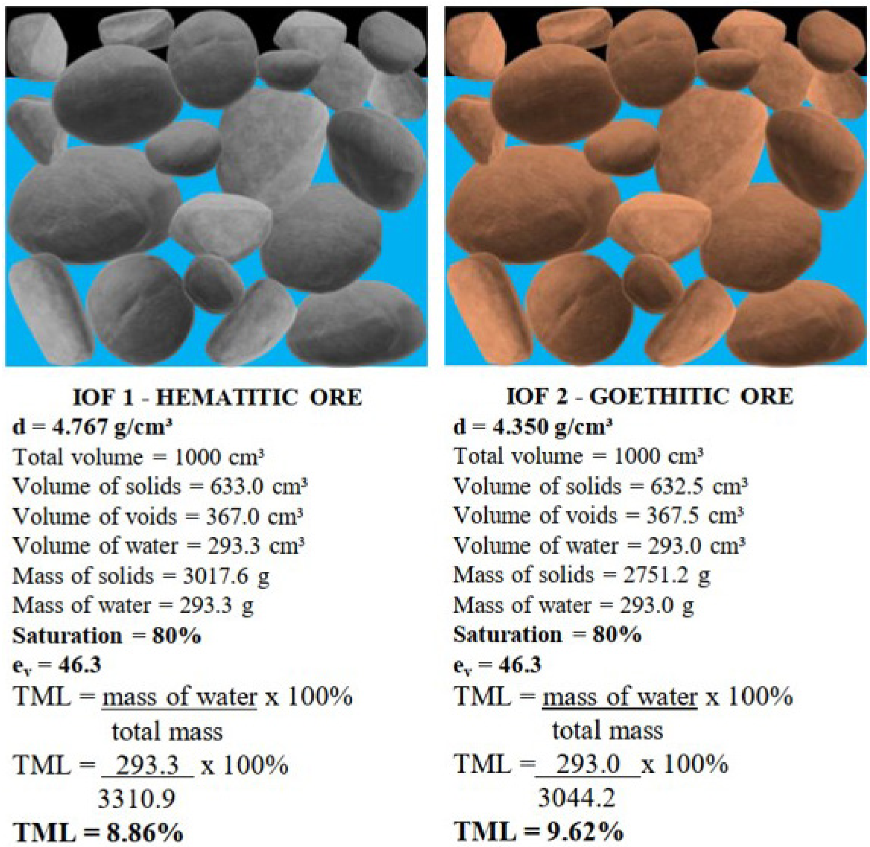

Figure 3. Hypothetical example of the solids density influence on TML.

The results span a TML range from 8.46% to 14.95%, which is a considerable range in terms of moisture content. Regarding solids density, the results vary from 3.95 g/cm³ to 5.19 g/cm³, with a mean of 4.80 g/cm³. This range reflects differences in mineral composition, ranging from quartz-rich itabirites (lower limit) to Fe-rich hematite (upper limit near pure hematite density). The void ratio corresponding to the TML (e_{TML}) varies from 0.58 to 1.07, with the latter value indicating a highly porous material with void volume exceeding solid volume at 80% saturation. The majority of samples show high Fe content, while the wide LOI distribution points to variability in goethite content. Particle size distribution also varies significantly, with samples exhibiting fines content ranging from very low to high, varying from well graded (high coefficient of uniformity) to poorly graded (low coefficient of uniformity) materials. Hematite, goethite, and quartz are the main minerals, but their relative abundance varies considerably across the dataset.

The correlogram in Figure 4 provides insights into the factors influencing solids density in the samples. This figure shows a visual map of correlations between solids density and the measured chemical and mineralogical variables (granulometric data are not expected to be relevant). Blue cells indicate positive correlations, brown cells negative. The shade of the color reflects the strength of the Pearson correlation, ranging from -1 (strong negative) to 1 (strong positive). Zero indicates no linear relationship. As expected based on mineral densities, the correlogram reveals a positive correlation between solids density and both Fe and hematite content and a negative correlation with LOI, goethite and quartz contents. Quartz stands out as the main factor negatively impacting the density of these samples. The analysis also confirms that hematite is the primary source of Fe in the dataset.

Figure 5 presents boxplots of TML for samples clustered by similar void ratio at 80% saturation (e_{TML}). As hypothesized, the figure confirms a proportional relationship between these variables. On average, for every 0.1 increase in void ratio, the TML increases approximately 1.5%. However, the graph also reveals a notable range of TML values within each e_{TML} group, reaching up to 2%. This suggests that additional factors beyond void ratio influence TML and should be investigated further.

Figure 6 presents the relationship between solids density and TML for samples with similar void ratios (e_{TML}). The figure highlights the influence of solids density, with higher density leading to lower TML, as expected in theory. This demonstrates how isolating the effect of variables like particle size and mineralogy that influence void ratio reveals the pure impact of solids density on TML. While the observed variation of TML with solids density is moderate compared to the dependence on void ratio seen in Figure 5, it remains significant. For a void ratio of 0.8 for example, a difference of 0.1 g/cm^3 in solids density leads to a variation of around 0.2% in TML. The level of variation increases proportionally with e_{TML} values.

Figure 7 presents a three-dimensional scatter plot, demonstrating the combined effects of solids density and void ratio on TML at 80% saturation. The experimental data points perfectly match the theoretical TML surface presented in Figure 2, clearly visualizing the impact of both variables, which has been discussed earlier. Having established the influence of solids density on TML, future research shall focus on understanding the factors influencing void ratio. Particle size distribution, already investigated by Ferreira and Lima [4], is just one piece of the puzzle. Particle porosity, morphology, and the interaction between water and minerals during compaction remain to be explored. This comprehensive approach will ultimately elucidate TML variations with ore characteristics, paving the way for optimizing moisture content control protocols for mineral cargo shippers.

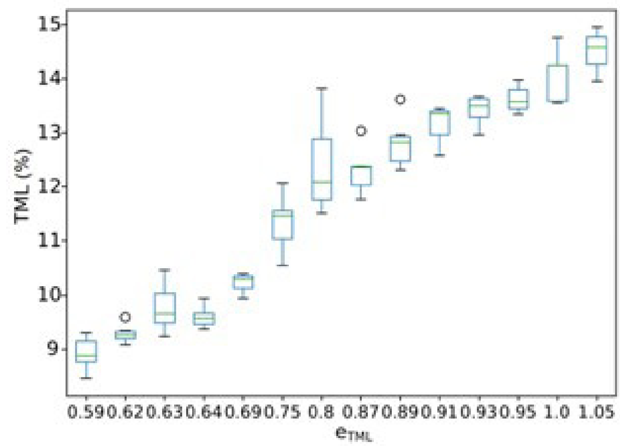

Figure 5. Boxplots of TML grouped by e_{TML}.

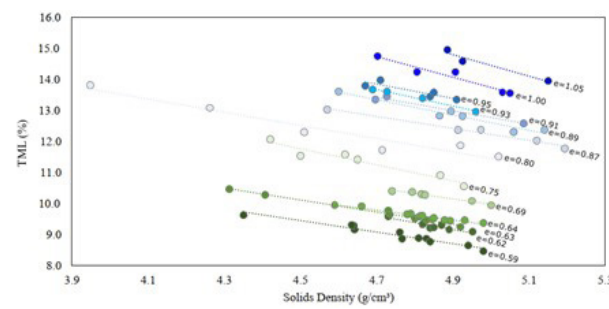

Figure 6. Scatterplot of TML and solids density.

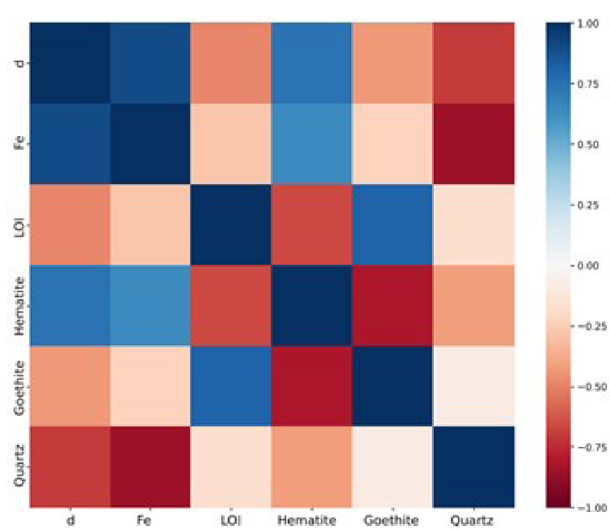

Figure 4. Correlogram between solids density and chemical and mineralogical variables.

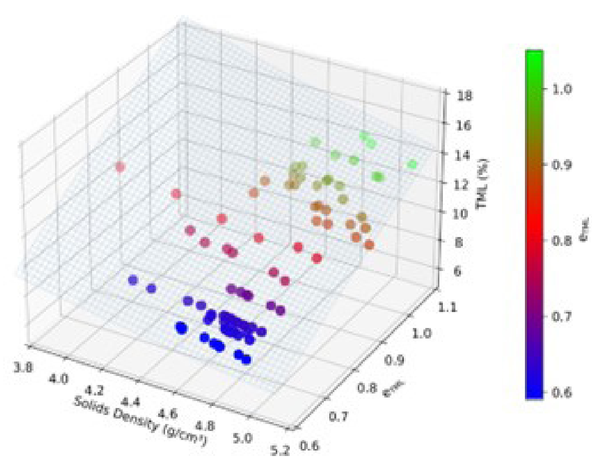

Figure 7. 3D scatterplot of TML, solids density and void ratio at 80% saturation

4 Conclusion

This study explored the relationship between Transportable Moisture Limit (TML) and solids density. As defined by Fagerberg, the moisture limit is experimentally determined as the amount of water filling a given volume. Expressed in moisture content in mass on a wet basis, it depends on solids density. Theory predicts that, for the same void ratio at 80% saturation – the condition defining TML – lower solids density leads to higher TML. The experimental approach, involving 86 iron ore fines samples with diverse densities, confirmed the theoretical predictions. The highly significant correlation between TML and solids density underscores the critical importance of precise solids density determination, as required by the IMSBC Code. The findings of the study demonstrate that a higher TML doesn't necessarily imply an ore can hold more water than another if their densities differ.

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References

1 International Maritime Organization. International Maritime Solid Bulk Cargoes Code. London: International Maritime Organization – IMO; 2022.

2 International Maritime Consultative Organization. The Bulk Cargoes Code. London: International Maritime Consultative Organization – IMCO; 1965.

3 Ferreira RF, Pereira TM, Lima RMF. A model for estimating the PFD80 transportable moisture limit of iron ore fines. Powder Technology. 2019;345:329-337.

4 Ferreira RF, Lima RMF. Relationship between particle size distribution and the PFD80 transportable moisture limit of iron ore fines. Powder Technology. 2023;414:118072.

5 Fagerberg B. Hazards of shipping granular ore concentrates – Part I. Canadian Mining Journal. 1965:53-57.

6 Fagerberg B. Hazards of shipping granular ore concentrates – Part II. Canadian Mining Journal. 1965:81-86.

7 Fagerberg B, Stavang A. Determination of critical moisture contents in ore concentrates carried in cargo vessels. In: Proceedings of the 1st International Symposium on Transport and Handling of Minerals; 1971 October; Vancouver, Canada. San Francisco: Miller Freeman; 1971. p. 174-185.

8 Munro MC, Mohajerani A. Determination of the transportable moisture limit of iron ore fines for the prevention of liquefaction in bulk carriers. Marine Structures. 2015;40:193-224.

9 Williams K, Honeyands T, Holmes R, Orense R, Roberts A, Pender M, et al. Maritime bulk cargo transportable moisture limit requirements for iron ore shipments. In: The Australasian Institute of Mining and Metallurgy, organizador. Proceedings of the Iron Ore Conference; 2015 Jul 13-15; Perth, Australia. Melbourne: AUSIMM; 2015. p. 399-410.

10 Rose TP. Solid bulk shipping: cargo shift, liquefaction and the transportable moisture limit [dissertation]. Oxford: University of Oxford; 2014.

11 AMIRA International. P1097 - Systematic evaluation of transportable moisture limit measurement methods for iron ore fines bulk cargoes — public final report. Perth: AMIRA; 2014.

12 International Maritime Organization - Iron Ore Fines Technical Working Group. Submission for evaluation and verification: iron ore Proctor Fagerberg test. London: International Maritime Organization - Iron Ore Fines Technical Working Group (IOFTWG); 2013.

13 ASTM International. ASTM D5550-14: Standard test method for specific gravity of soil solids by gas pycnometer. West Conshohocken: ASTM International; 2014.

14 Associação Brasileira de Normas Técnicas. NBR ISO 3082: Minérios de ferro – procedimentos de amostragem e preparação de amostras. Rio de Janeiro: ABNT; 2011 [in Portuguese].

15 Associação Brasileira de Normas Técnicas. NBR ISO 3087: Minérios de ferro – determinação do teor de umidade de um lote (corrected version: 2016). Rio de Janeiro: ABNT; 2012 [in Portuguese].

16 Mckinney W. Data structures for statistical computing in Python. In: Proceedings of the 9th Python in Science Conference; 2010 June 28-July 3; Austin, USA. Austin: SciPy; 2010. p. 51-56.

17 Harris CR, Millman KJ, van Der Walt SJ, Gommers R, Virtanen P, Cournapeau D, et al. Array programming with NumPy. Nature. 2020;585:357-362.

18 Hunter JD. Matplotlib: a 2D graphics environment. Computing in Science & Engineering. 2007;9(3):90-95.

19 Waskom ML. Seaborn: statistical data visualization. Journal of Open Source Software. 2021;6(60):3021.

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