# Use of tensiometers to determine the Moisture Characterization Point in ores

Arjan Wijdeveld<sup>1</sup>, Tim Evens<sup>2</sup>, Johan Pennekamp<sup>3</sup>

<sup>1</sup>Technical University of Delft, Stevinweg 1, 2628 CN, Delft // Deltares, Boussinesqweg 1, 2629 HV, Delft, the Netherlands, Arjan.Wijdeveld@deltares.nl

<sup>2</sup>Rio Tinto, Sídney, Australia, Tim.Evans@riotinto.com

<sup>3</sup>Deltares, Boussinesqweg 1, 2629 HV, Delft, the Netherlands, Johan.Pennekamp@deltares.nl

### Abstract

The Moisture Characterization Point (MCP) of an ore is based on the moisture behavior in the unsaturated pores, close to but below the field capacity of the ore. The MCP is an alternative method to determine safe moisture limits for the shipment of ore as compared to geotechnical test methods used to determine the Transportable Moisture Limit (TML) in ores, such as the Proctor Fagerberg test.

In this study a soil tensiometer (Hensley 2009) was adapted to measure the suction pressure (pF) as function of the moisture content in the ore. The Moisture Characterization Point (MCP) was defined as a suction pressure of -0,25 bar. This point is below, but relative close to, the field capacity of the ore. The MCP was correlated with the physical and chemical properties of the ores, like particle size, specific surface area and mineral composition of the ores.

The results showed a wide variation in the MCP's for nine different ores, varying between a MCP moisture content of 3.8% up to 21.5 %. The results where compared with the TML limit based on the Proctor Fagerberg tests. For most ores the MCP and TML levels are close (on average a relative difference of 10% between the TML and MCP). For ores with a substantially larger difference then 10%, the ores mineralogy is a dominant factor. The ore mineralogy could be linked to the ore surface area, leading to the conclusion that shipment of ore with goethite content above 35% is not liable to liquefy.

A better understanding of the moisture behavior in ores has assisted in the development of safer cargo schedules (IMO 2013) to ensure the safe shipping of bulk cargoes.

Key words: IMWA 2016, moisture, ores, suction, shipping

### Introduction

In 2010, 44 seafarers lost their lives when a number of bulk carrier vessels capsized due to liquefaction of the cargo. These and later incidents have triggered the International Maritime Organization to review cargo schedules defined in the International Maritime Solid Bulk Cargoes Code (IMO 2011). Group A cargoes are liable to liquefaction, while Group C cargoes are not prone to liquefaction. The safe carriage of Group A cargoes is possible by ensuring they are shipped at moisture contents below their Transportable Moisture Limit (TML).

One aspect that determines if liquefaction might occur is the degree in which pores are saturated. As long as pores are unsaturated (below the ores field capacity) pore water pressure buildup due to ship roll cannot occur, and a wet failure mode is not possible. The Moisture Characterization Point (MCP) is based on the moisture behavior in the unsaturated pores, close to but below the field capacity of the ore. A suction pressure of -0.25 bar was chosen as reference point for the MCP. The MCP is an alternative method to determine safe moisture limits for the shipment of ore as compared to geotechnical test methods used to determine the TML in ores, such as the Proctor Fagerberg test.

The MCP adds information by characterizing how moisture is bound within the pores of the ore for what can still be described as a drained (unsaturated) ore. At low moisture contents, the pore water is bound

to the mineral matrix as hydration water. This water is irreversibly bound and will not drain. With increasing moisture contents, the pores in the ore are filled with water. The smallest pores have the highest capillary binding force (soil moisture potential, or suction pressure, expressed in KPa) and will be filled first. With increasing moisture contents the larger pores are filled. As long as the moisture content is below the field capacity, the ore will not drain. Above the field capacity of the ore the pore suction is less than the gravital forces, and the ore can drain (Brouwer 1985). For soil sciences this is known as the three soil moisture states describing unavailable water, plant available water and drainable water (O'Geen 2012). Figure 1 illustrates these three soil moisture states.



**Figure 1:** Water content and water potential at saturation, field capacity and permanent wilting point. The difference in water content between field capacity and permanent wilting point is (plant) available water. Drainable porosity is the amount of water that drains from macro pores by gravity between saturation to field capacity typically representing three days of drainage in the field.

© 2012 Nature Education Adapted from Brady & Weil 2002 and McCauley et al. 2005 All rights reserved.

As long as the ores are below the field capacity, suction is greater than gravity. Moisture will not migrate (seep) due to ship roll and pore water pressure cannot build up. Hence the ore behaves as a drained ore.

Soil tensiometers are designed to measure the pore suction under static conditions in hydrostatic equilibrium (Klute 1962). The response time to changes in moisture content is on a daily scale. More recent developments in miniature tensiometers shorten this time interval to hours (Cui, 2008). To establish the moisture versus suction relation for different ores and different grain sizes, a step wise small increase in moisture content is needed to determine the slope of the pore suction pressure versus the moisture content. Therefore a smaller miniature tensiometer was developed to measure changes in pore suction pressure at given moisture content on a timescale of minutes. By a step wise increment of the moisture content in the ore a moisture versus pore suction pressure response curve was measured, similar to a soil pF curve (Blaskó Lajos 2011).

Characterization of the ore mineralogy, the ore pore size distribution and the ore surface area was carried out. These supportive measurements correlate to the water adsorption properties of the ore due to the binding of hydration water, capillary suction and the storage capacity based on pore volume.

# Methods

The used ore samples were iron ores, bauxite and sand. The ores where characterized by their; bulk density using a pycnometer, initial moisture content (as total mass percentage), grain size distribution for particles between 6.3 mm and 0.063 mm by wet sieving (NEN 2006) and for particles smaller than 0.063 mm by laser diffraction (Vdovic 2010). The ores were characterized on their surface area (in m2/gram) by mercury and nitrogen intrusion (Zong 2014). The meso pore size distribution was calculated by means of the Barrett-Joyner-Halenda (BJH) pore size model (Lowell 2012), using the Brunauer, Emmet and Teller (BET) theory for surface area estimation (Kreysa 2000).

The tensiometers used were from RHIZO INSTRUMENTS in Wageningen, the Netherlands. Each tensiometer has three essential parts:

- An air permeable / water impermeable plastic hollow fiber (pore size 0.03 µm). The fiber is protected by a ceramic cup (pore size 0.45 µm) with a diameter of 3.0 mm.
- A flow through pressure transducer (12 mV/bar output at 3.0 V input, resolution 0.001 mV, -1.0 to +1.0 bar range).
- A vacuum chamber source: for single point measurements a syringe with spacer, for the titration setup a membrane vacuum pump (Baltalab N86KN.18).



Figure 2 illustrates the different tensiometer elements.

*Figure 2,* tensiometer setup. On the right the porous plastic hollow fiber protected by a ceramic cup. In the middle the pressure conductor with a range of plus or minus 1 bar (12 mV/bar output at 3 V). To the lift the vacuum chamber, in this case a syringe.

By applying a vacuum the pore suction pressure in the tensiometer drops to -0.95 bar. When the suction pressure in the pores increases to a value above -0.95 bar water flows into the tensiometer. The suction pressure in the tensiometer decreases. If the pore suction pressure equals the suction pressure inside the tensiometer, the flow of water stops and the tensiometer is in equilibrium. The tensiometer response curve as function of moisture content can be divided in three regimes:

1. At a moisture contents above the field capacity the tensiometer response drops to zero. The tensiometer completely fills up with water and there is zero pore suction pressure. This is reflected by a quick drop to 0% signal.

- 2. At moisture concentrations close to, but below, the field capacity, the tensiometer reaches equilibrium with the pore suction pressure (range -0.00 to -0.95 bar). The tensiometer response represents the actual pore suction pressure.
- 3. Closer to the wilting point the applied vacuum (-0.95 bar) inside the tensiometer is not strong enough to compete with the pore suction pressure. The signal stays close to 100%.

This divides the tensiometer response curves into three types of response curves, which are illustrated in Figure 3 for one of the nine ores (ore C) at different moisture contents.



*Figure 3, Tensiometer response near full saturation (in orange/red), near the field capacity (in yellow/green)* and near the wilting point (in purple/blue). The x-axis plots the response after closing the vacuum from 0 to 6 minutes. On the y-axis the signal (%) is plotted

# Results

The soil permeability, or hydraulic conductivity, relates to the pore size distribution (Burdine 1953) and soil texture data (Ghanbarian-Alavijeh 2010). Table 1 summarizes the ore texture characteristics, the ore surface area and the MCP and gives the calculated regression coefficient between the parameters and the measured MCP based on a first order fit.

**Table 1,** Ore texture characteristics for nine ores and sand (grain size distribution per class (d10, d50 and uniformity coefficient d60/d10), the ore surface area (BET)) and the MCP (75% loss of signal, pore suction pressure -0.25 bar). Calculated correlations between different ore texture characteristics and the MCP based on a first order fit.

<b></b>						
		00/010 010/010	g d10	m d50	LJ BBS m2.g-1	(%) WCP
ore A	iron ore	200	0.01	1.00	13	14.0%
ore B	iron ore	97	0.02	0.73	4.4	8.5%
ore C	iron ore	292	0.01	1.26	5.5	9.8%
ore D	iron ore	68	0.03	0.68	6.6	7.3%
ore E	iron ore			0.18	0.7	6.8%
ore F	iron ore	2.7	0.07	0.16	0.5	3.8%
ore G	iron ore	38	0.12	3.23	20	15.0%
ore H	iron ore	54	0.05	1.68	46	12.0%
ore I	aluminium ore, pre-dried	2.3	2.95	5.86	72	21.5%
sand	sand, pre-dried	2.5	0.25	0.45		9.3%
first order fit						R <sup>2</sup>
Correlation between particle size uniformity coefficient (UC) and the MCP						
Correlation between d10 particle size and the MCP						0.15
Correlation between d50 particle size and the MCP						0.83
Correlation between BET and MCP						0.82
Correlation between d10 and BET						0.21
Correlation between d50 and BET					0.89	

In addition to the properties listed in Table 1, XRD measurements where done to characterize the mineral composition of the ores. The minerals detected by XRD are given in Table 2.

Table 2 Minerals detected by XRD for all nine ores and sand,	including the mineral formula.
The data was not quantified.	

XRD detected minerals			
Quartz	SiO2		
Calcite	CaCO3		
Gypsum	CaSO4		
Gibbsite	AI(OH)3		
Goethite	FeO(OH)		
Hemathite	Fe2O3		
Magnetite	Fe3O4		
Siderite	FeCO3		
Muscovite (mica)	KAI3Si3O10(OH)1.8F0.2		
Dickite (kaolinite)	Al2Si2O5(OH)4		
Illite	K0.6 (H3O)0.4 AI1.3 Mg0.3 Fe2+0.1 Si3.5O10 (OH)2		

The MCP was established for nine ores and sand. Figure 4 gives the percentage of signal loss as function of the moisture content. The MCP as defined by a pore suction pressure of -0,25 bar (a 75% loss of signal) is plotted as a dotted red line.



*Figure 4* Signal loss (pressure potential drop) for nine ores and sand as function of the moisture content. The x-axis gives the moisture content (% gross weight). The y-axis plots the percentage loss of signal at a given moisture content for each of the nine ores. The MCP is plotted horizontally as the point of 75% signal loss.

The TML has been determined according to the IMSBC code (IMO 2013). The TML, with a 70% degree of moisture saturation, is plotted in Figure 6. For comparison the MCP, as defined by a 75% loss of signal, is also plotted in Figure 5.



Figure 5 On the x-axis the nine ores and sand. On the y-axis the TML moisture content (% gross mass) for eight of the nine ores as determined by the IMSBC code (IMSBC 2015). The results are compared to the MCP for all nine ores and sand.

### Conclusions

Based on the (Van Genuchten 1980) closed-form equation for predicting the permeability of an unsaturated soil, it was expected that there would be a positive correlation between the increase in MCP and a larger amount of smaller ore particles (as expressed by an increase in the UC), and between the MCP and the ore surface area (as expressed by the BET). Small particles tend to have a larger surface area (Lecloux 2015) and the micro pore volume between small particles has a lower suction pressure as compared to the macro pores (Mancuso 2012). The relation between the MCP and the amount of smaller particles (as expressed by the d10 and d50 and a high UC value) could not been found in the tested ores, as is illustrated by the low regression coefficients in Table 5. However, the correlation between the MCP and larger ore particles (high BET, high value of the d50) was good ( $R^2$  of > 0.8). Since the ore surface area is not correlating with the amount of smaller particles, the ore mineralogy plays a role.

The ore mineralogy plays an important role when it comes to the mineral surface area. In general goethite tends to have the largest surface area (Hiemstra 1998), although the surface area of goethite varies as function of the formation conditions (Villalobos 2003). The correlation between the goethite percentage of the ore and the ore surface area is moderate (a  $R^2$  of 0.7). For hemathite this correction is weak (a  $R^2$  of 0.4). On average goethite has a 3.5 times as strong contribution to the ore surface area as compared to an equal mass percentage of hemathite. With an increase in the mass percentage of goethite, the MCP also increases.

The goal of this research was to ensure safe moisture standards for shipment of ore based on the transition of the ore between the drained and undrained state of an ore (the field capacity). Ores in a drained state cannot build up excess pore water pressure due to ship roll. The relation between the ore moisture content and the pore suction pressure was determined, leading to the ore specific field capacity. The Moisture Characterization Point (MCP) is based on this relation and defines a safe shipping standard with a safety margin (remaining pore suction pressure of -0.25 bar) with regard to the actual field capacity (the transition from an undrained to a drained state) of the ore. The field capacity and the curve describing the loss of suction pressure as function of moisture content is primary dependent on the type of ore minerals, and to a lesser extent on the ore grain size.

The MCP gives additional information based on ore mineralogy as compared to Transportable Moisture Limit (TML) standard, which is based on the ores compaction behavior versus moisture content (Proctor/Fagerberg Test). In general the TML and MCP correlate well, with a relative difference of around 10%.

#### Acknowledgements

The authors want to thank Rio Tinto Hamersley iron for not only funding the research, but also for their active role in the development of new measurement equipment and the interpretation of the results.

#### References

- Blaskó, Lajos (2011), Soil science, Debreceni Egyetem. Agrár- és Gazdálkodástudományok Centruma, TÁMOP-4.1.2-08/1/A-2009-0032 pályázat keretében készült el.
- Brady, Nyle C. and Ray Weil (2002), "The Nature and Properties of Soils", Pearson; 13th edition
- Brouwer, C. (1985). "Introduction to Irrigation". L. a. W. D. D. The International Support Programme for Irrigation Water Management. Rome, Italy
- Burdine, N. (1953), "Relative permeability calculations from pore size distribution data". Journal of Petroleum Technology, 5(03), 71-78.
- Cui, Y. J., et al. (2008), "Monitoring field soil suction using a miniature tensiometer." Geotechnical Testing Journal 31(1): 95-100
- Ghanbarian-Alavijeh, B. et al (2010), "Estimation of the van Genuchten Soil Water Retention, Properties from Soil Textural Data", Pedosphere 20(4): 456–465

- Hensley, David, and James Deputy (1999), "Using tensiometers for measuring soil water and scheduling irrigation." Landscape 50.10
- Hiemstra T., J.C.M De Wit, W.H Van Riemsdijk (1989), "Multisite proton adsorption modeling at the solid/solution interface of (hydr)oxides: A new approach: II. Application to various important (hydr)oxides", Journal of Colloid and Interface Science, Volume 133, Issue 1, pp. 105-117
- IMO (International Maritime Organisation) (2011), International Maritime Solid Bulk Cargoes Code (IMSBC), resolution MSC 268(85), mandatory under the pro-visions of the SOLAS Convention
- IMO (International Maritime Organisation) (2013), International Maritime Solid Bulk Cargoes Code (IMSBC), amendment 02-13 and amendment 03-15, as described in the DSC.1/Circular.71
- Klute, A. and W. Gardner (1962), "TENSIOMETER RESPONSE TIME." Soil Science 93(3): 204-207
- Kreysa, G., et al. (2000), Characterisation of Porous Solids V, Elsevier Science
- Lecloux, A. J. (2015), "Discussion about the use of the volume-specific surface area (VSSA) as criteria to identify nanomaterials according to the EU definition: First part: theoretical approach." Journal of Nanoparticle Research 17(11): 1-18.
- Lowell, S., et al. (2012), Characterization of Porous Solids and Powders: Surface Area, Pore Size and Density, Springer Netherlands
- Mancuso C., Cristina Jommi, Francesca D'Onza (2012), "Unsaturated Soils: Research and Applications, Volume 1" Springer Science & Business Media
- NEN 5753:2006/C1:2009 (2006), Soil Determination of clay content and particle size distribution in soil and sediment by sieve and pipet
- O'Geen (2012), A. T., Soil Water Dynamics. Nature Education Knowledge 3 (6): 12
- Van Genuchten, M. T. (1980), "A closed-form equation for predicting the hydraulic conductivity of unsaturated soils". Soil science society of America journal, 44(5), 892-898.
- Vdovic, N. N. N. (2010), "Revisiting the particle-size distribution of soils: Comparison of different methods and sample pre-treatments." European Journal of Soil Science 61(6): 854-864
- Villalobos, M., Maya A. Trotz and James O. Leckie (2003), "Variability in goethite surface site density: evidence from proton and carbonate sorption", Journal of Colloid and Interface Science 268, 273–287.
- Zong, Y. Y. (2014), "Characterizing soil pore structure using nitrogen adsorption, mercury intrusion porosimetry, and synchrotron-radiation-based X-ray computed microtomography techniques." Journal of Soils and Sediments 15(2): 302-312