Available online at www.sciencedirect.com
ScienceDirect
Applied Clay Science 40 (2008) 15–19
Applied CLAY Science
www.elsevier.com/locate/clay

# Determination of clay plasticity: Indentation method versus Pfefferkorn method ☆

Cláudio de Oliveira Modesto [a], Adriano Michael Bernardin [b,c,*]

[a] *Instituto Maximiliano Gaidzinski, Rua Dr. Edson Gaidzinski 352, Centro, 88.845-000, Cocal do Sul, SC, Brazil*
[b] *Programa de Pós-Graduação em Engenharia Química, Universidade Federal de Santa Catarina, Campus Universitário, 88.040-900, Florianópolis, SC, Brazil*
[c] *Engenharia de Materiais, Universidade do Extremo Sul Catarinense, Avenida Universitária 1.105, Bairro Universitário, 88.806-000, Criciúma, SC, Brazil*

Received 2 April 2007; received in revised form 9 June 2007; accepted 16 June 2007
Available online 5 July 2007

**Abstract**

As a basis of ceramic process development, raw material selection plays a fundamental role in the final product design. For correct material selection, knowledge regarding all the properties of the raw materials should be obtained. Plasticity is one of the most important properties for molding clay products. Nevertheless, the standard methods in use, including the Pfefferkorn method, are not precise and reliable. They are based on subjective qualitative information and are dependent on technical skills. The literature shows some attempts to improve the test method, all based on the force needed to indent a clay sample surface with specific water content. Four different clays were studied and the plasticity values due to the Pfefferkorn method and the indentation test were compared. The results showed that the indentation method is reliable and more precise compared to Pfefferkorn. It is also faster and more practical.


*Keywords:* Clay minerals; Plasticity; Indentation; Pfefferkorn test method

## 1. Introduction

Plasticity is a basic clay property that permits this material to form a plastic body (Fenili et al., 2004). When a plastic body is submitted to the action of a force it becomes deformed and perfectly conserves that form after the force applied is discontinued (Atterberg, 1975; Burst, 1991; Murray, 1991; Ancey, 2007; Yu et al., 2007). A relation exists between geological formation and plasticity. Some clays possess higher plasticity than others probably because some interstitial materials are well preserved from weathering (Peters, 1991; Kolmayer et al., 2004).

The same occurs among sedimentary clays that have been transported from their place of formation; during transportation they suffer a comminuting process and contamination with certain products, turning them into a more plastic material when compared to residual clays that have not suffered transformations during transport; as residual clays still contain unaltered rocks they

☆ Presented at the VIII World Congress on Ceramic Tile Quality (QUALICER), February 10th 2004.

* Corresponding author. Engenharia de Materiais, Universidade do Extremo Sul Catarinense, Avenida Universitária 1.105, Bairro Universitário, 88.806-000, Criciúma, SC, Brazil. Tel.: +55 48 3431 2639; fax: +55 3431 2750.
*E-mail addresses:* modesto@imgnet.org.br (C. de Oliveira Modesto), adriano@unesc.net (A.M. Bernardin).

0169-1317/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.clay.2007.06.007

present little plasticity (Basma et al., 1996; Malkawi et al., 1999). The transport is always accompanied by size-fractionation, and the changes in the particle sizes and size distribution have an important influence (Bergaya et al., 2006).

The mineralogical composition of distinct argillaceous materials can influence their plasticity; the clay mineral fraction and types and the quantity and type of natural accessory materials can alter plasticity. The most important plastic clays used in the ceramic industry are kaolinite, illite and montmorillonite and mixed-layer minerals (Atterberg, 1975; Burst, 1991; Murray, 1991; Schmitz et al., 2004).

Generally, plasticity is influenced by the basal spacing of the 2:1 clay minerals; the larger the basal spacing, the higher its water adsorption capacity (Basma et al., 1994; Al-Shayea, 2001; Sánchez-Girón et al., 2001). The water adsorption capacity of smectites is larger because of the different structure in comparison with kaolinite and the ability of interlayer water adsorption (intercalation) (Bergaya et al., 2006).

Among the main impurities that possess nonplastic properties are iron minerals (mainly $Fe_2O_3$), aluminum oxide ($Al_2O_3$), sodic and potassium feldspars, soluble salts ($K_2SO_4$, NaCl, $Na_2CO_3$, etc.), calcium minerals (mainly calcite) and silica (Peters, 1991; Ancey, 2007).

However, when the impurities have particle size under 4 μm they can remain suspended in the clay mineral raising the ceramic body plasticity. The relation between particle size and clay mineral plasticity is inversely proportional, because the smaller specific surface area of larger particles reduces the amount of water adsorbed (Peters, 1991; Malkawi et al., 1999; Basma et al., 1996).

The presence of organic substances in clays is higher in sedimentary clays. The organic substances have a high specific surface and good molding capacity, improving the plasticity of the clays (Malkawi et al., 1999).

The Pfefferkorn method is the most commonly used method to measure plasticity. This method determines the amount of water required to achieve a 30% contraction in relation to the initial height of a test body under the action of a weight standard. The results are normally expressed as graphs showing height reduction as a function of moisture content.

The Pfefferkorn analyses are often incoherent and imprecise, resulting in the erroneous analysis of green and dry mechanical resistances of ceramic products, as well as drying and conformation characteristics.

The main problems regarding plasticity determination using the Pfefferkorn method are related to moisture analysis, the relation between residual and sedimentary clay and test delay and difficulty (Dafalias et al., 2002; Conil et al., 2004). As previously cited, the Pfefferkorn method determines raw material plasticity as water content and not as the resistance to penetration or plastic deformation. This fact causes failures in plasticity analyses of similar clays.

Due to changes during the transport as discussed above, sedimentary clays should be more plastic than residual clays. Tests, however, showed that the Pfefferkorn method fails in this analysis, showing a higher plasticity of residual clays (Peters, 1991; Basma et al., 1994; Ancey, 2007; Yu et al., 2007).

Finally, the Pfefferkorn test is laborious and time consuming. It requires successively removing and adding the sample moisture in order to reach 30% height contraction in relation to the initial height of the test body. Additionally, to finalize the test, the sample needs to be dried.

Due to the failures of the Pfefferkorn method, some new methods for plasticity determination have been presented in the last few years, demonstrating greater accuracy in spite of simpler procedures (Christaras, 1991; Doménech et al., 1994; Baran et al., 2001). This article presents a very simple method for plasticity analysis of clays. A simple device is used, derived from indentation equipment.

## 2. Materials and methods

The equipment is an adaptation of a simple penetrometer, to which a cylindrical cement base was adapted. However, the use of a cast iron cylinder as the base was preferred. The main accessories include a digital display (0.01 mm resolution), for displacement measurement; interchangeable cylinders containing an internal spring (50 N/cm), for the reaction force determination; a control spring, that guarantees a constant and gradual application of an indentation force on the sample; and a cylindrical cone, with a 30 mm base and an angle of 30°.

The test procedure is based on the application of an indentation force on a clay sample, the plasticity is directly given by the indentation force measured on the digital display. If the perforation mark printed on the sample presents no cracks or any kind of plastic flow, the test is considered valid and the force value displayed on the penetrometer device is the plasticity value.

The samples were prepared by 30 min milling of 700 g of each raw material in a porcelain jar eccentric mill. The ceramic suspension obtained was dried in an infra-red oven. The granules were disaggregated and sieved through a 500 mesh sieve. The dried powder was added to water adjusting a water content of approximately 25 wt.%. The resulting paste was homogenized in a mechanical stirrer. Finally, each

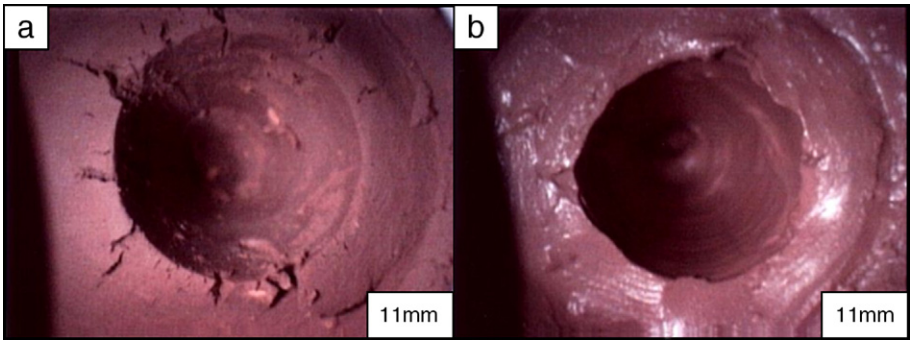


Fig. 1. Clay indentation, showing (a) a lack of water and (b) an excess of water.

sample was indented in order to obtain a smooth and uniform mark and when necessary the moisture content was adjusted for that.

The milling process changes the plasticity property of the raw material. Since both tests (indentation and Pfefferkorn) were carried out with the same clays prepared in the same way, possible errors may occur with both methods.

Also, it must be stated that the mineralogical phases are not only responsible for the plasticity. The organic matter is also important for the plasticity but it was not tested in this article because the classical plasticity tests also do not test the organic matter.

Marks with cracks or plastic flow mean, on the one hand, a lack of plasticity (a lack of water, Fig. 1a), and on the other hand, a lack of consistency (an excess of water, Fig. 1b). These extreme situations show two behaviors, the plastic limit (lack of plasticity) and the liquid limit (lack of consistency), established as Atterberg limits (Atterberg, 1975). Several methods for plasticity determination use these limits as standards. The difference between them is determined as the plasticity (Bergaya et al., 2006).

Good plasticity occurs when the marks do not present cracks nor extreme humidity, the wall formed is sufficiently smooth and, therefore, this is the humidity chosen for plasticity determination.

After indentation, the next step is the preparation of a test body for plasticity determination. The samples that present good wall formation are then reshaped into cylindrical forms and the penetration test is performed. The penetration force for the specific moisture content determined during this indentation test is the plasticity value.

## 3. Results and discussion

Four raw materials were used to test the proposed method, two residual clays (designated P and F) and two sedimentary clays (E and R). The chemical and mineralogical analyses are presented in Table 1.

Table 1
Chemical and mineralogical analyses for the raw materials used in the tests

Chemical analysis

| R.M.[a] | $SiO_2$ | $Al_2O_3$ | $K_2O$ | $Na_2O$ | MgO | CaO | $Fe_2O_3$ | $TiO_2$ | $P_2O_5$ | MnO | S[b] | L.O.I.[c] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| F | 64.5 | 22.1 | 2.8 | 0.1 | 0.8 | 0.1 | 2.3 | 0.9 | 0.1 | • | <0.1 | 6.4 |
| P | 55.0 | 30.1 | 0.6 | • | 0.2 | 0.1 | 2.7 | 1.1 | • | • | <0.1 | 10.2 |
| R | 69.2 | 15.2 | 4.5 | 0.6 | 1.5 | 0.2 | 3.8 | 0.6 | • | 0.1 | <0.1 | 4.3 |
| E | 71.8 | 11.8 | 3.9 | 1.6 | 1.5 | 2.0 | 2.6 | 0.4 | 0.1 | 0.1 | <0.1 | 4.2 |

Mineralogical analysis

| R.M.[a] | Kaolinite | Illite | Quartz | Calcite | Potash feldspar | Soda feldspar | Magnetite | Accessories[d] |
|---|---|---|---|---|---|---|---|---|
| F | 31.0 | 27.0 | 36.5 | • | <2.0 | • | • | 3.5 |
| P | 71.0 | 6.7 | 19.3 | • | • | • | • | 3.0 |
| R | 7.5 | 25.0 | 41.0 | • | 13.0 | 5.0 | 3.8 | 4.7 |
| E | 13.0 | <2.0 | 41.0 | 3.6 | 23.0 | 14.0 | 2.6 | 0.8 |

The mineralogical phases were quantified by rational analysis.
[a] Raw materials.
[b] S total.
[c] Lost of ignition (1000 °C).
[d] Unidentified phases.

Table 2
Comparison of the Pfefferkorn and indentation tests

| Raw material | Pfefferkorn method [a] | Indentation method [b] |
|---|---|---|
| Residual clay F | 31.0 | 19.9 |
| Residual clay P | 24.3 | 28.8 |
| Sedimentary clay R | 24.5 | 24.5 |
| Sedimentary clay E | 25.0 | 26.9 |
| | wt.% water amount | Force (N) |

[a] Uncertainty: 5%.
[b] Uncertainty: 2.5%.

P presented low plasticity compared to F, because the latter may contain more illite than the former and probably thinner particles and smaller average particle size (speculation based on workability, not proven facts). Clay F originated from a Paleozoic geologic formation and is a hillside residual clay with low quantities of organic material.

Based on its mineralogical composition the sedimentary clay R should be more plastic than E (25.0 wt.% of illite), since sedimentary clay R originates from a less preserved geological formation in comparison to E. However, R possesses a larger quantity of clay minerals and a lower quantity of nonplastic materials and impurities, when compared with E. Since they present similar size distribution R should be more plastic than E.

The results of the Pfefferkorn and indentation methods are listed in Table 2.

Due to the Pfefferkorn test the plasticity of the two sedimentary clays and the residual clay P was the same, approximately 24.5 wt.% of water. This clearly demonstrates the lack of accuracy of this method.

In contrast, the indentation method showed differences among the plastic behavior. P was the least plastic clay, an aspect not shown by the Pfefferkorn method. This result was confirmed by the mineralogical analysis, which showed a minimal quantity of clay minerals for P, in comparison with F, R and E, regardless of their particle size distribution.

As the indentation method showed the residual clay F was the most plastic clay, because the force needed to deform it was the smallest, in agreement with high contents, about 70%, of kaolinite and illite.

Finally, the time required for the indentation tests using humidified samples took a maximum of 10 min; the Pfefferkorn method needed 3 to 5 h.

## 4. Conclusion

The proposed method is based on the indentation force needed to penetrate a sample; it is much more precise and quicker than the Pfefferkorn method. Other methods based on the penetration force have been developed, but these are more expensive because they use automated devices.

Finally, the proposed indentation test is more suitable for laboratory uses because industrial clays always present surface heterogeneities that could cause errors in plasticity measurements. As the indentation areas are very small, at least five or more measurements are needed to obtain a reliable value of plasticity.

## References

Al-Shayea, N.A., 2001. The combined effect of clay and moisture content on the behavior of remolded unsaturated soils. Engineering Geology 62 (4), 319–342.

Ancey, C., 2007. Plasticity and geophysical flows: a review. Journal of Non-Newtonian Fluid Mechanics 142 (1–3), 4–35.

Atterberg, A., 1975. Plasticity of clays: US ARMY C.R.R.E.L. International Journal of Rock Mechanics and Mining Science & Geomechanics Abstracts 12 (7), 91.

Baran, B., Ertürk, T., Sarikaya, Y., Alemdaroğlu, T., 2001. Workability test method for metals applied to examine a workability measure (plastic limit) for clays. Applied Clay Science 20 (1–2), 53–63.

Basma, A.A., Al-Homoud, A.S., Al-Tabari, E.Y., 1994. Effects of methods of drying on the engineering behavior of clays. Applied Clay Science 9 (3), 151–164.

Basma, A.A., Al-Homoud, A.S., Malkawi, A.I.H., Al-Bashabsheh, M.A., 1996. Swelling-shrinkage behavior of natural expansive clays. Applied Clay Science 11 (2–4), 211–227.

Bergaya, F., Theng, B.K.G., Lagaly, G., 2006. Handbook of Clay Science. Elsevier, Amsterdam.

Burst, J.F., 1991. The application of clay minerals in ceramics. Applied Clay Science 5 (5–6), 421–443.

Christaras, B., 1991. A comparison of the Casagrande and fall cone penetrometer methods for liquid limit determination in marls from Crete, Greece. Engineering Geology 31 (2), 131–142.

Conil, N., Djeran-Maigre, I., Cabrillac, R., Su, K., 2004. Poroplastic damage model for claystones. Applied Clay Science 26 (1–4), 473–487.

Dafalias, Y.F., Manzari, M.T., Akaishi, M., 2002. A simple anisotropic clay plasticity model. Mechanics Research Communications 29 (4), 241–245.

Doménech, V., Sánchez, E., Sanz, V., García, J., Ginés, F., 1994. Assessing the plasticity of ceramic masses by determining indentation force. Proceedings of the III World Congress on Ceramic Tile Quality. Official Chamber of Commerce, Industry and Navigation, Castellón, Spain.

Fenili, E.P., Biz, F., Boa Hora, P.R.M., Madeira, R., Modesto, C.O., Bernardin, A.M., 2004. Determination of clay plasticity: indentation method vs. Pfefferkorn method. VIII World Congress on Ceramic Tile Quality. Official Chamber of Commerce, Industry and Navigation, Castellón, Spain, pp. 47–53.

Kolmayer, P., Fernandes, R., Chavant, C., 2004. Numerical implementation of a new rheological law for argillites. Applied Clay Science 26 (1–4), 499–510.

Malkawi, A.I.H., Alawneh, A.S., Abu-Safaqah, O.T., 1999. Effects of organic matter on the physical and the physicochemical properties of an illitic soil. Applied Clay Science 14 (5–6), 257–278.

Murray, H.H., 1991. Overview: clay mineral applications. Applied Clay Science 5 (5–6), 379–395.

Peters, J.F., 1991. Determination of undrained shear strength of low plasticity clays. International Journal of Rock Mechanics and Mining Science & Geomechanics Abstracts 28 (1), 13.

Sánchez-Girón, V., Andreu, E., Hernanz, J.L., 2001. Stress relaxation of five different soil samples when uniaxially compacted at different water contents. Soil and Tillage Research 62 (3–4), 85–99.

Schmitz, R.M., Schroeder, C., Charlier, R., 2004. Chemo-mechanical interactions in clay: a correlation between clay mineralogy and Atterberg limits. Applied Clay Science 26 (1–4), 351–358.

Yu, H.-S., Khong, C., Wang, J., 2007. A unified plasticity model for cyclic behaviour of clay and sand. Mechanics Research Communications 34 (2), 97–114.