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Review Article

Measuring the plasticity of clays: A review

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ABSTRACT

Plasticity is the outstanding property of clay–water systems. It is the property a substance has when deformed continuously under a finite force. When the force is removed or reduced, the shape is maintained. Mineralogical composition, particle size distribution, organic substances and additives can affect the plasticity of clays. Several measuring techniques and devices were proposed to determine the optimal water content in a clay body required to allow this body to be plastically deformed by shaping. In this review, methods of evaluating the plasticity of clay–water systems are presented. Despite the advance in the theory of the plasticity and the methods of measurement, a common procedure for all types of materials does not exist. The most important methods are those that simulate the conditions of real processing.

1. Introduction

Plastic behavior involves many areas of science and engineering and has applications in various materials, such as soils, clays, concrete, plastics and metals. In the beginning, the concept of plasticity was used to explain and to characterize the rheological behavior of materials in the solid or liquid state.

Research on plasticity began with the studies of Coulomb in the 18th century (Smith, 2006) on the stability of piles and embankments. In the last century, the work of Mohr served as a base of some concepts currently used such as elastic and plastic deformation, yielding (critical state), shear localization and post-failure behavior (Ancey, 2007).

Plasticity in the processing of clay-based materials is a fundamental property since it defines the technical parameters to convert a ceramic mass into a given shape by application of pressure (Norton, 1938; 1974; Moore, 1963; 1965; Astbury et al., 1966; Singer and Singer, 1979). Plasticity, in this case, and particularly in clay mineral systems, is defined as "the property of a material which allows it to be repeatedly deformed without rupture when acted upon by a force sufficient to cause deformation and which allows it to retain its shape after the applied force has been removed" (Perkins, 1995). A clay–water system of high plasticity requires more force to deform it and deforms to a greater extent without cracking than one of low plasticity which deforms more easily and ruptures sooner (Brownell, 1977).

The plasticity of clays is related to the morphology of the plate-like clay mineral particles that slide over the others when water is added, which acts as a lubricant. As the water content of clay is increased, plasticity increases up to a maximum, depending on the nature of the clay. Clay workers are accustomed to speak of "fat" or highly plastic clay such as ball clay or "lean", relatively non-plastic clay such as kaolin, but it is very difficult to express these terms in measurable quantities. In the industry, plasticity is also referred to as "extrudability", "ductility", "workability" or "consistency" (Händle, 2007).

Reed (1995) uses the term "consistency" referring to states of ceramic raw materials, namely dry powder, granules, plastic body, paste and slip, which are dependent on the liquid content. Fig. 1 presents the apparent shear resistance as a function of the water content for a typical clayish material. When water is added to dry clay, the first effect is an increase in cohesion, which tends to reach a maximum when water has nearly displaced all air from the pores between the particles. The minimum amount of water necessary to make clay plastic is commonly called the "plastic limit" (PL). Addition of water into the pores induces the formation of a fairly high yield-strength body that, however, may crack or rupture readily on deformation.

A plastic clay body can withstand the addition of considerable amounts of water, passing through a stage in which it remains dry to the fingers and is easily molded. As the water content increases, the clay becomes a paste, in which the yield strength steadily diminishes. The clay becomes wet and sticky to the fingers and can no longer maintain a molded shape. The water content which corresponds to

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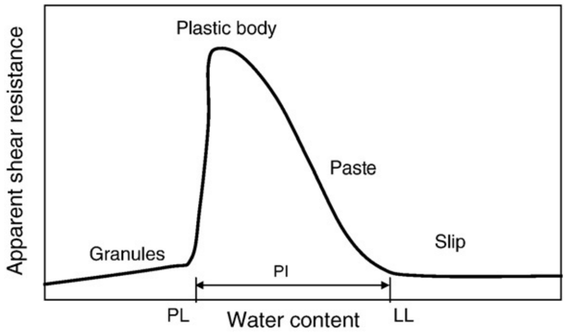

Fig. 1. States of consistency and plasticity limits of clays (adapted from Reed, 1995), PL = plastic limit, LL = liquid limit, PI = plasticity index.

this state is called "liquid limit" (LL). With still higher water contents, the system becomes a dispersion (slurry or slip). The difference in the water amounts at these two limiting points, related to the dry mass of the clay, is expressed as the "plasticity index" (PI), according to Fig. 1.

In traditional clay containing ceramic materials, the measurement and control of the plasticity are needed to characterize the system and to optimize the conditions of the processing (Ribeiro et al., 2005).

Factors influencing plasticity may be related to the clay itself or to the molding process (Henry, 1943; Carman, 1949; Marshall, 1955). Clay-related factors are moisture content, mineralogical composition, particle size distribution, type of exchangeable cations, presence of salts and organic material (Talwalkar and Parmelee, 1927; Wilson, 1936; Whitaker, 1939; Lawrence, 1958; West and Lawrence, 1959; Dumbleton and West, 1966; Barna, 1967; Onoda, 1996; Schmitz et al, 2004; Bergaya et al., 2006). Process-related factors are application of pressure, temperature and characteristics of water and additives used (Jefferson and Rogers, 1998; Malkawi et al., 1999; Ribeiro et al., 2004; Uz et al., 2009; Zentar et al., 2009). A deeper discussion on the role of clay composition and processing parameters on plasticity is beyond the scope of this review.

In this review, techniques commonly used for assessing the plasticity of clays are presented and discussed.

2. Measuring methods

There are several methods for measurement and characterization of the plasticity of a clay body. The experimental determination, in some cases, is operator dependent, which in turn may produce different results when different methods are compared. Among these methods, Atterberg, Pfefferkorn, stress/strain curves, indentation and rheological measurements are the most used techniques (Table 1).

Atterberg and Pfefferkorn tests are widely used owing to the low cost of the equipment employed (Moore, 1965; Van der Velden, 1979; Bekker, 1981). The measurement is based on the moisture content at which the material has some arbitrarily defined consistency. In these tests, high moisture contents are associated with high plasticity and vice versa.

Rheometry (McCabe, 1960; Alfani and Guerrini, 2005), indentation methods (Doménech et al., 1994; Vaillant; 2008; Modesto and Bernardini, 2008) and techniques which evaluate the relationship between an applied force and the resulting deformation (Baran et al., 2001; Ribeiro et al., 2005) are also used for measuring the plasticity of clays. These methods are often more cost intensive due to the equipment used. Nevertheless, they can supply important parameters such as modulus of elasticity, yield strength, maximum deformation and rupture strength.

2.1. The Atterberg method

Albert Atterberg (1846–1916), a Swedish chemist and agricultural scientist, found that plasticity is a particular characteristic of clay. He defined the consistency limits, called Atterberg limits (Atterberg, 1911). According to his findings, there is a defined amount of water at which the clay is easily moldable. With lower moisture content, the body cracks when molded. The Atterberg plastic limit is the lowest water content (expressed in mass percent of the clay dried at 120 °C) at which the body can be rolled into threads without breaking (Bergaya et al., 2006). The Atterberg liquid limit is the water content at which the body begins to flow, using a specific apparatus (Fig. 2). The difference between both values is called the plasticity (or plastic) index (Fig. 3).

The liquid and plastic limits define the transitions between liquid and plastic behavior. Arthur Casagrande (1902–1981), an Austrian-born American civil engineer, standardized the method to determine such limits in soil consisting of clayish and non-clayish materials. These limits can give significant information about the behavior of clay (Jefferson and Rogers, 1998). Casagrande (1958) studied different types of soil and evaluated plasticity by the Atterberg limits.

Although it is the most used method to evaluate plasticity, the large number of variables involved hinders a detailed correlation of the parameters with the behavior of the clay. To solve this, Gutiérrez (2006) proposed a rigorous probabilistic approach according to a regression analysis as a technique to express the linear behavior of the Atterberg limits for a given soil.

Two methods for determining the liquid limit are standardized (ASTM D4318, 2005): multipoint or one-point test. The correlation on which the calculations of the one-point method are based may not be valid for certain soils, such as organic soils or soils from a marine environment. It is recommended to use the multipoint method in cases where higher precision is required. Due to the fact that the one-point method requires the operator to judge when the test specimen is approximately at its liquid limit, it is particularly not recommended for use by inexperienced operators. The method proposed by Atterberg has some advantages such as low cost and sensitivity. In spite of this, the method's lack of precision is a significant drawback, mainly in one-point method, which limits its use in controlling materials (Doménech et al., 1994).

Special care must be taken during the execution of the tests. The specimens must be thoroughly mixed and be permitted to cure for a sufficient period before testing. Erroneous results may be caused by

Table 1
Methods for evaluating the plasticity of clays.

Method	Atterberg	Pfefferkorn	Penetrometer	Capillary rheometer	Brabender rheometer	Tension versus deformation
Measuring principle	Molding	Impact deformation	Penetration	Pressure	Torque	Pressure
Parameters measured or calculated	PI (LL and PL)	Water content (mass percent)	Force	Viscosity, pressure extrusion, flow curve	Torque, shear stress, viscosity, extrusion head pressure	Tension, deformation
Speed	Low	Low	Average	Average	Average	Average
Reproducibility	Low	Average	Average/high	High	High	High
Cost	Low	Low	Average	High	High	Average
Standard	ASTM D4318 (2005)		BS 1377 (1990)			

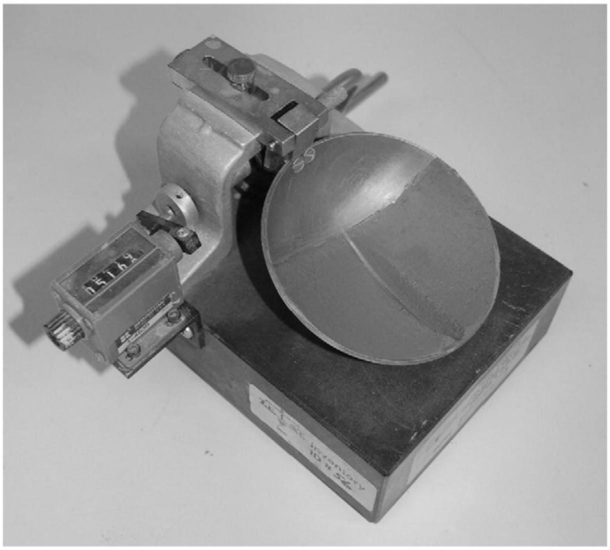

Fig. 2. Casagrande apparatus for measuring the liquid limit (Timely Engineering Soil Tests, 2010).

the loss of colloidal material when removing particles coarser than 0.42 mm (sieve #40) or by testing air-dried or oven-dried soils. Inaccurate determination of the water content would greatly affect the determined liquid and plastic limits if small, non-representative quantities of material are available for the water content determinations. Another source of errors can be the incorrect measure of the final thread diameter, or stopping the rolling process too soon.

2.2. The Pfefferkorn method

The Pfefferkorn 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 standard mass (Pfefferkorn, 1924). The results are normally expressed as graphs showing height reduction as a function of moisture content.

Measuring of plasticity according to Pfefferkorn is based on the principle of impact deformation (Fig. 4). A defined sample with a diameter of 33 mm and an initial height of 40 mm, produced either manually or by extrusion, is deformed by a free falling plate with a mass of 1.192 kg. The initial height is related to the impact deformation height, the result of which is the ratio of deformation. As a rule, this measurement is taken with bodies of varying moisture content. The ratios of deformation or the impact deformation heights (H_0, initial height; H_f, final height) are plotted against the moisture content (Fig. 5). The steeper the curve, the “shorter” the body, i.e. the more intensely the body will react to variations of the moisture content. The deformation heights for bodies to be extruded lie between ~25 mm for soft extrusion and ~37 mm for stiff extrusion (Händle, 2007).

Fig. 4. Pfefferkorn apparatus (Sassuolo Lab, 2010).

The Pfefferkorn method is widely accepted in practice and was originally developed for soft silicate ceramic materials. The method is less suitable for stiffer bodies, as usually processed in the advanced ceramics industry, as the low resolution at small deformation heights reduces reproducibility.

The Pfefferkorn test is laborious and time consuming. It requires changing the moisture content in order to reach 30% contraction. At the end of the test, the sample has to be dried. The main problems regarding plasticity determination using this method are related to the determination of the moisture, and to the relation between residual and sedimentary clays (Modesto and Bernardini, 2008).

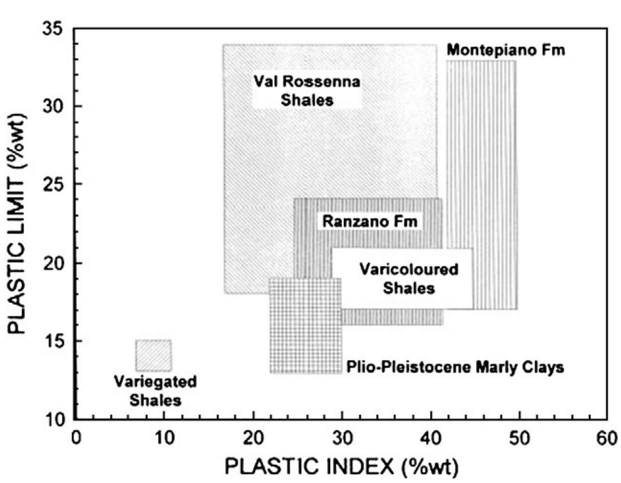

Fig. 3. Atterberg plastic index versus plastic limit of clay materials from Sassuolo, Italy (Dondi, 1999).

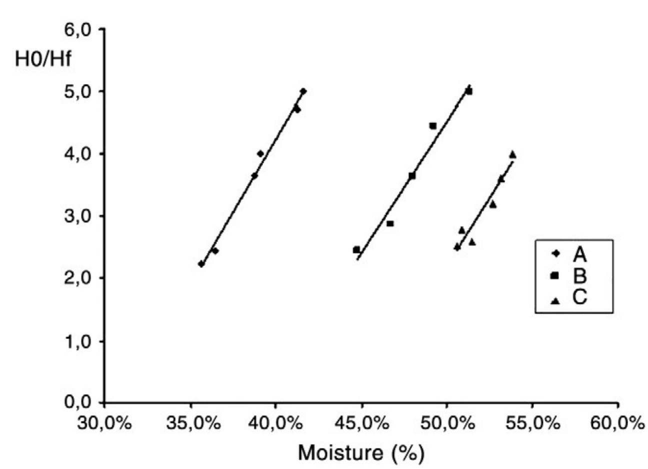

Fig. 5. Typical chart of Pfefferkorn for three clays.

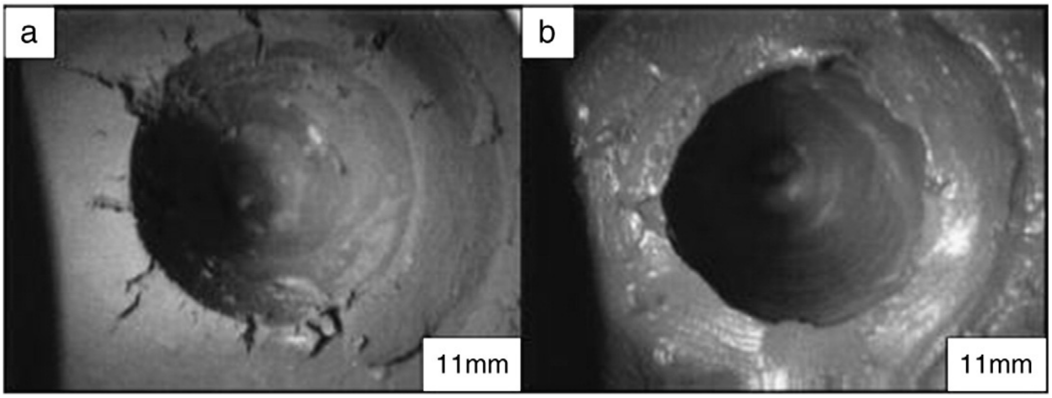

Fig. 6. Clay indentation, showing (a) too low water content and (b) an excess of water (Modesto and Bernardini, 2008).

2.3. Penetration methods

The penetration (or indentation) method is based on the measurement of the necessary force that a tool produces to make a mark in the test body. This mark, according to the geometry of the used tool, will serve to indicate the resistance of the mass to the penetration, and thus providing information about its plasticity. The measuring instruments of the penetration method devised for soil mechanics may be also related to those used for hardness measurement (Doménech et al., 1994; Händle, 2007).

In the fall cone test, a cone with an angle of 30° and total mass of 80 g is suspended above, but just in contact with, the clay sample. The cone is permitted to fall freely within 5 s. The water content corresponding to a cone penetration of 20 mm defines the liquid limit. The plastic limit is determined by repeating the testing with a cone of similar geometry, but with a mass of 240 g (Yu and Mitchell, 1998).

Some authors (Doménech et al., 1994; Feng, 2004) proposed the use of a sample-holding mold of circular cross section, so that the edge effects could be neglected. Doménech et al. (1994) used a cylindrical plate of 50 mm diameter and 50 mm height, while Feng (2004) used a smaller sample holder (20 mm diameter and 50 mm height). The specimen ring facilitates the sample preparation and increases the quality of the sample. For lower cone penetrations, when the clay sample is relatively stiff, the traditional specimen cup apparently reduces the measuring resolution of the plastic limit. As the fall cone is recommended in several standards for determining the liquid limit, it is advantageous to use it also for determining the plastic limit.

Vaillant (2008) investigated the utilization of a modified Vicat Apparatus (utilized in cement consistency analysis) to evaluate the plasticity of clays. He adapted an aluminum cone in place of a rod, reducing the weight to evaluate more accurately the liquid and plastic limits.

Modesto and Bernardini (2008) presented a method based on an indentation equipment. When penetration occurs, marks with cracks or plastic flow mean a lack of plasticity (low water contents, Fig. 6a), and when there are no cracks, a lack of consistency (high water contents, Fig. 6b). These extreme points correspond to the Atterberg plastic and the liquid limit. Adequate plasticity occurs when the marks do not present either cracks or extreme moisture and the wall formed is sufficiently smooth.

Measurements of plasticity with the penetrometer are considered to be more consistent, have better reproducibility, be easier to determine and less operator dependent (Feng, 2004). Some authors did not find significant differences between fall cone standard methods for liquid limit determination (Jefferson and Rogers, 1998; Vaz and Hopmans, 2001).

However, Benbow and Bridgwater (1993) reported some factors that can limit the accuracy of the penetration test. If the depth of penetration is too small, the accuracy is limited. If the sample is predominantly viscous rather than plastic, the penetration will depend on the time of penetration. Moreover, forces due to deceleration of the cone are not taken into account.

2.4. Capillary rheometer

The plasticity of extrudable materials can also be measured by a capillary rheometer. Various instruments are available, in the form of either single-bore capillary or twin-bore capillary rheometers. Using a pressure piston, the ceramic body is forced through a nozzle of a

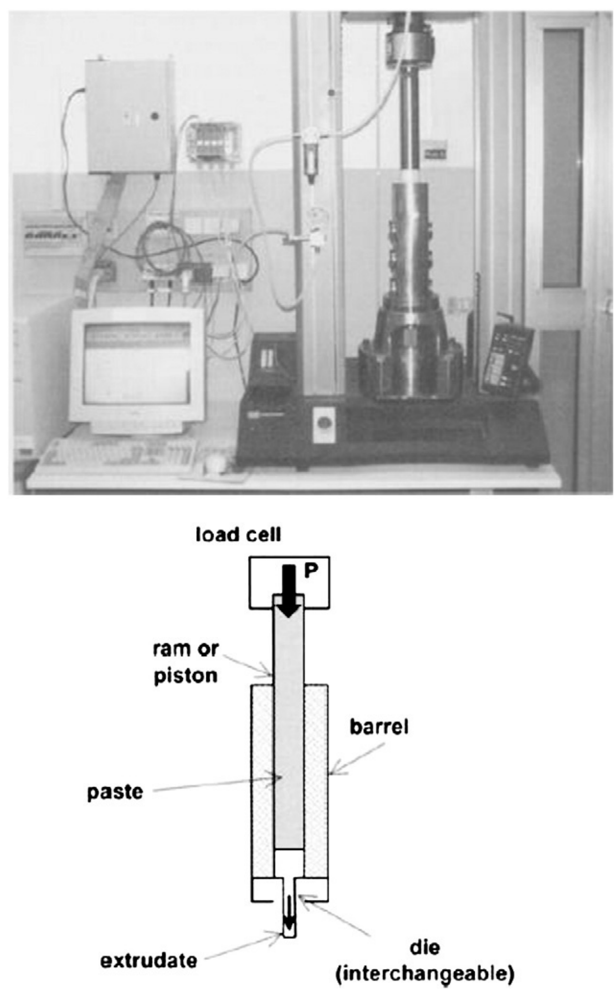

Fig. 7. Capillary rheometer (Alfani and Guerrini, 2005).

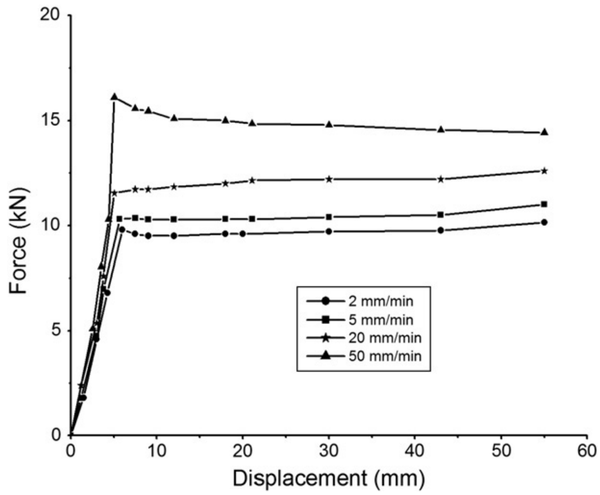

Fig. 8. Data obtained with a capillary rheometer (adapted from Alfani and Guerrini, 2005).

defined geometry at different feed rates (Fig. 7). The resistance of the ceramic body against the deformation in the nozzle causes a pressure drop within the capillary, which corresponds to a certain shear stress (σ). This pressure drop is the measured value, taken in the in-feed zone of the nozzle (Händle, 2007).

This test could be also applied for determining the material's apparent viscosity. To calculate the real viscosity of the materials as a function of the flow rate, the Mooney–Rabinowitsch correction is applied (Alfani and Guerrini, 2005) and a viscosity curve as a function of the shear rate (γ) is derived

$$\eta(\dot{\gamma}) = \frac{\sigma_w}{\dot{\gamma}_w}. \tag{1}$$

Fig. 9. Torque rheometer (C.W. Brabender Instruments, 2010).

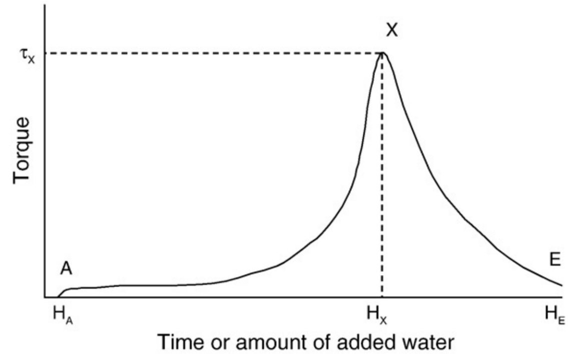

Fig. 10. Torque rheometer test (adapted from Sanchez et al., 1998).

According to the Bagley correction, the measured pressure ΔP_{tot} can be calculated by:

$$\Delta P_{tot} = \Delta P_{ent} + \left(\frac{\Delta P}{L}\right)_{die} L \tag{2}$$

where ΔP_{ent} represents the pressure drop in the static zone, and $(\Delta P/L)_{die}$ is the pressure drop along the die length. Eq. (2) shows that the pressure decreases along the die as the capillary length increases. The evaluation of $(\Delta P/L)_{die}$ is essential to determine the flow of the material inside the rheometer and to measure the viscosity. Typical capillary rheometer data are in Fig. 8.

The main advantage in this method is the possibility to evaluate more accurately the operational conditions on the extrusion process, as different geometries of the die can be used.

2.5. Torque rheometer

The torque rheometer or Brabender plastograph (McCabe, 1960) consists of a mixer with eccentric blades, inside which powdered clay is mixed with rising quantities of water by a proportioning system that allows keeping a steady liquid flow (Fig. 9). The torque reflects the change of consistency of dry powdered state to a plastic solid. The data represent the work required by the motor to move the blades inside the sample at a constant rotating speed and are recorded as torque versus time or amount of water (Sanchez et al., 1998).

When water is added, a point (A) is reached at which the material's consistency starts to increase (Fig. 10) and reaches a maximum (τ_x). Adding more liquid, the consistency decreases. At point E, the solid is no longer plastic. In general, plasticity can be defined by the maximum relative consistency (τ_x) or the range of plastic behavior (He-Hx) (Sanchez et al. 1998).

An advantage of this method is to perform an extrusion test, by installing a barrel with an extrusion screw and several types of die in the rheometer, monitoring the force by a pressure transducer. This

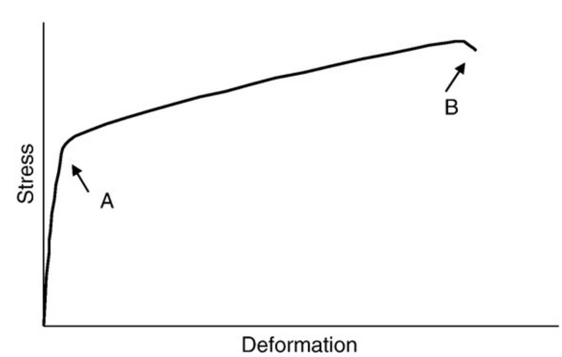

Fig. 11. Stress–deformation curve (adapted from Ribeiro et al., 2005).

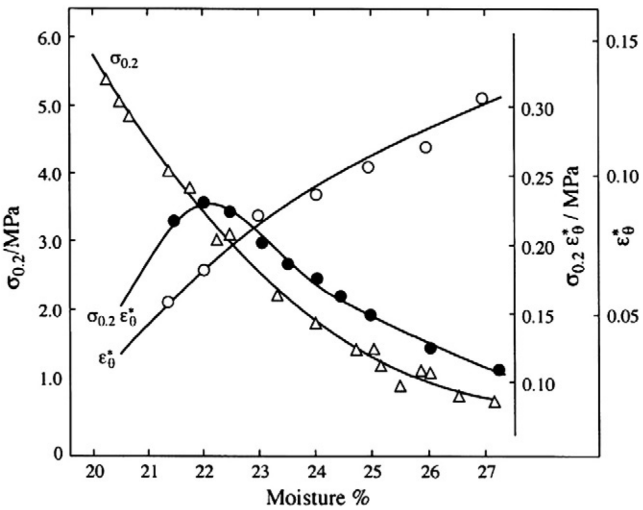

Fig. 12. Workability test (adapted from Baran et al., 2001).

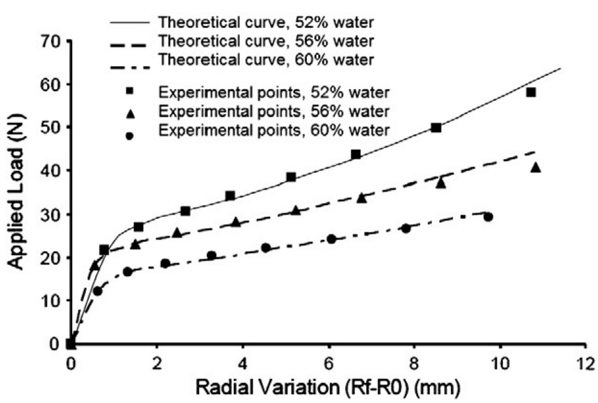

Fig. 13. Experimental and theoretical data obtained in stress–deformation tests of clay with different water contents (Flores et al., 2010).

apparatus works with small quantities of material, which has to be considered when passing into the industrial scale (Alfani and Guerrini, 2005). The test is very fast (<20 min), compared to traditional plasticity measuring methods.

2.6. Stress–strain curves

As for other types of materials, a compression test can be used to evaluate the plasticity of clays. The typical test curve gives information about the modulus of elasticity, yield strength, maximum deformation and rupture strength. As shown in Fig. 11, the material shows elastic behavior up to point A, then plastic behavior until reaching point B where cracks start to appear. Due to the small effective area, the tension increases quickly until the test body breaks (Ribeiro et al., 2005).

Some parameters obtained in the compression test are strongly influenced by the chemical composition and moisture of the clay. Therefore, this method shows a great potential to be used in the evaluation of the plasticity of clays used in extrusion. The high precision and reproducibility of the test make it possible to evaluate and to compare different clay–water systems.

Baran et al. (2001) applied the workability test for metals to measure the yield stress ($\sigma_{0.2}$) and the plastic tensile strain limit ($\varepsilon_{*\theta}$). The product of the two characteristic values ($\sigma_{0.2} \times \varepsilon_{*\theta}$) was defined as the workability. The variation of these three values as a function of the moisture content of the green bodies is shown in Fig. 12. From the maximum point of the $\sigma_{0.2} \times \varepsilon_{*\theta}$ curve, called the workability curve, the optimum moisture was determined (in this case, 22%).

Flores et al. (2010) modeling of plasticity of clays determined several parameters such as the coefficient of friction and the effective compressive stress (μ and $\overline{\sigma}$ in Eq. (3)), from the curves of the compression test (Fig. 13). The developed mathematical model is a potential and useful tool for the evaluation of clay-based materials with optimized properties for a given application.

$$F = -2\pi\overline{\sigma}\left[-\frac{h}{2\mu}\left(r_f + \frac{h}{2\mu}\right) + \frac{h^2}{4\mu^2}\,exp\left(\frac{2\mu r_f}{h}\right)\right] \quad (3)$$

where h is the final height of clay body sample, r_f is the final sample radius.

This method, where the clays are compressed until the cracks appear (Fig. 14), seems to be more precise and independent of the operator ability. It is faster to evaluate diverse types of clay bodies and supplies parameters to specify the extrusion process (Andrade et al., 2010).

2.7. Other methods

According to Linseis and Hofmann's method (Händle, 2007), the materials to be extruded at different moisture contents are forced through a nozzle of approximately 1 cm^2 cross section by means of a piston extruder, and the shearing strength required for this process is determined. The column is subsequently torn apart and the tear resistance measured. The degree of plasticity is the ratio of tear resistance to the shear strength. Highly plastic bodies are those which offer little resistance to deformation, but nevertheless they still have a high tear resistance.

In the Dietzel method (Händle, 2007), the same equipment is used as in the Pfefferkorn method. Rather than using a high deformation speed, the cylinder is compressed slowly, until cracks form. The

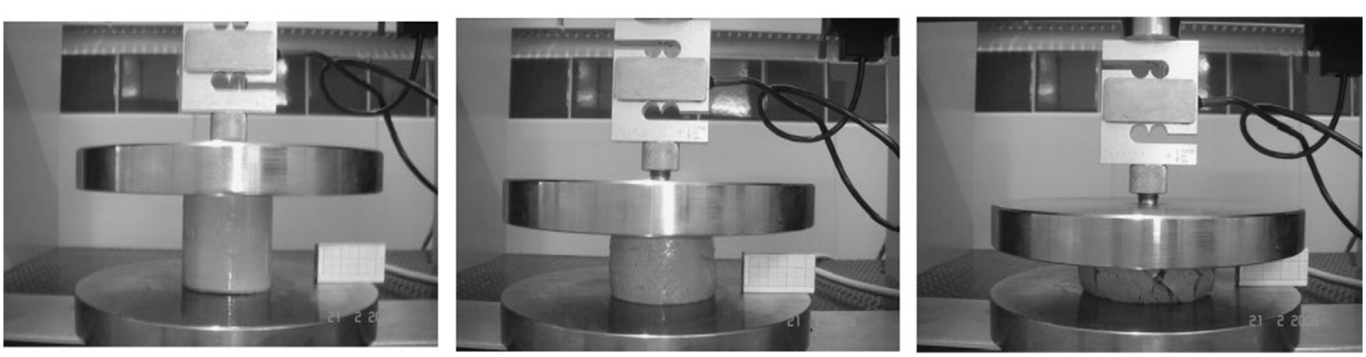

Fig. 14. Compression test of clays (Flores et al., 2010).

compression in percent of the original height is considered to be a measure of plasticity.

Based on the Atterberg method, the plasticity number according to Riecke (Händle, 2007) is considered to be the range between the roll-out limit and the make-up requirement, which is defined to be the moisture content at which the mass just stops sticking to a person's hand.

3. Conclusions

The plasticity concept is employed in many areas of engineering and science. Therefore, it is a hard task to choose a method that can be used for any type of raw material or processing condition. The main criteria that must be taken into account in the choice of the measurement method are the required information, the type of processing, as well as verifying the influence of one or more parameters on the plastic behavior of the clay body.

In laboratory scales, for developing new formulations, more than one method should be used. In the industry, where fast methods and low cost are required, the automated methods will be preferred for tests of raw materials or control process parameters.

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