DETERMINATION OF CLAY PLASTICITY: INDENTATION METHOD VERSUS PFEFFERKORN METHOD

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ABSTRACT

As a basis for developing the ceramic process, raw material selection plays a fundamental role in final product design. For correct raw materials selection, knowledge of all the properties regarding the raw materials should be obtained. Plasticity is one of the most important properties for moulding clay products. Nevertheless, current standard methods – the Pfefferkorn method is one of these – are not precise and reliable. They are based on qualitative information, which is extremely subjective and dependent on technical skills. The literature shows some attempts to improve the test method, which are all based on the force needed to indent a clay sample surface, due to water content.

In this study, the Pfefferkorn method and the Indentation method are compared. Four different clays were studied and the results have been compared using the water content (Pfefferkorn) and the force necessary to indent the samples (Indentation). The results show that the Indentation method is more reliable and accurate than the Pfefferkorn method. It is also faster and more practical.

Key words: *clay minerals; plasticity; indentation; Pfefferkorn test method.*

1. INTRODUCTION

Plasticity is a basic clay property that enables forming a plastic body. When a plastic body is subjected to the action of a force, it becomes deformed and conserves that form perfectly after the force application has ceased. Clays are predominantly made up of hydrated aluminium silicates. They are products of igneous, metamorphic and sedimentary rock alteration, mainly granites, feldspars and pegmatites, caused by the action of weather agents (rain, winds, etc.), providing them with their plastic characteristic.

However, there is a relation between geological formation and plasticity. There are argillites that possess a greater plasticity than others. This occurs because some interstitial materials are well preserved from the action of weather agents compared with other argillites. The same occurs with sedimentary clays which have been carried from the places where they were formed; during their transport they suffered a comminuting process and contamination with certain products, turning them into a more plastic material when compared with residual clays that not suffered transformations in transport. This is the case for example of residual clays that still contain unchanged rocks, which have little plasticity.

The mineralogical constitution of the distinct argillaceous materials can influence their plasticity. The clay mineral fraction and the types, amount and kind of auxiliary natural materials can change raw material plasticity. Some of the main clay minerals that make up industrial clays, which are responsible for the plasticity of the ceramic body, have been widely studied and are well known. The most important natural plastic minerals used in the ceramic industry are Kaolinite, Illite, Montmorillonite, etc.

Generally, plasticity is influenced by the planar basal distance of the clay minerals constituents; however, the bigger the clay mineral planar basal distance, the bigger is its water absorption capacity, making it necessary to perform separate analysis of each microcrystal. The bigger the planar distance values in the c axis, the bigger will be the mole number of water that the structure supports; e.g., Kaolinite has a c distance of 7Å and Montmorillonite, 14Å, so that the latter has a bigger water absorption capacity than the former.

Such spaces can be filled with water or other non-polar molecules and partially with other materials that diminish the microcrystal capacity of water absorption or, in other words, their plasticity. These materials can come from clays, as impurities, or be added to reduce the basal spaces, as in ceramic paste formulations. Amongst the main impurities with non-plastic properties, there are iron minerals (mainly Fe_2O_3), aluminium oxide (Al_2O_3), sodium and potassium feldspars, soluble salts (K_2SO_4, NaCl, Na_2CO_3, etc), calcium composites (calcite mainly), free silica, etc.

The cited impurities normally act as non-plastic materials. However, in specific cases, when their particle size is under $4\mu m$, i.e. a colloidal size, these materials can act as suspending particles, raising ceramic body plasticity when present with clay minerals. The relation between particle size and clay mineral plasticity is inversely proportional, because if particle size is bigger, their specific will be smaller, diminishing their plasticity due the lesser capacity of water absorption at their surface.

The presence of organic substances in clays derives from the decomposition of leaves and wood, appearing more intensively in sedimentary clays. Normally in colloidal form with a very small particle size, the organic substance has a high specific area and a good moulding capacity, providing the unctuous and plastic characteristics of clay minerals.

For plasticity measurement, the Pfefferkorn method is the most widely used. This method determines the plasticity coefficient of materials, i.e., the amount of water necessary to achieve a contraction of 30% in relation to the initial height of a test body, under the action of a standard weight. The results are normally expressed as graphs of height reduction as a function of moisture content.

The Pfefferkorn method results in plasticity analyses with some degree of error. Therefore, the analyses are not coherent or precise, resulting in an erroneous analysis of green and dry mechanical resistance of the ceramic products, as well as drying and forming characteristics.

The main problems of the plasticity determination using the Pfefferkorn method are related to moisture analysis, relation between residual and sedimentary clay, and test delay and difficulty. As already mentioned above, the Pfefferkorn method determines the raw material plasticity as water content and not as a resistance to penetration or plastic deformation. This fact cause mistakes in the plasticity analysis of argillaceous and similar minerals.

Regarding the relation between residual and sedimentary clays, sedimentary clays suffer a natural comminution during their transport process by water, reducing their particle size, and normally they have a greater amount of organic material. Therefore, sedimentary clays should be more plastic than residual clays. Tests carried out with these two clays, however, show that the Pfefferkorn method fails in this analysis, showing an inverted result, with residual clays being more plastic than sedimentary clays, which is unexpected.

Finally, the Pfefferkorn test is laborious and time consuming. It is necessary to successively remove and add the sample moisture in order to reach 30% height contraction in relation to the initial height of the test body, making this procedure very slow and tedious. Furthermore, to finalize the test it is necessary to dry the sample and to plot a graph for the final results.

Due to the errors and failures presented by the Pfefferkorn method, some new methods for plasticity determination have been presented in recent years with better accuracy and simpler procedures. This article presents a very simple method for plasticity analysis of clays. It uses a simple device, derived from indentation equipment. Sample preparation is the same for indentation analysis and the clay plasticity is determined by reaction forces.

2. MATERIALS AND METHODS

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

The test procedure is based on the application of an indentation force on a previously prepared clay mineral sample, promoting the perforation of the sample. Based on the sample reaction against the indentation force applied on it, the plasticity value is the direct reading of this reaction force measured on the digital display. The measured reaction force is the clay mineral resistance against the applied indentation force, i.e., its resistance to plastic deformation. If the perforation mark printed on the sample presents no kind of crack or any kind of plastic flow, the trial is considered valid and the force value displayed in the penetrometer device is the plasticity value.

The sample preparation was preceded by a 30min milling of 700g of raw material in a porcelain jar eccentric mill. The obtained ceramic suspension was dried in an infrared oven. The granules were disaggregated and sieved on a 500 mesh sieve. The dried powder was added with approximately 25 wt% water, and the paste was adequately homogenized. Finally, the paste was indented, adjusting the moisture content in order to obtain a smooth and uniform wall in the indented mark.

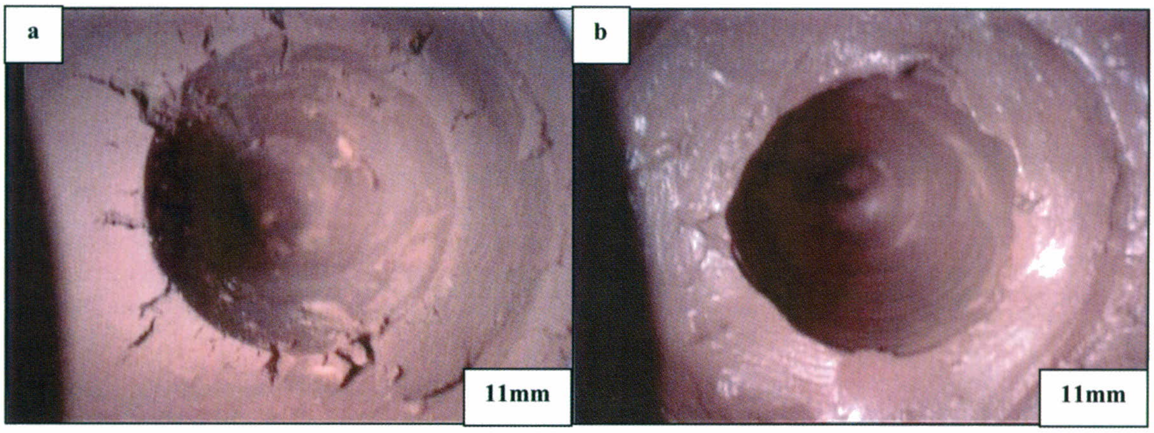

Figure 1. Clay indentation, showing (a) lack of water and (b) excess water.

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

After indentation, the marks can present two aspects in their sides: cracks, as a result of low moisture in the sample (figure 1a), and lack of consistency, as a result of excess moisture (figure 1b). Good plasticity occurs when the marks do not present any cracks or extreme humidity, the formed wall is sufficiently smooth and, therefore, this will be the humidity for the plasticity determination (figure 2).

After indentation, the next step is the preparation of the test body for the 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 the indentation test, is the plasticity value for the clay mineral being analyzed.

3. RESULTS AND DISCUSSION

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

CHEMICAL ANALYSIS											
R.M.[1]	SiO_2	Al_2O_3	K_2O	Na_2O	MgO	CaO	Fe_2O_3	TiO_2	P_2O_5	MnO	S[2]	Ppc[3]
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

[1]Raw materials; [2]S total; [3]Loss on ignition (1000°C).

MINERALOGICAL ANALYSIS								
R.M.[1]	Kaolinite	Illite	Quartz	Calcite	Potash Feldspar	Soda Feldspar	Magnetite	Accesories[4]
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

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

Clay P has low plasticity when compared with clay F because the latter contains more illitic phase than the former and probably thinner grain size distribution and average particle size (not proven, only assumed, based on its workability). Clay F originated from a Palaeozoic geological formation, being hillside residual clay with a low amount of organic material.

Based on its mineralogical composition, argillite R should be more plastic than E (25.0 wt% Illite). Argillite R is made up of a less well-preserved geological formation in comparison with E. However, argillite R possesses a larger amount of clay minerals and a smaller amount of non-plastics and impurities, when compared with E. As they have a similar size distribution, argillite R is more plastic than the E.

The raw materials were tested and analyzed by both methods, the Pfefferkorn and the proposed method, for comparison of possible relations and failures between them. The results are listed in table 2.

Analysing the results obtained by the Pfefferkorn method, it can be observed that for the two argillites and for clay P, the plasticity would be the same – approximately 24.5 wt% water. This shows the lack of accuracy of this method.

RAW MATERIAL	PLASTICITY	
	Pfefferkorn	Indentation
Clay F	31.0	19.9
Clay P	24.3	28.8
Argillite R	24.5	24.5
Argillite E	25.0	26.9
Units	wt% water content	Force (N)

Table 2. Results of the Pfefferkorn and the proposed method for the raw materials used in the tests.

The indentation method, which is the proposed method, based on the force necessary to penetrate each sample, shows differences among the plastic behaviour of the studied minerals. Clay P is the less plastic mineral, an aspect not shown with the Pfefferkorn method. This result is confirmed by the mineralogical analysis, which shows a smaller amount of clay minerals for clay P when compared with clay F and argillites R and E, regardless of their particle size distribution.

As the indentation method shows, clay F is the most plastic mineral, because the force needed to deform it is the smallest; its mineralogical analysis can confirm its plastic behaviour due the massive presence of clay minerals, in the form of Kaolinite and Illite (almost 70 wt% of its composition).

Finally, the Pfefferkorn test is a time consuming and laborious method. The time needed for the execution of the indentation tests using humidified samples takes at most 10 minutes; the Pfefferkorn method takes 3 to 5 hours to perform. It is necessary to dry the test bodies for the humidity determination, as well as to plot graphs for the determination of the plasticity coefficients.

4. CONCLUSION

The Pfefferkorn method is based on the amount of water, in order to determine the plasticity of ceramic raw materials. It is not coherent in its plasticity analysis regarding the raw materials mineral composition, nor is it accurate in the plasticity values, because it seems that the amount of water is not directly related to the plasticity, as an isolated matter. Particle size distribution, organic material content and other factors indeed affect plasticity, but methods based only on moisture content to determine plasticity are vague and imprecise.

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

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