CLAY QUALITY CONTROL IN TILE BODY PRODUCTION

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ABSTRACT

At present clay suppliers and spray-dried powder producers do not apply the same sampling and control methods, which complicates the definition of clay quality parameters. Furthermore, these methods generally do not satisfy the requisite criteria of speed, simplicity and reliability for suitable preventive control.

In this study, a sampling system was established in cooperation with six clay suppliers and spray-dried powder producers, which allows obtaining a representative lot sample. Inspection frequency was defined in terms of the variability of the analysed clay.

Clay quality parameters were defined as well as the necessary test procedures for verifying conformity with these parameters. The tests were classified in two groups according to testing speed and simplicity, designated basic acceptance control and supplementary acceptance control. To adequately evaluate the data obtained from these controls, a basic method is also proposed involving statistical control graphs.

Finally a clay sample preparation method is set out, which yields reproducible results that suitably represent the material's behaviour in the industrial process.

1. INTRODUCTION

The characteristics of the clays used for ceramic tile manufacture generally exhibit great fluctuations that can produce continuous changes in production unless the clays are suitably homogenised. However, sometimes in spite of the homogenisation progressive fluctuations in time can lead production parameters outside tolerance limits.

Consequently, to maintain these production parameters at optimum values, a homogenisation system needs to be established to obtain consistent supplied clay quality, while also performing a series of controls that allow monitoring the evolution of clay characteristics to establish whether they are suitable for the manufacturing process.

Obtaining a satisfactory product requires close cooperation between producer and raw materials supplier. This cooperation needs to be reflected in inspection and test methods that will ensure consistent clay quality.

This is unfortunately not the case in the ceramic floor and wall tile industry. Thus, most suppliers, which are family businesses, run no type of determination on the raw materials that they supply. On the other hand, although spray-dried powder producers generally, and some large clay suppliers in particular, run periodic controls, each company defines its own quality parameters so that there are no agreed universal control methods in place amongst suppliers and producers, which allow verifying the suitability of a raw material. Moreover, the systems and work methods employed vary greatly, while the equipment used is generally not calibrated, nor is the measurement's degree of uncertainty known.

As an example, Fig. 1 shows the results corresponding to a reject on a 63 μm mesh screen of a sample of Moró clay. The tests were carried out by 6 control laboratories belonging to spray-dried powder producers and raw materials suppliers. It can be observed that in spite of the simplicity of the test, the differences between the various laboratories were quite considerable.

On the other hand, sampling is often inappropriately done, since it not carried out according to any systematic method, nor are there documented procedures that clearly indicate the steps and methodology to be followed. It should furthermore be pointed out that clay is a difficult material to sample owing to its heterogeneity, variability in grain size, and batched supply (usually by truck). Consequently, the results found are usually incorrect, even though the errors produced by the analytical procedures may be small.

It should finally be mentioned that the sample preparation and control methods do not generally meet the requisite criteria for effective, fast, simple and reliable preventive control. Some companies currently need 12-24 hours just for sample preparation.

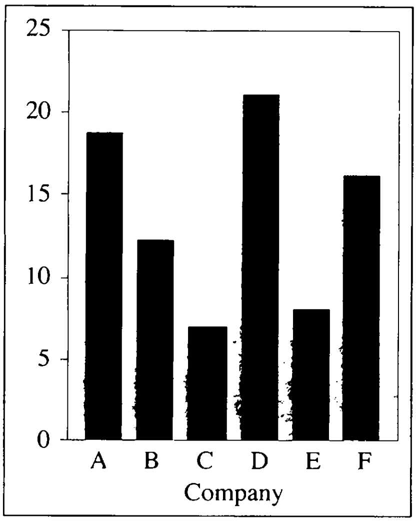

Fig. 1. Reject on a 63 μm mesh screen of a sample of Moró clay, obtained by 6 different control laboratories.

The present study summarises some of the results obtained in the research project "Harmonisation of the methodology for quality control of clays used in manufacturing tile bodies." The project, carried out in the period 1996-1997, was coordinated by the Instituto de Tecnología Cerámica (ITC), with the participation of the Spanish Tile Manufactures' Association (ASCER) as intermediate organisation, and the following cooperating companies: ARCITRAS, S.L; AZULIBER, S.A; GRES DE NULES, S.A; CERÁMICA SALONI, S.A; EUROARCE and TAULELL, S.A.

A Manual of Clay Quality Control Procedures is to be published in the last project stage, with a view to suitably disseminating project results amongst clay mining companies and spray-dried powder producers.

2. OBJECTIVES

In accordance with the above, the objectives of the present study were as follows:

- Drawing up a quality control plan for quality assurance of supplied clays.
- Definition of the characteristics that enable clay quality and conformity to be verified.
- Application of statistical control diagrams to clay raw materials.
- Definition of a sampling plan for obtaining representative samples of supplied clay lots.
- Establishing a preparation procedure for test samples.

3. PROPOSED QUALITY CONTROL PLAN

As set out in the introduction, quality assurance of the supplied raw material requires a clear agreement between supplier and producer, in which both define the methods or control procedures to be used in verifying conformity to specified product (clay) requirements. Documented procedures are required for such an agreement, which clearly set out the steps and methodology to be followed.

The raw materials acceptance controls to be run by both supplier and producer can be divided into two groups:

- *Basic control.* The tests involved in this control are characterised by their speed (maximum time 6 hours) and simplicity, and serve to initially verify raw materials conformity.
- *Supplementary control.* Generally, the tests involved in this more thorough type of control are to be run on samples that do not pass the foregoing control, in order to verify non-conformity. The tests may be more complicated and last considerably longer (over 24 hours).

On the basis of these levels of control, a chart is depicted in Fig. 2, which schematically illustrates the proposed quality control plan, involving the following steps:

- The clay supplier brings together clays from different mining operations and subjects these to grinding and homogenisation. Homogenisation is carried out by stockpiling.

- The clay mining company verifies the quality of the raw materials to be supplied to the producer, by running a *basic control*. These tests are conducted before or during stockpiling.

- If the raw material meets the agreed requirements, the sampled lot can be mixed with other lots to form the pile that will subsequently be supplied to the customer. Otherwise, a *supplementary control* is required.

- If on running a supplementary control the sample is shown to comply, the sampled lot can be added to the stockpile; otherwise the material can only be added if mixing with other lots reduces existing deviations. In an extreme case, the material shall be rejected

- Upon reception, the user shall run *basic acceptance control*. The tests to be performed are identical to those carried out by the supplier.

- If the raw material meets the agreed requirements, homogenisation takes place by the usual stockpiling system, followed by incorporation into the production process; otherwise a *supplementary control* is to be run.

- Finally, if the lot sampled meets supplementary control requirements, it can be incorporated into the production process after homogenising; otherwise the composition formula needs to be modified or in an extreme case, the material shall be rejected.

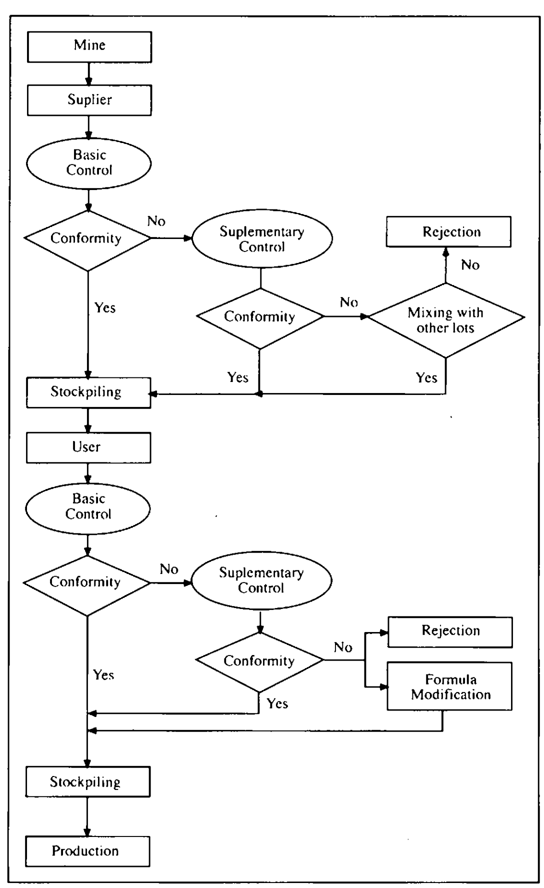

Figure 2. *Schematic of the proposed quality control plan.*

One of the key objectives in quality strategies is reaching a state of mutual confidence in customer-supplier relationships. Such an understanding can lead to the reduction or virtual elimination of inspections by the customer. Going a step further, the supplier might even reach a quasi user company department status. In this situation, the supplier fulfils an important role in the customer's plans for ongoing improvement.

Unfortunately, at present such objectives are rarely encountered among clay suppliers and users. Consequently, in view of the absence of any such quality control plans amongst suppliers, the users need to assume all clay quality control themselves.

4. QUALITY CHARACTERISTICS

After defining the two types of controls to be employed in verifying conformity or non-conformity of a given lot of clay, it is necessary to select the tests to be used in each control. The characteristics measured or assessed by these tests will define raw materials quality. These defining characteristics will be referred to hereinafter as *quality parameters*.

4.1. BASIC CONTROL

Numerous characteristics are involved in-plant in production, so that a selection of characteristics is needed. In order to choose the tests that are to form part of basic control, priority must be attached to the characteristics that are most sensitive to physico-chemical changes in clay materials and that most affect finished product quality. At the same time, the simplicity and cost of the method, time needed to run the measurements, and reproducibility of results are also to be taken into account.

After several technical meetings with the group of companies that participated in the project, bearing in mind the above premises, agreement was reached on the tests that were to make up basic acceptance control. These were in turn divided into two groups, depending on whether the quality parameter was measurable (control by variables) or not (control by attributes):

a) Control by variables

- Moisture content

- Reject on a 63 μm mesh screen

- Carbonates content

- Loss on ignition

- Viscosity of a clay suspension.

- Determination of impurities: carbonate content of the reject at 63 or 125 μm.

As Table 1 shows, the data that can be rapidly obtained by these tests provide considerable information concerning the physico-chemical characteristics of the clays, and hence allow establishing whether the materials conform.

Qualitiy parameter	Clay physico-chemical characteristics
Reject at 63 µm	Clay mineral/quartz ratio, PSD*
Carbonates	Alkaline-earth carbonate content
Loss on ignition	Clay mineral, quartz and alkaline-earth carbonate content, nature of the clay mineral (illitic/kaolinitic)
Viscosity	Presence of soluble salts (sulphates) and clay mineral/quartz ratio

* Particle-size distribution.

Table 1.Relation between the tests included in basic clay acceptance control and certain clay physico-chemical characteristics.

b) Control by attributes

- Visual appraisal of the clay (colour, presence of impurities, etc.).
- Visual appraisal of the clay reject at 63 or 125 µm (presence of impurities).

4.2. SUPPLEMENTARY CONTROL

Besides performing the determinations involved in basic acceptance control, thorough clay characterisation can take place by performing the following tests:

- Determination of soluble sulphate content.
- Chemical and mineralogical analysis.
- Plasticity.
- Deflocculation curve.
- Dry bulk density.
- Linear shrinkage, water absorption and bulk density of the fired test specimens at various firing temperatures.
- Dilatometric analysis of the fired test specimen.
- Black core.
- Firing colour.

As described in the literature, many other tests could be included in the above list, however the conformity or non-conformity of a given lot of clay can usually be assessed by just a few of the tests listed. For instance, if after basic control of a clay, loss on ignition were found to fluctuate beyond admissible limits, simply determining plasticity and firing behaviour (linear shrinkage and water absorption as a function of temperature) would suffice to establish conformity.

5. STATISTICAL CONTROL DIAGRAMS

The data presentation and analysis procedure known as the control graphics method can be used to indicate when the variations recorded in the clay quality parameters remain within acceptable random limits. A graphic is involved, which records the evolution of a quality parameter or material property versus time. This allows controlling whether the characteristics of the parameter or property involved remain steady in time.

Fig. 3 depicts an example of a control graph. In this type of graph the following values are plotted:

- The mean value M of a given parameter, obtained from tests on at least 25 consecutive samplings.
- The upper and lower tolerance limits (LT_s and LT_i respectively), represented by two fine lines, one at each side of the mean value of the parameter involved. These indicate the maximum and minimum values of this parameter or property. When the quality parameters do not lie within these limits, using the raw material is likely to produce risky or uncertain conditions for satisfactory process operation and final product quality, so that this raw material may be rejected. The position of these bounds depends on the manufacturing process involved, and not on clay variability.
- The upper and lower statistical control limits (LC_s and LC_i respectively), represented by two dashed lines, are symmetrically located at each side of the mean value of the parameter involved. These limits help evaluate the degree of variation arising in a clay quality parameter, and are a function of the point values of this parameter. These bounds, unlike the former however, do not serve to determine whether a material complies with specifications, and are not used to accept or reject a given lot.

Statistical control limits are calculated from the following equation:

$$LC_s = M + 3S, \; LC_i = M - 3S$$

where M and S are respectively the mean value and standard deviation of the point values.

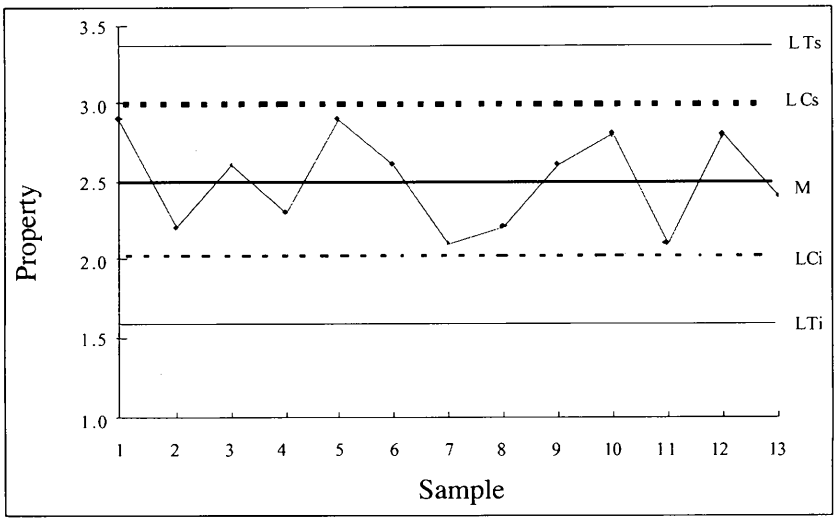

Figure 3. Statistical control diagram.

The presence of one or more points outside limits LC_s and LC_i is a first sign that the clay is no longer under control. As points beyond the limit are highly unlikely (probability of 2.7 per thousand), such extreme values are normally taken to have special causes, which require investigation. A point beyond the control limit usually indicates:

a) Alteration in the clay since the last sampling.

b) The measurement has been changed, as a result of a change in personnel or measuring instrument.

c) The control point or limit has been incorrectly indicated or has been improperly calculated.

d) Normal clay variability. This will rarely occur since the probability of a point falling outside the limits is very small, as indicated above.

For the above to be valid, the quality parameter studied shall fit a normal distribution. However, this assumption can not always be applied to clays. As natural raw materials are involved, their variability will largely depend on the characteristics of the deposit, as well as on the mining and homogenisation processes used.

Control diagrams should therefore not be used for clays in the same way as for other materials or products. An individual value is not the most important datum to be taken into account, unless excessive deviation is involved outside tolerance limits, but it is rather the comparison of this value with data from previous or subsequent tests. Thus, the existence of trends, even though all the points may lie within the set limits, can also be indicative of inadequate clay and warn of changes in the material. Consequently, the origin of the variation must be identified and corrected before the clay characteristics cross beyond tolerance limits.

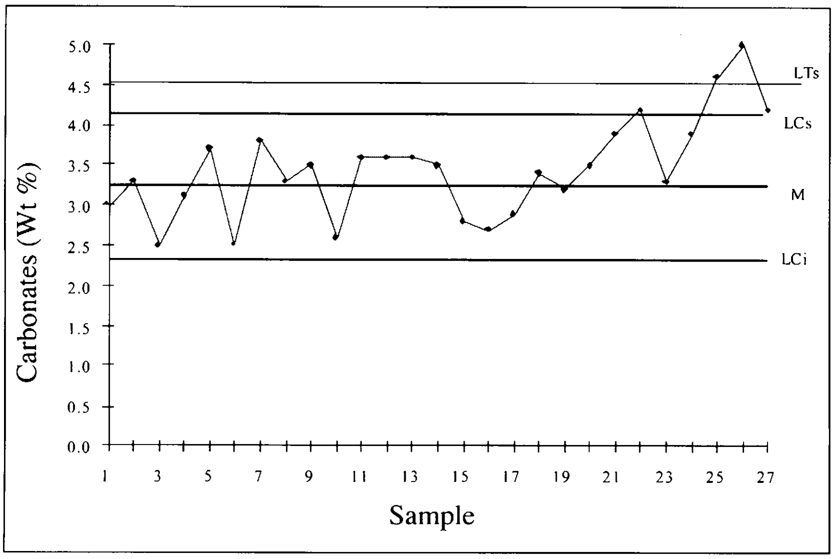

Figure 4. Example of a clay no longer under control.

Fig. 4 presents an actual example of a clay used for redware tile, which was no longer under control. It can be observed that after a period in which the results fluctuated around the mean value between the two control limits, the carbonate content progressively rose, lifting the clay outside tolerance limits.

6. HOMOGENISATION

Although the study was not intended as an in-depth study of the clay homogenisation process, it is convenient to briefly discuss this operation, as it directly affects clay quality control.

At present, homogenisation takes place almost exclusively by the linear layer stockpiling system. The method involves forming parallel, uniform layers of clay of greater or lesser height.

The effectiveness of these systems becomes clear in Fig. 5, which presents the sampling data on a single type of red-firing clay used by two companies for manufacturing stoneware floor tiles. Company (A) used no homogenisation system at all, whereas in company (B) stockpiling took place as mentioned. It can be observed that the fluctuations of the analysed parameter (carbonate content) were appreciably flattened by the homogenisation system.

Two precautions need to be taken to ensure the effectiveness of any homogenisation system. In the first place, clay characteristics must be determined prior to or during stockpiling, so as to be able to reject the lot or modify the composition formula (see Section 3) should this be necessary. Secondly, it needs bearing in mind that the creation of the stockpile is as important as its use. These are two independent operations, which should not occur concurrently in the same storage area. In the ceramic industry, it is common practice to have two stockpiles, one that is being formed and the other that is being used as illustrated in the schematic of Fig. 6. The clay may in turn come from a stockpile prepared in the mining area itself. In this case, the final degree of homogenisation is much higher.

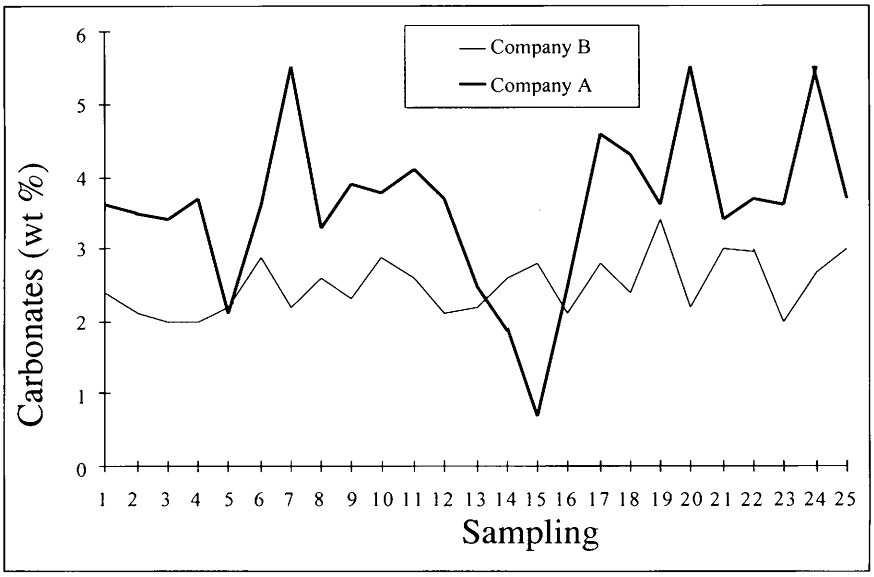

Figure 5. Variation of carbonate content with time for two companies (A and B). Company (A) used no homogenisation system, whereas company (B) used the linear layer stockpiling system.

Stockpile size depends on clay needs and available space. In general, a clay stockpile usually covers 2-4 month consumption. Consequently, the larger a company's production, or the larger the proportion included in the composition, the bigger the stockpile, whose size typically ranges from 10,000 to 200,000 t.

7. SAMPLING

7.1. DEFINITIONS

A sample is defined as the portion of the lot that is collected for analysis, in order to assess lot quality. Sample quantity shall in general be considerably larger than the amount required in the laboratory for testing but should not however be unduly large.

The sample is obtained by bringing together a certain number of sample units that have been systematically collected. In general, a sample unit may be a single object, a pair, a set, a length, area, volume, mass, etc. In the specific case of a raw material, a sampling unit is a quantity of material. On the other hand, the lot is the amount of clay that undergoes inspection. A lot may be composed of one or more consignments or subconsignments.

In order to avoid mistaken interpretations, it is important to correctly define the sample to be tested. As no specific studies are available on sampling clay batches, it was decided to undertake a study, within the framework of the project involved, to determine the most suitable conditions for conducting this type of sampling. The experiment design method was used, using batches of certain common clays in redware tile manufacture. The following variables were studied:

- Size of agglomerates making up the sample: large (>5 cm), intermediate (1-5 cm), and small (<1 cm.).

- Sample quantity: small (1kg), or large (10kg).

- Sampling units: few (2), or many (10).

On the other hand, the determined clay quality parameters were:

- Reject on a 63 µm screen.

- Carbonate content.

- Loss on ignition at 1000°C.

The study was performed by defining a portion of material of around 250 g as the sampling unit, which was taken from a random point in the lot (25 t clay).

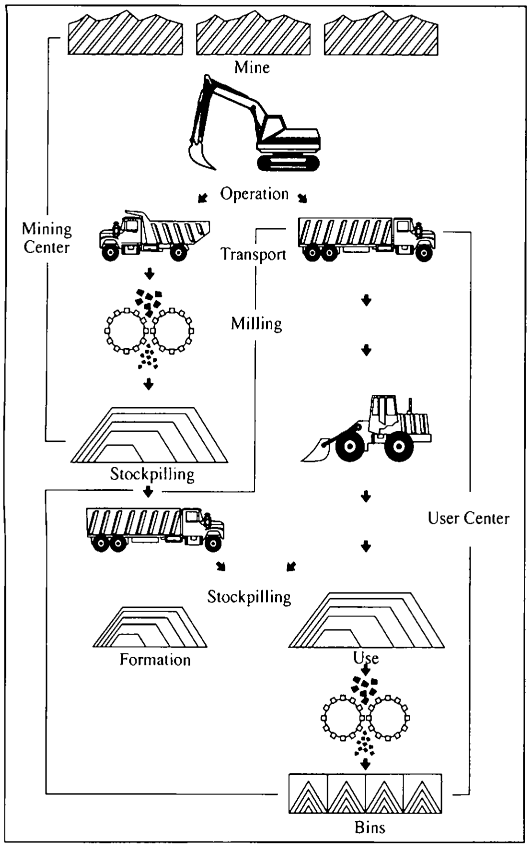

Figure 6.Usual homogenisation system for clays used in ceramic tile manufacture.

Fig. 7 depicts the influence of the sample's clay agglomerate size on the carbonate content of one of the studied clays. The reference value was obtained from a ground and homogenised 50 kg sample, taken from the feeding belt to the storage bins. It can be observed that the values were closer to the reference values when agglomerate size was less than 5 cm. It was also shown that the outcome was closer to the reference value when the number of sample units or sample amount was greater. The trends were similar for the other two quality parameters analysed (reject at 63 μm and loss on ignition), and for the other studied clays.

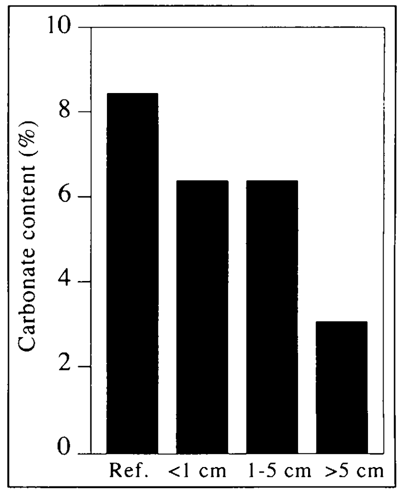

Fig. 7. Effect of sample agglomerate size on carbonate content.

In accordance with the study carried out, a sample was defined as a quantity of material comprising between 10 and 30 sampling units of 250g each (capacity of a small sampling shovel), with a maximum agglomerate size of 5 cm.

Defining the size of the lot to be inspected may become the hardest problem to solve. Generally, it is more convenient to form small lots, but inspection costs are greater and the number of tests larger. Therefore, as will be further discussed below, the size of the lot to be inspected will be defined in terms of user needs and characteristics of the material involved in the most convenient manner.

7.2. INSPECTION FREQUENCY

7.2.1. BASIC CONTROL

It appears obvious that more or less rigorous sampling plans should be used according to the behaviour of successive lots. If lots are progressively accepted, a less strict sampling plan may be implemented, since the supplier is providing greater assurance. If any lot is rejected, a stricter sampling plan should be applied in order to reduce the risk of accepting lots of an unacceptable quality for the process involved.

Sometimes a *concerted quality* arrangement may be applied. This is the case when the supplier guarantees to supply clays under fully controlled conditions. Supplies covered by this guarantee will pass directly into the manufacturing process without any previous inspection by the customer. Working under such conditions implies that any arising problem will have important economic repercussions, besides the supplier losing the agreed status. Customer-supplier relations of this kind are usually set down contractually.

Regardless of who carries out the control, whether it be the supplier, the manufacturer or both, the relation between lot size and stockpile size will be determined by the *level of inspection*. Hence, depending on the lot or quantity of material to be inspected, the following levels of inspection may be defined: Limited Inspection (when the amount of clay to be inspected equals 10-25% of the stockpile), Normal Inspection (equal to 5-10%), and Strict Inspection (1-5%).

To establish a level of inspection, the relationship shall be taken into account of the mean quality parameter value (M), its standard deviation (S), and the tolerance limits

(LT). The mean value and standard deviation are to be calculated from values determined beforehand in periodic tests, using the results corresponding to a minimum of 25 consecutive samplings. On the other hand, the tolerance limits LT for each quality parameter shall be fixed according to reason and experience, so that consulting the company's *technical report* will be quite useful.

Having calculated M and S, and knowing the tolerance limits, the following operation can be performed:

- If $3S \leq |LT\text{-}M| / 2$ for all parameters, Limited Inspection shall be applied.
- If $3S \ |LT\text{-}M|$ for some parameters, Strict Inspection shall be applied.
- In the remaining cases, Normal Inspection shall be applied.

Homogenised clay	**Non-homogenised clay**
Carbonates (wt%)	**Carbonates (wt%)**
2.4	2.5
2.1	2.4
2.0	2.3
2.0	2.6
2.2	1.0
2.9	2.5
2.2	4.4
2.6	2.1
2.3	2.8
2.9	2.7
2.6	3.0
2.1	2.6
2.2	1.4
2.6	0.8
2.8	0.1
2.1	1.4
2.8	3.5
2.4	3.2
3.4	2.5
2.2	4.4
3.0	2.3
3.0	2.6
2.0	2.5
2.7	4.4
3.0	2.6
M=2.5% 3S=1.2% LT_s=4%	M=2.5% 3S=3.2% LT_s=4%
$3S \geq (LT_s\text{-}M)/2=0.75\%$ $3S \leq (LT_s\text{-}M)=1.5\%$ Normal Inspection	$3S \geq (LT_s\text{-}M)/2=0.75\%$ $3S \geq (LT_s\text{-}M)=1.5\%$ Strict Inspection

Table 2. *Examples of level of inspection calculation.*

Table 2 presents two examples of level of inspection calculations by the user. In the first example, the clay had been homogenised beforehand by the supplier, whereas in the second case the same clay had not undergone such treatment, but had been sent straight to the customer. The effect of homogenisation on the stockpile allowed the level of inspection to drop from Strict to Normal.

The upper and lower limits of a given quality parameter may sometimes not be symmetrical with regard to the mean value. The two values M+3S and M-3S may not lie within the same range, or one of the tolerance limits may even not exist. This last situation is actually the case in the example given in Table 2 for the lower tolerance limit. In these cases the most unfavourable situation is to be considered and the highest inspection level should be chosen. On the other hand, the levels of inspection that are derived from the quality parameters do not necessary have to coincide. In these situations the most unfavourable outcome is to be selected.

Initially, until there are sufficient values to determine M and S, Strict Inspection is to be implemented. When more data are available, the level of inspection can be applied according to the relationship found between the various quality parameters. The level of inspection should be periodically reviewed, in order to modify it in terms of the evolution exhibited by clay variability.

7.2.2. SUPPLEMENTARY CONTROL

Just as in the case of basic acceptance control, supplementary control should be run with greater or lesser frequency according to the behaviour exhibited by successive lots.

In general, the periodicity with which supplementary control is conducted will be as follows:

- If the clay is under control, the frequency adopted shall be the one the technician considers most suitable. The sample to be analysed shall be taken from the volume of material received since the last verification control, and the rests left from the various basic control samplings can be used for this purpose.

- If one or more of the quality parameters determined in basic control exhibits non-conformity, supplementary control frequency shall be the same as basic control frequency. (See Section 3)

- When a different layer from the current clay or a new raw material is involved, the sample to be analysed shall be taken from at least a 25t volume of clay.

8. SAMPLE PREPARATION

The preparation of the clay sample for carrying out the various determinations plays a key part in obtaining reproducible results, which will therefore be comparable among different laboratories. The steps involved in sample preparation are milling and conditioning of the powder, pressing and firing.

As an example of the importance of sample preparation, Table 3 describes the effect of the type of milling on some clay quality parameters. In the first case, the clay had been dry milled in a hammer mill (starting screen of 1 mm); in the second case, the same clay had been wet milled in a laboratory ball mill, subsequently drying the resulting suspension, then milling the dry extract in the same hammer mill at the same starting screen aperture. Both methods are currently being used indiscriminately in clay control laboratories. It can be observed that the dry bulk density was greater when the test specimens were formed from powder prepared by the dry method, owing to the lower degree of milling. These differences were also observed in the properties of the fired specimens. Thus, the specimens pressed from the powder prepared by the wet method exhibited lower water absorption as a result of the powder's greater specific surface area. This fact, together with the specimens' lower bulk density led to greater firing shrinkage.

		Firing temperature = 1125°C	
Type of milling	**Bulk density (g/cm^3)**	**Linear shrinkage (%)**	**Water absorption (%)**
MVS	2.106	3.3	7.7
MVH+MVS	2.015	6.1	4.8

MVS: dry milling in a hammer mill
MVH: wet milling in a laboratory ball mill

Table 3. *Effect of the type of milling on certain characteristics of the material analysed.*

As the foregoing example reveals, milling is a decisive stage in sample preparation. Thus, with a view to obtaining results that could be compared and having more sensitive acceptance controls, which more reliably reproduce industrial behaviour, it is advisable to prepare the sample by the wet method. However, significant differences have also been found when wet milling systems with very similar characteristics were used. Thus, it can be observed in Fig. 8 that on wet milling the same raw material in two similar ball mills of different brands (mills 1 and 2), the reject found on a 63 μm screen after a given operating time was quite different. This fact can affect other clay quality parameters such as bulk density, water absorption, etc. In order to obtain results that can be compared with those of other laboratories, it is thus advisable to use reference materials that allow calculating the appropriate milling time in terms of the equipment used.

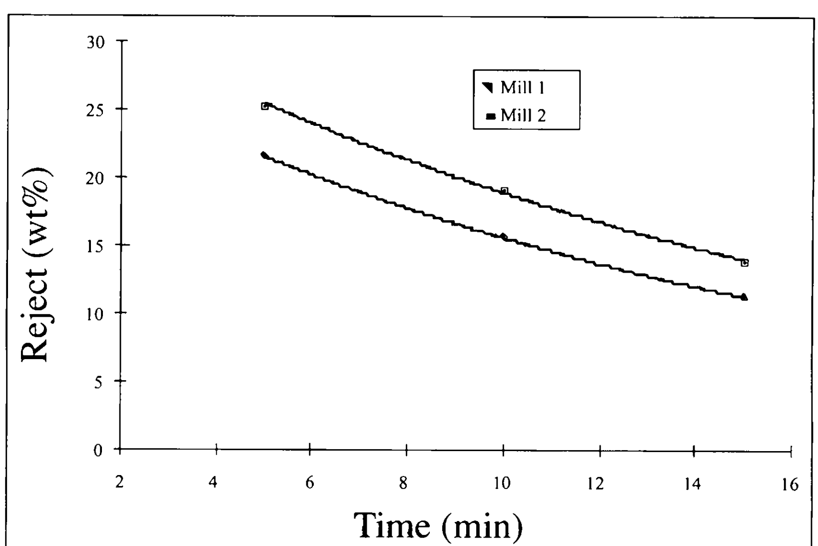

Figure 8. *Effect of raw material milling time on the reject obtained on a 63μm screen, for two similar ball mills of different brands.*

Other important steps in sample preparation are forming (usually by pressing) and firing. Suitable scientific instrumentation, ensuring accurate, reproducible data, is required to correctly perform these steps. Hence, kiln and press calibration and verification need to be conducted systematically in an organised manner.

After performing numerous tests at the laboratories of the companies co-operating in the project, in which the various clay sample preparation steps for clay quality control were studied, a procedure was proposed, which has been summarised in Fig. 8. Initially the clay needs to be crushed or dry milled until maximum agglomerate size does not exceed 5 mm. Subsequently, after taking the corresponding sample fraction, the material is wet milled or dispersed depending on the peculiarities of the industrial preparation process in which the composition will be used. The suspension obtained is dried and the resulting dry extract is dry milled again until agglomerate maximum size does not exceed 1 mm. The powder obtained is conditioned at a moisture content that depends on the characteristics of the clay (usually 5.5% dry base) and pressed (usually at 300 kg/cm^2). Finally, the test specimens are fired at a given peak temperature for a set dwell time according to the properties of the clay, the process it is to undergo, and the quality parameter to be determined.

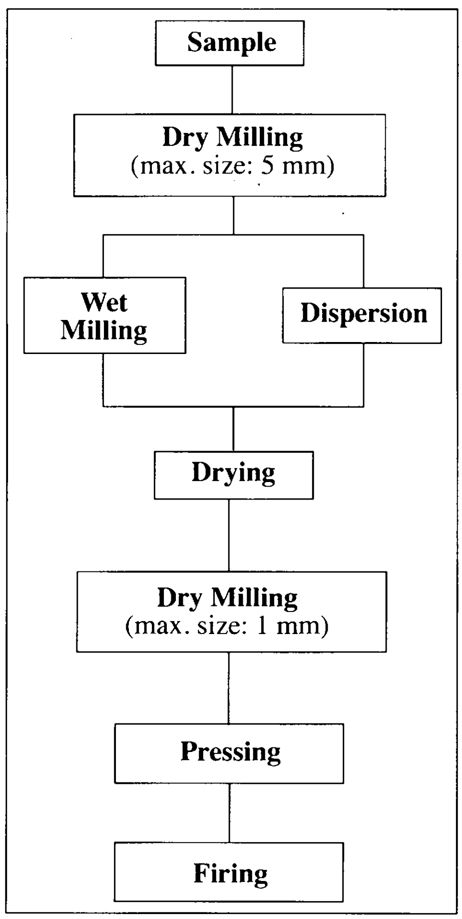

Figure 9. Proposed clay sample preparation method.

9. CONCLUSIONS

The ceramic tile sector does not at present have quality parameters or universal testing procedures for verifying the conformity or non-conformity of the clays used in preparing spray-dried powder. Many suppliers run virtually no controls on the clays that they supply; the spray-dried powder producers and certain large suppliers that do run periodic controls do so independently, and define their own quality parameters.

A quality plan has been drawn up in cooperation with six clay mining and spray-dried powder producing companies, which allows implementing suitable preventive control of clay raw materials.

The proposed quality plan essentially consists of controlling certain clay characteristics (*quality parameters*), after systematic, planned sampling (whose optimum conditions have also been described). Such controls can be carried out by the supplier and the customer and have been divided into two groups. First, a so-called *basic control* is run, involving fast, simple tests. If the material is found not to conform, a subsequent more thorough control termed *supplementary control* is conducted, which allows verifying more reliably whether conformity or non-conformity of the material is the case, and hence its acceptance or rejection. Moreover, a procedure has been proposed to adequately assess the outcomes of these tests, based on statistical control graphs.

The validity of the proposed methods (shortly to be published in a quality control manual) has been confirmed in cooperation with the above-mentioned companies, and its implementation in industry may thus be recommended for clay quality control.

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10. CONSULTED LITERATURE

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