

PRODUCTION DEFECTS IN CERAMIC TILING AND PAVING

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SUMMARY

This study reproduces material from a book of the same name, due to be published shortly; it is a catalogue of defects, compiled over 10 years in the Institute of Ceramic Technology and in the Research Association of Ceramic Industries.

The methods used to study the defects are described and a detailed examination of a concrete example is performed.

1. INTRODUCTION

This paper is an extract from a book which tries to bring together common defects that arise in the manufacture of ceramic paving and tiling. The book has been produced by compiling actual cases from the manufacture of these products and has involved more than 10 years work.

From the beginning, the study of production defects presents a challenge to human knowledge. One defect can arise from a group of circumstances which, coinciding to a greater or lesser degree, cause the apparition of the defect in an installation. As a result, the same defect seems to appear in many cases for diverse reasons, when what is really happening is that the conditions which gives rise to the defect are occurring simultaneously.

As knowledge of the ceramic process has developed, with the adaptation of Chemical Engineering to Ceramic Technology and with the availability of the most sophisticated methods, the possibility of studying and resolving these defects has increased enormously.

This systematic study of production defects attempts to act as a point of departure for subsequent studies, since delving into the causes and possible solution of a certain defect is an endless task.

To prepare this book an exhaustive bibliographic study was carried out, and the direct or indirect participation of a large number of professionals in the sector was availed of, thus resolving or helping to resolve many of the defects examined; archives of cases studied by the Institute of Ceramic Technology in the University of Valencia and by the Research Association of Ceramic Industries were also used.

2. QUALITY OF CERAMIC PIECES

The increase in competitiveness that has come about in recent years has been caused, on one hand, by a more exigent market, and, on the other, by an excess of products that differ little from each other.

To cope with this new situation, significant changes in production, technical and marketing systems have been implemented in the companies in the sector, in order to maintain or increase profits. The actions taken have generally been geared towards reducing production costs (personnel, energy,...) and, as far as possible, towards increasing the added value of the product (design, format).

In this sense, improvement of quality in the products, taken as a better adaptation of the product characteristics to the real needs of the client, has become an essential factor for re-activating the industry.

The age in which significant profits were obtained regardless of the quality of the product has passed into history. This situation caused an excess of confidence from which it has been necessary to evolve towards new ideas of quality improvement in the context of an overall profitability.

Investing in the improvement of quality has a cost which should be well considered, since in reality a product is sold through a balance of price and quality.

The increase in quality in the ceramic industry must be translated into immediate consequences (cost reduction, greater added value) of improving the relationship between the top quality pieces produced in the process and those which could be potentially produced.

These improvements should be consolidated, ensuring a homogeneity and continuity in the essential characteristics of the product.

Quality control is the instrument necessary to continuously and systematically check the variables and parameters that govern each stage of the ceramic process. This control must be dynamic and of a preventative nature in order to detect well in advance any changes that may arise and to quickly correct any deviations, using regulatory actions that will yield manufactured products within the predetermined limits of tolerance.

In each stage the process outlined in the following diagram will take place:

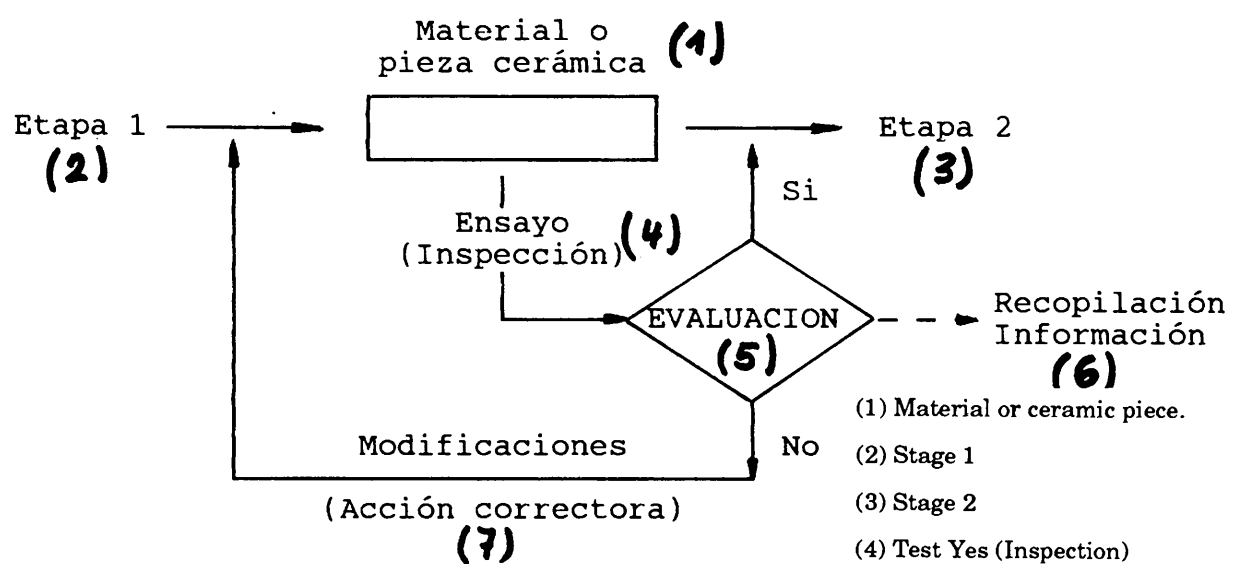


Figure 1 - Dynamic control of a system.

(1) Material or ceramic piece.

(2) Stage 1

(3) Stage 2

(4) Test Yes (Inspection)

(5) EVALUATION

(6) Compilation Information

(7) Alterations (Corrective Action)

The term quality control includes everything from the very definition of the product and the selection and receipt of raw materials, through all production phases, to the finished product and the verification of its suitable response to the client's requirements.

However, in spite of adopting all these production systems, there are certain imbalances in manufacturing processes, that are normally associated with changes in human factors, in raw materials, in machinery or power, which give rise to the apparition of production defects.

A defect can be defined as a lack or absence of qualities characteristic to a product, taking a standard piece as reference.

The rigorous study of production defects requires: an evaluation of the order of magnitude involved, a proposal of its nature and of the causes that gave rise to it, and an indication of the action needed to solve the defect. To finish the cycle it would be necessary to introduce a preventative control system for the variables that give rise to the most relevant defects.

In production plants, due to the large number of variables that exist and their margins of tolerance, a defect that appears is usually the result of an accumulation of various parameters that are outside their permissible limits.

Modern technology has developed towards automatic production control through the use of suitable measuring devices. Once the data obtained is processed and checked by a central unit, it must be converted into concrete instructions that will quickly reduce the cause of the defect and re-establish consigned values.

3. CLASSIFICATION OF DEFECTS

To carry out a systematic study of the defects that are produced in the manufacture of ceramic paving and tiling, a classification of the same must be produced.

This classification allows us: to more easily relate a detected defect to others of the same type, which have been previously studied and resolved; to establish the probable cause of the defect more quickly; to decide on the tests necessary to determine its origin; and, finally, to adopt an adequate solution to the problem.

3.1 Classification Criteria

The classification of defects in ceramic pieces can be carried out according to various criteria, depending on the facet of the defect that we wish to emphasize.

Defects can be classified according to origin, physical aspect, frequency, gravity, selection procedure, the area of the piece affected, or the process stage where the defect arises. The different methods of classification are complementary, together offering a more complete description of the defect.

3.1.1 Origin of defect

In any production process there are three essential factors that influence product quality: human resources, equipment or machinery, and raw materials. A primary classification can be developed considering in which one of these lies the principle cause of the defect. This criterion for classification, although unspecific, is useful from the point of view of an overall check at plant level. In general, the majority of ceramic defects are not exclusively caused by one factor.

3.1.2 Physical Aspect

This criteria gives an immediate classification based on the appearance of the defect. Classification includes cracks, desventados, eyes, dissymmetries, surface holes,....

The classification, for the most relevant defects, is in agreement with standard EN-98, "Ceramic tiles. Determination of dimensional characteristics and surface aspect." (UNE-67-098- 85), in which maximum permissible limits for each subgroup of tiles are established.

In standard ISO TC/189 WG 2 there is a classification similar to the european standard, but in this case definitions are provided for the following defects:

- Cracks - Any fracture on the body of the tile visible on the upper face, lower face, or on both.
- Crazing- Glaze fracture that appears as an irregularly traced crack.
- Lack of glaze - Small areas on the glazed side that are not glazed.
- Dips- (Dimples). Unintended depressions on the surface of the tile.
- Surface holes- Tiny holes on the surface of the glazed piece.
- Coloured stains or particles- Any unintended point that is in contrast with the glaze surface.
- Decoration defects- Marks or scratches. -Borders- Large accumulation of glaze at the edges of the piece.

Defects under the glaze:

- Alterations in the surface of the piece caused by unintentioned bulges, lumps, cracks, etc.....
- Bubbles- Small bubbles developed during firing that reach the surface, and that come from the expulsion of gas generated during firing.

These definitions are the same as those catalogued in the standard ASTM C-242-83 "Standard definitions of terms relating to ceramic whitewares and related products."

The fundamental inconveniences of this type of classification are: very little information about the origin of defects, the subjectiveness of assigning a class to a defect, and the high number of sections that are not adequately classified. As a result, the system is usually used as a sub- classification within other systems employing different criteria.

3.1.3 Classification according to Frequency

With regard to frequency of apparition, defects can be classified in three sections: sporadic, habitual, and permanent or inherent in the process.

The first of these are defects caused by an alteration in the process which exceeds the limit of admissible tolerances, these defects disappear within a short space of time.

Habitual defects are those which systematically appear in certain products or process stages. They can be avoided by establishing margins of variation using specific production parameters for each type of product.

In contrast, permanent defects can not easily be avoided, since they are governed by the production process itself. To eliminate them requires a substantial modification of the production system, affecting equipment as well as human resources and raw material.

3.1.4 Gravity of defect

According to this criteria, defects are classified as critical or serious defects, major defects and minor defects or irregularities.

Critical defects are those that do not allow for subsequent use of the piece, its residual value being zero.

Major defects reduce the durability and reliability of the piece, although they do not prevent its use. They require classification by category, which means a decrease in added value for products of lesser quality.

Minor defects or irregularities are more difficult to notice, they do not affect any of the essential characteristics of the piece and are acceptable as far as the quality-price ratio of the product is concerned.

3.1.5 Classification according to Selection Procedure

Classification according to this criterion includes: inherent support defects (those that influence mechanical resistance of the support such as desventados, flaking, fissures, etc....), dimensional defects (lack of planarity, dissymmetry, curving,...) and glaze surface defects which influence the aspect of the piece (shading, stains, surface holes, scratches,...).

This classification is normally used in selection lines of the finished product and is used to define the qualities of a piece as far as its commercialization is concerned.

3.1.6 Classification according to the zone affected

According to this criterion defects are classified as: defects that affect the whole piece, those that affect the support and those that appear in the glaze.

This method is interesting because the same defect, located in different areas of the piece, can be considered as a larger defect or an irregularity, since defects that affect the glaze will cause greater loss of value than those that only affect the support.

3.1.7 Classification according to process stage at which defect arises.

This classification is made on the basis of the stage from which the defect originates: pressing, glazing, firing, etc....

This criterion has a systematic ordering process, that places the defect within the productive system. In this way the defects that may be generated in each phase are known, and also with which process variables they are related. From this point of view, this classification provides a dynamic control that can prevent the apparition of defects.

The relationship that is drawn between the problem and its cause makes a more effective solution possible, acting directly on the parameters of the defect's stage of origin.

3.2 Classification proposal

In this study we propose to classify defects by their origin within the production process. This classification allows us to directly relate the defect with the changes (process variables of the stage at which it appears) that cause it, improving separation of variables and achieving a greater effectiveness in resolving the defect.

However, for this classification the origin of the defect must be located exactly, this implies greater complexity, since in a continuous production system there is an interaction between the different parameters, which makes individual analysis difficult.

It should also be added that in many cases a defect is not only due to one cause, but due to a combination of factors.

The following groups may be differentiated within this category:

3.2.1 Defects associated with raw materials

3.2.2 Defects in the preparation of the paste

3.2.3 Pressing defects

3.2.4 Drying defects

3.2.5 Glazing defects

3.2.6 Firing defects

Although the general classification proposed is based on process stages, it must be completed using the other criteria described above; the criteria are not exclusive and, instead, complement each other. So a defect can be from pressing, flaky, sporadic, serious, etc...

To quantify the relative importance of a defect and its incidence in production it was considered necessary to define the following parameters:

- Frequency: this term refers to the regularity with which a defect appears.
- Amplitude: measures the incidence of the defect, i.e. the percentage of production affected by it.
- Intensity: this parameter represents the gravity of the defect, i.e. that value a piece loses on account of this defect.

3.2.1 Defects from the presence of impurities in raw materials

The presence of random impurities near the surface of the support causes surface defects: basically surface holes or stains in the glaze. In the case of more serious impurities, massive defects such as efflorescence or blackheart are produced. The classification of these defects can be seen in Table I.

3.2.2 Defects in the preparation of the paste.

Errors in preparing the paste facilitate the apparition of cracks and dimensional variations: crazing, desventados, dissymmetries, bores, etc. The classification of these defects is given in Table II.

3.2.3 Pressing Defects

Pressing defects can be produced at any stage in this operation (filling the moulds, de-aeration, compressing and extraction).

Amongst the most frequent defects that appear in paving and tiling due to faulty pressing, the following can be emphasised: lack of pressing, dissymmetries, curving, cracks, acufados, different thicknesses, sticking to the mould, repressings dribbles, repegados, eyes, etc. These defects are given in Table III.

3.2.4 Drying defects

The most common defects that are produced here are cracks, warping and defects derived from the presence of residual damp in the piece at the end of the drying cycle. Classification of these defects is given in **Table IV**.

3.2.5 Glazing Defects

The most common defects in this area arise from bubbles in the slip, caused by an insufficient application of glaze or by lack of adherence between the glaze and support. These defects are translated as irregularities in the glazed piece, drops and lumps in the glaze, scratches on the glaze surface, etc (**Table V**).

3.2.6 Firing defects

Many defects appear in the firing stage that actually originate in previous stages of the process. Strictly speaking, firing defects are those associated with the phases of this stage (pre-heating, firing and cooling). This type of defect is described in **Table VI**.

4. METHODOLOGY FOR STUDYING DEFECTS

To conclusively resolve a production defect its repercussions in the product and the causes that give rise to it must be known as accurately as possible.

In general, when embarking on the study of a production defect, it will be necessary to follow a working method that allows for the orderly compilation of as much data as is available. The decisive phase of the study will be the separation of variables and their individual treatment, this will allow the really important factors to be distinguished from the more irrelevant ones.

The different aspects that must be considered in the study of a defect are presented on the following page in diagrammatic form (**Figure 2**).

Tabla I.

3.2.1. DEFECTOS ASOCIADOS A LAS MATERIAS PRIMAS		FORMA DEFECTO	I	A	F
3.2.1.1. Defectos puntuales debidos a impurezas presentes en las materias primas	- carbonatos	- pinchados, caliches	O	↑	↑
	- piritas	- pinchados, manchas en el esm.	O	↑	↓
	- pirolusita	- puntos negros	O	↑	↓
	- carbón	- pinchados	O	↑	O
	- mica		O	↑	↓
	- otros	-			
3.2.1.2. Defectos puntuales debidos a impurezas extrañas	- hierro u óxido de hierro	- coloración puntual	↑	↓	↑
		- bultos superficiales	↑	↓	↓
	- grasa	- protuberancias, cráteres	↑	↓	↓
	- caucho	- bultos, cráteres	↑	↓	↓
	- inquemados	- protuberancias, cráteres	↑	↓	↓
	- otros	-			
3.2.1.3. Defectos no puntuales producidos por otros tipos de impurezas	- materia orgánica	- corazón negro	O	↑	↑
	- sales solubles	- eflorescencias	↑	↑	↓

TABLE I

3.2.1 DEFECTS ASSOCIATED WITH RAW MATERIALS FORM OF DEFECT

3.2.1.1 Random defects due

- carbonates
- holes, saltpetre to impurities present
- pyrites
- holes, glaze stains in raw materials
- pyrolusite -black points
- carbon
- holes
- mica
- others

3.2.1.2 Random defects due

- iron and iron oxide
- random colouring to foreign bodies.
- surface bulges
- grease
- craters, protuberances
- rubber
- bulges, craters
- unburnt substances
- craters, protuberances
- others

3.2.1.3 Non random defects produced by other types of

- organic material
- blackheart impurities
- soluble salts
- efflorescence

Tabla II

3.2.2. DEFECTOS DE PREPARACION DE LA PASTA		FORMA DEFECTO	I	A	F
3.2.2.1. Defectos de composicion	- excesiva proporción de carbonatos	- calibres y falta de acoplamiento del esmalte	○	⬆	⬇
	- excesiva proporción de cuarzo y desgrasantes	- curvaturas - desventados - baja resistencia mecánica	○	⬆	⬆
	- composición variable - intervalo de cocción reducido - impurezas	- diversos defectos - descuadres y calibres	⬇	⬆	⬇
3.2.2.2. Defectos de preparación	- molienda insuficiente o excesiva	- modificaciones de propiedades en crudo, reactividad, textura, calibres.	○	⬆	⬇
	- aglomerados de elevado tamaño y humedad variable	- agrietamiento puntual del esmalte	○	○	○

Table II

3.2.2 DEFECTS IN PREPARATION OF THE PASTE FORM OF DEFECT

3.2.2.1 Composition defects -excessive proportion

- bores and lack of of carbonates glaze coupling
- excessive proportion
- curvature of quartz and
- desventados cleansing
- low mech resistances agents
- variable comp.
- various defects
- reduced firing time
- dissymmetries and bores
- impurities

3.2.2.2 Defects in preparation

- insufficient or
- changes in basic excessive grinding properties, reactivity, texture, bores
- large agglomerates
- random cracks variable humidity in the glaze

Tabla III

3.2.3. DEFECTOS DE PRENSADO	FORMA DEFECTO	I	A	F
3.2.3.1. Defectos asociados al material que alimenta a las prensas	- flojos	⬆	○	⬇
	- ahogado	○	⬆	⬇
	- ojos	○	○	⬇
3.2.3.2. Compacidad heterogénea en una misma pieza (defectos de alimentación)	- descuadre	⬇	⬆	⬆
	- luneta			
	- abombamiento			
	- acañados			
3.2.3.3. Compacidad deficiente de las piezas	- grietas	⬆	⬆	⬇
	- calibres	⬇	⬆	⬆
	- curvaturas	⬇	⬆	⬆
3.2.3.4. Defectos de extracción	- grietas	⬆	⬆	⬇
3.2.3.5. Otros	- rebabas	⬇	⬇	○
	- reprensados			
	- pegados a molde			

Table III

3.2.3. PRESSING DEFECTS FORM OF DEFECT

3.2.3.1 Defects associated with the material fed to presses.

-soft spots
-flaking
-eyes

3.2.3.2 Heterogeneous Compression in the same piece.

-dissymmetries
-luneta
-bulging
-marking

3.2.3.3. Deficient compaction in the pieces

-cracks
-bores
-curvature

3.2.3.4 Extraction defects - cracks

3.2.3.5 Others -dribbles -repressing -sticking to mould

Table IV

Tabla IV.

3.2.4. DEFECTOS DE SECADO	FORMA DE DEFECTO	I	A	F
3.2.4.1. Elevada velocidad de secado	- grietas	↑	↑	○
	- roturas	↑	↑	↓
3.2.4.2. Defectos producidos por un secado diferencial	- curvaturas	↓	↑	↓
	- grietas	↑	↑	↓
3.2.4.3. Humedad residual a la entrada del horno	- explosiones	↑	↑	↑
	- retraso en las desgaseificaciones	↓	↑	○

Tabla V.

3.2.5. DEFECTO DE ESMALTADO	FORMA DEFECTO	I	A	F
3.2.5.1. Defectos asociados al soporte	- sucios	↓	↑	↓
	- elevada T del soporte	↑	↑	↓
3.2.5.2. Defectos de formulación de esmaltes	- exceso de alcalinotérreos	○	↑	↓
	- esmalte excesivamente fundente o refractario	↑	↑	↓
	- baja proporción de elementos plásticos o ligantes	○	↑	↓
3.2.5.3. Defectos de preparación del esmalte	- molienda deficiente	↓	↑	○
	- burbujas en barbotinas	↓	↑	↑
	- condiciones reológicas inadecuadas	↓	↑	↑
	- modificación del color	↓	↑	↑
	- defecto de opacificantes	○	↑	↓
	- exceso de finos	↑	↑	↓

Table V

3.2.5 GLAZING DEFECTS FORM OF DEFECT

3.2.5.1 Defects associated with the support

- dirtiness removal of glaze
- high support temp. bubbles

3.2.5.2 Glaze formulation defects

- excess alkaline earths tinting, crystallization
- glaze excessively molten or rechupados, rough refractory surfaces,
- low proportion of lack of adherence plastics or binders to support,

3.2.5.3 Defects in glaze preparation

- insufficient grinding glaze removal, alterations when melting,...
- bubbles in slip craters, pockets,...
- inadequate rheological bubbles, changes in conditions applied layer
- modifications in colour shading
- defect in opacities transparency
- excess of purities cracks, glaze removal

Tabla V. (Continuación)

3.2.5. DEFECTOS DE ESMALTADO		FORMA DEFECTO	I	A	F
3.2.5.4. Defectos de aplicación	- aplicación por campana	líneas y ondulaciones en la capa de esmalte	○	○	⬆
	- aplicación a disco	gotas, desuniformidad de aplicación	○	○	⬆
	- aplicación por aerógrafo	gotas, grumos, desuniformidad de aplicación	○	○	⬆
	- serigrafía	rayas de espátula, salpicaduras de tinta	○	○	⬆
3.2.5.5. Defectos asociados a la movimentación de la pieza esmaltada	- funcionamiento anómalo de desbarbadores	recogidos, sucios, rayas	○	⬆	⬆
	- otros	rozaduras	⬆	⬆	⬆
		grietas	○	⬆	⬆

Table V (Continued)

3.2.5 GLAZING DEFECTS FORM OF DEFECT

- application by bell lines and undulations in the glaze layer
- disc application drops, non-uniformity in application
- application by aerograph drops, lumps, non- uniformity in application.
- serigraph spatula scratches, splashes of ink.

3.2.5.5. Defects associated with the movement of the glazed piece.

- anomalous operation of trimmers bunching, dirt, scrapes.
- others abrasions cracks

Tabla VI.

3.2.6. DEFECTOS DE COCCION		FORMA DEFECTO	I	A	F
3.2.6.1. Defectos de precalentamiento	- explosiones		⬆	⬆	⬆
	- desgasificaciones (corazón negro, pinchados)		⬆	○	○
	- condensaciones del horno		⬆	⬆	⬆
3.2.6.2. Defectos de cocción	- crudos		⬆	⬆	⬆
	- sobrecocidos		○	⬆	⬆
	- calibres		⬆	⬆	⬆
	- pegados		⬆	⬆	⬆
	- deformacion pirolástica		⬆	⬆	⬆
	- tonos		⬆	⬆	⬆
	- atmósfera reductora		⬆	⬆	⬆
3.2.6.3. Defectos de enfriamiento	- cristalizaciones, matizaciones		⬆	⬆	⬆
	- desventados		⬆	⬆	⬆
	- cuarteo y desconchado		⬆	⬆	⬆
	- curvaturas		○	⬆	⬆

Table VI

3.2.6 FIRING DEFECTS FORM OF DEFECT

3.2.6.1. Preheating defects

- explosions
- degassing (blackheart, surface holes)
- oven condensations

3.2.6.2. Firing defects

- unfired
- overfired
- bores
- adhesions
- pyroplastic deformation
- shading
- reducing atmosphere

3.2.6.3. Cooling defects

- crystallizations
- desventados
- crazing, chipping
- curvature

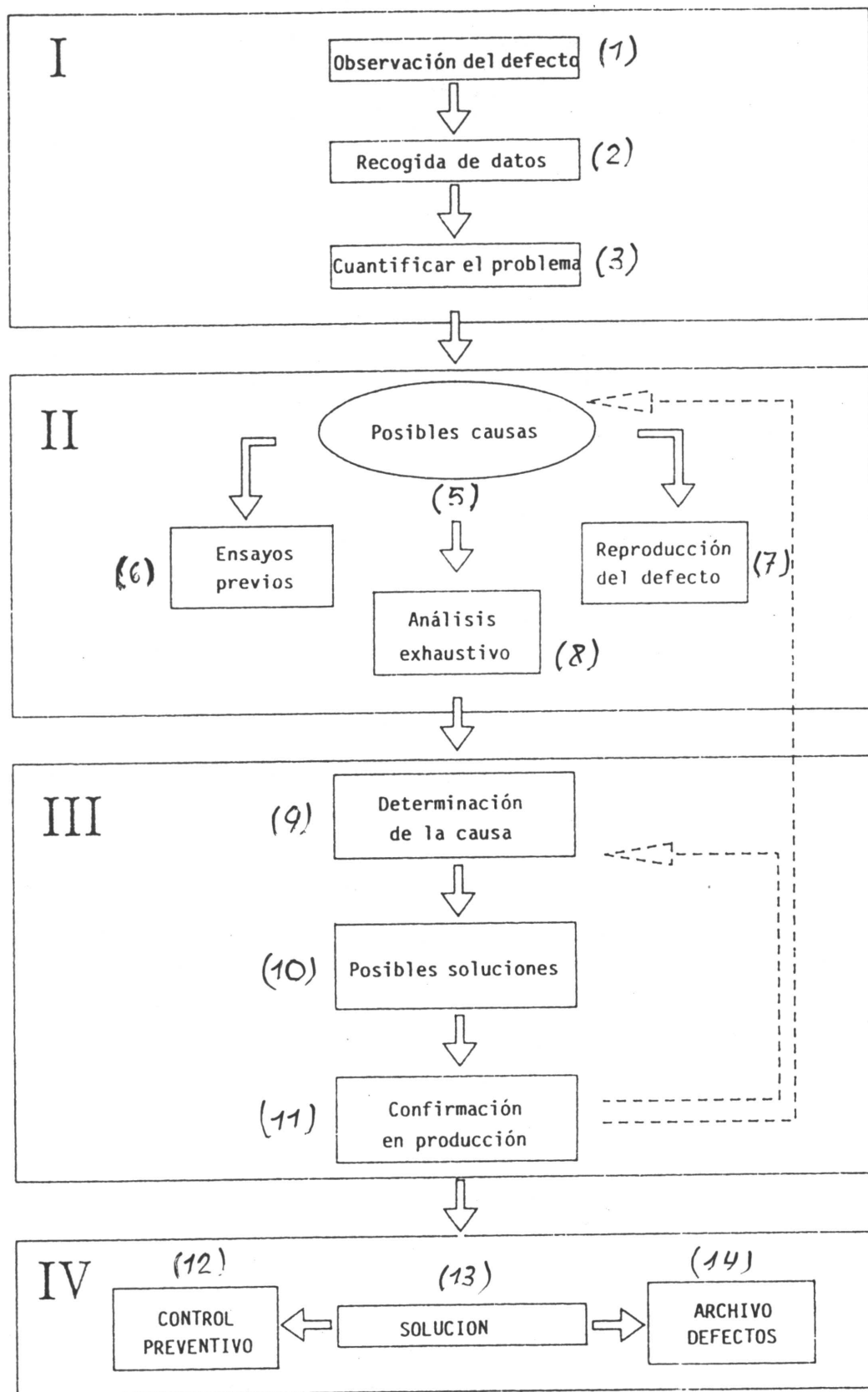


Figura 2.

- | | | |
|---------------------------|----------------------------|---------------------------------|
| (1) Examination of defect | (6) Previous Tests | (10) Possible solutions |
| (2) Compilation of data | (7) Reproduction of defect | (11) Confirmation in production |
| (3) Quantify problems | (8) Exhaustive Analysis | (12) Preventative Control |
| (5) Possible Causes | (9) Determination of cause | (13) Solution |
| | | (14) Defects file |

In all cases the procedure to follow starts with the detailed observation of the defect, together with the collection of data about the factors that have led to the apparition of the defect (change in one of the process variables, etc.), and the necessary quantification of the seriousness of the defect. It should be borne in mind that an isolated defect in a piece provides little information unless its history is known. This initial stage is of crucial importance and will be definitive in the establishment of a hypothesis about the possible causes of the effect, since the same defect can be produced by very different causes, as has been seen in the classification.

The second step involves the proposal of a hypothesis, on the basis of all the information gathered, on the possible causes of the defect; this should be progressively reduced to the minimum amount of factors possible after carrying out relevant tests. The tests will be, on one hand, of an analytic-deductive nature, from the most simple (screening, moisture, apparent density, calcination, magnetization, etc....), which can be performed in the production plant, to those tests that require more sophisticated equipment (X-ray diffraction, Thermic Differential Analysis, Chemical Analysis, Electronic Microscopy,...), which will only be used in the most exhaustive tests. Where it is possible an inductive procedure will be used, trying to reproduce the defect on a laboratory scale or by industrial testing.

Once the analytic determinations are completed, the third phase of the study begins, this will consist of the determination of the possible cause that gave rise to the defect. Solutions to the problem should then be posed on the basis of this supposition, these will be confirmed in production.

In many cases, once this point in the procedure is reached only partial solutions can be found to the defect, on account of the impediments that the different factors making up the production system cause. It must be remembered that the defect should be solved without causing losses in the other parameters and production process characteristics.

If the solution proposed is negative, a reconsideration of the hypothesis and initial suppositions should be made. In the affirmative case the solution must bear its validity with time. It is interesting, in quite serious cases, to establish some control system (product or process) that detects well in advance if the defect may be produced again, in order to be able to take action before it may occur.

Finally, it is important to have information obtained in other defect studies available, and suitably catalogued. As, on many occasions, defects appear in a cyclic or repetitive form (with small variations). On other, more obvious, occasions, this comparison will allow a significant number of possible causes to be discounted, which will hasten the study and its solution.

5. APPLICATION OF THE METHODOLOGY FOR THE STUDY OF DEFECTS TO A CONCRETE EXAMPLE: CRACKS DUE TO A RANDOM COMPRESSING FAULT.

5.1 Compilation of data.

5.1.1 Observation of defect

The first step in the study of a defect involves the meticulous observation of its appearance in order to obtain the maximum amount of information possible and to outline some initial hypotheses about the origin of the defect. The use of a magnifying glass or optic microscope is very useful for this primary observation.

In this case the defect appears as small 3 to 4mm long cracks, on the surface of the glazed piece. These cracks come from the support, they are not very abundant (1 or 2 defects per piece) and are found randomly distributed throughout the piece. Figures 3 and 4 show photographs of the defects on the glazed surface.

5.1.2 Quantification of the defect

The quantification of the defect is important for knowing actual incidence in production as well as for estimating the number of stages in the process at which the defect can appear.

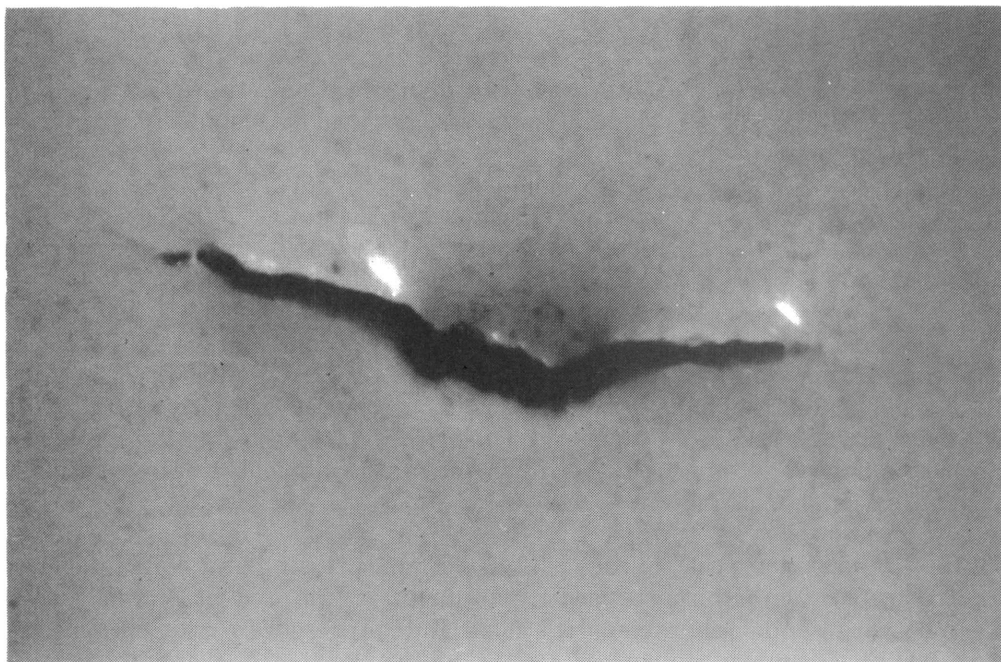


Figure 3. Defect in the surface of the glazed piece; magnified 9.6 times.

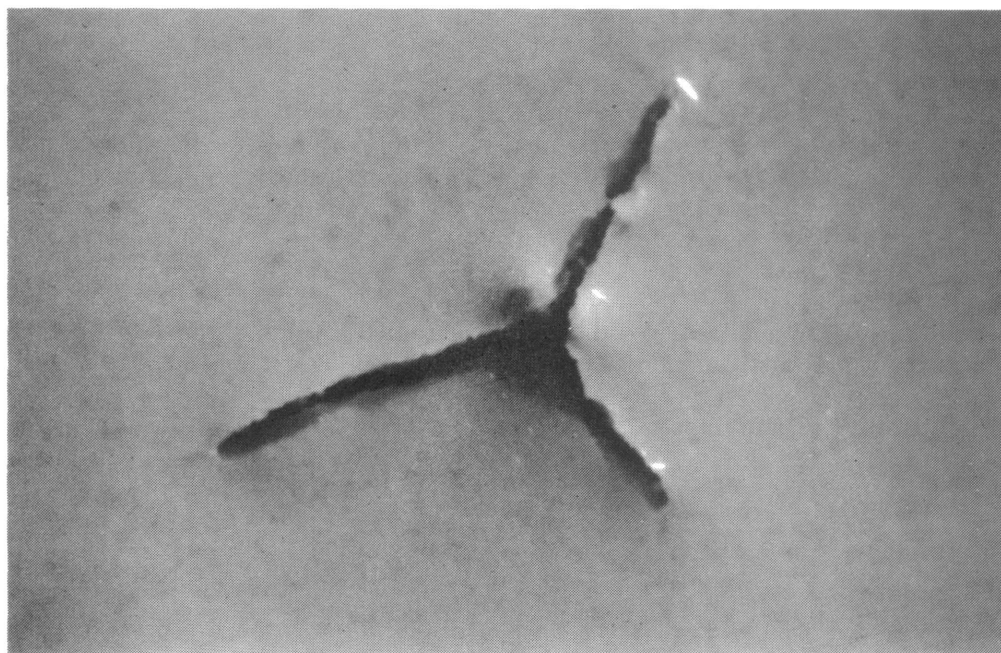


Figure 4. Defect in the surface of the glazed piece; magnified 9.8 times.

This defect has the following classifications:

- Frequency: sporadic
- Amplitude: high
- Intensity: major/grave

5.1.3 First location in plant

With the data gathered from the observation and quantification of the defect, a placement of the origin of the defect within the plant is made. Those operations where it would be difficult for the defect to have originated are not considered.

Drying and firing phases are discounted as causes of the defect. This decision is taken after comparing the aspect of the defect with those of other defects which come from either of these two stages. (See defects file).

Drying cracks are normally bigger in size and are usually situated at the ends of the piece. They are generally due to non- uniformity in the distribution of heat in the piece, which provokes differential dilation-contraction behaviour, this is translated as the escape of tension in the form of a crack. The presence of cracks is on some occasions detected in these stages, although the cracks originate from compressed heterogeneities or from random mechanical stresses (knocks) applied to the piece, which exceed its elastic limit.

After this first selection possible causes of defect are: pressing and glazing operations, and composition and preparation of the paste used in manufacture of the product. Similar reasons to those previously put forward (random character of the defect and the presence of one or two cracks per piece) indicate that the pressing and glazing processes are not the principal causes of the defect, although they may considerably influence its apparition.

Having arrived at this point it is advisable to check all parts of the controls that are used in these operations, in an attempt to find a variation in the working parameters that would indicate which of these might be producing the problem. If some change is found in the operation variables of one of the stages, action should be taken in order to reduce or eliminate the defect.

Where there is no variation in the normal operation parameters a more complete study of the defect is necessary.

5.2 Study of the defect

The object of the study will be, on one hand, to know the exact nature of the defect, its origin and causes, and on the other, to determine the influence of some of the basic production parameters, that can accentuate or reduce its intensity.

5.2.1 Possible Causes

With the information available it can be affirmed that the origin of the defect must be located in the composition of the paste and/or its preparation process.

5.2.1.1 Influence of the paste.

In this section three possible origins of the defect can be distinguished:

- a) Contaminants in the paste
- b) Formulation errors
- c) Errors in the preparation of the paste (this is taken to be the process followed from formulation until the material enters the presses).

a) Contaminants in the paste.

If particles of a contaminative material or granules of a different composition to the rest are found near the surface of the support, these will react differently when fired. Possible degasifications and/or differences in contraction between the contaminative granules and the rest of the piece will generate tensions that can lead to the formation of small cracks.

To prove this hypothesis it is necessary to study the microstructure and composition of the support in the defect zone.

b) Formulation Errors

The apparition of this defect in pieces suggests a random lack of compacting, that could be due to the presence of very hard granules (low deformation) in the surface surroundings.

The granules of considerable hardness can come from a paste that is too plastic (rich in clayey minerals) or from an excessive proportion of additives introduced in the preparation process, that act as binders.

c) Preparation of the paste.

If the paste has a very heterogeneous humidity from the start, i.e. if there are granules that are much drier or damper than the rest, small cracks can arise, especially if there are large sized agglomerations (greater than 750 micra).

The presence of very damp or very dry granules near the support surface produces a zone of different compression to the rest of the piece. The behaviour of this zone during glazing (superficial wetting of the piece) and its subsequent firing can lead to the formation of cracks.

5.2.1.2 Influence of Pressing

The configuration of the piece has an influence on the defect, although it is not one of the causes that directly provoke it.

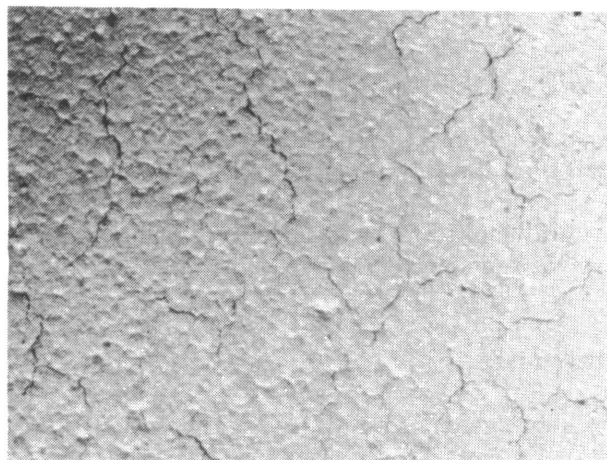
If the whole piece has a low compactness, the effect will be more pronounced, since there will be less cohesion between the granules.

The use of coated moulds for very deformable materials (gums, rubbers, etc...) greatly aids the prevention of cracking and subsequent cohesion of the granules on the surface.

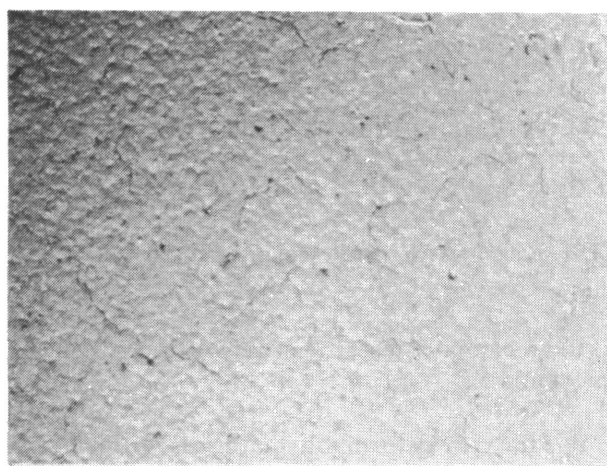
Figure 5 shows the apparition of cracks on the surface of pieces formed at low pressing pressure, when equivalent quantities of water and glaze are applied, and the absence of these cracks, when there is higher compression of the pieces.

5.2.1.3 Influence of glazing

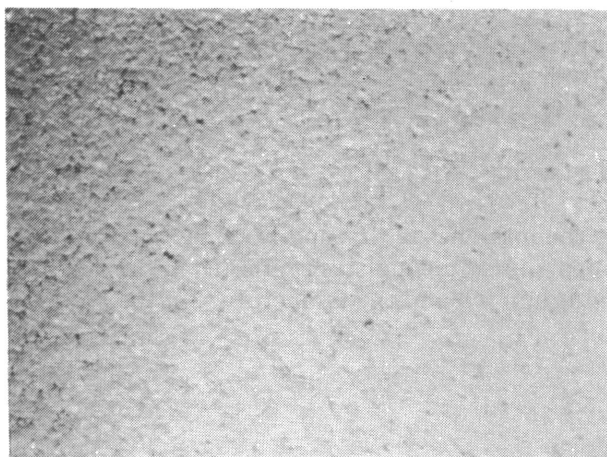
As mentioned earlier the influence of this operation is rooted in the greater or lesser quantity of water applied to the support. Also the moisture retained in the support surface will be a function of the total quantity of water applied and the temperature of the piece.



P= 125 Kg/CM2



P= 175 Kg/cm2



P= 225 Kg/cm2

Figure 5. Effect of pressing pressure on the apparition of cracks on the surface of the piece.

Changes in compactness, mechanical resistance and drying contraction of the glaze layers can also exert an influence on this defect.

Figure 6 shows the appearance of a support surface when the quantity of water applied is increased, the temperature of the piece being kept constant (T_{ambient}).

The effect of temperature of the support is shown in Figure 7. In this group the quantity of water applied and the compression of the pieces were maintained constant. It can be clearly seen how the quantity of the cracks decreases as the application temperature increases.

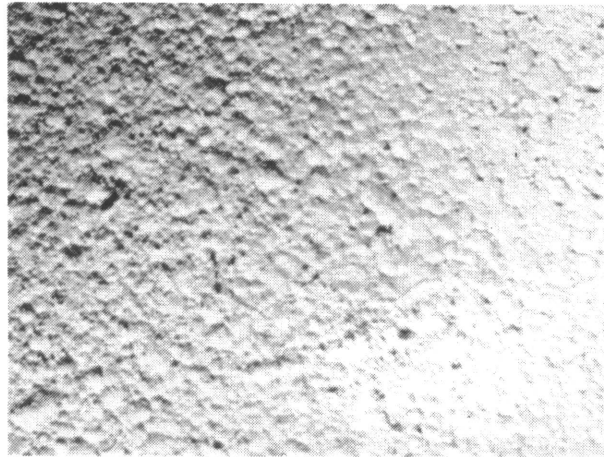
5.2.2. Tests to perform

These tests will help to discount or accept as certain the possible causes of the defect mentioned in the previous section.

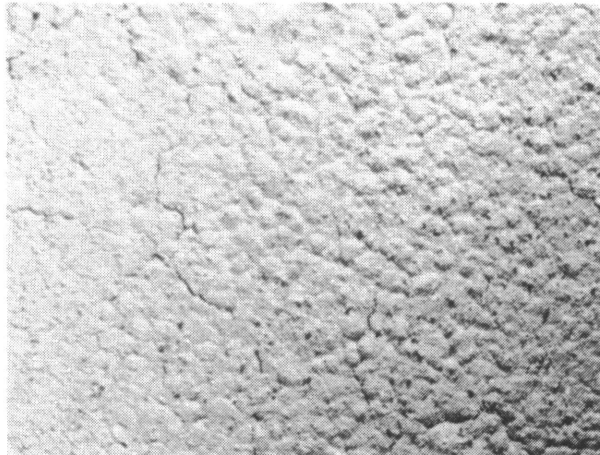
a) Determination of the presence of impurities in the paste. (Differences in composition).

For a detailed study of the composition of the support in the area of the defect an electronic microscope, associated with micro analysis, should be used.

In the case that concerns us, there are no appreciable differences in the composition of the defect zone and the composition of the rest of the piece, the analyses being performed in both areas of a vitreous composition. (Figures 8 and 9).



$m_{\text{water}} = 20 \text{ g}$



$m_{\text{water}} = 40 \text{ g}$



$m_{\text{water}} = 60 \text{ g}$

Figure 6. Effect of the quantity of water in the glaze on the apparition of surface cracks.



T = ambient



T = 110 °C



T = 210 °C

Figure 7. Effect of temperature of the support on the apparition of surface cracks.

02-JAN-90 19:54:45 EDAX READY
 RATE = 4CPS TIME = 80LSEC
 FS = 5747CNT PRST = 200LSEC
 B = zona defecto

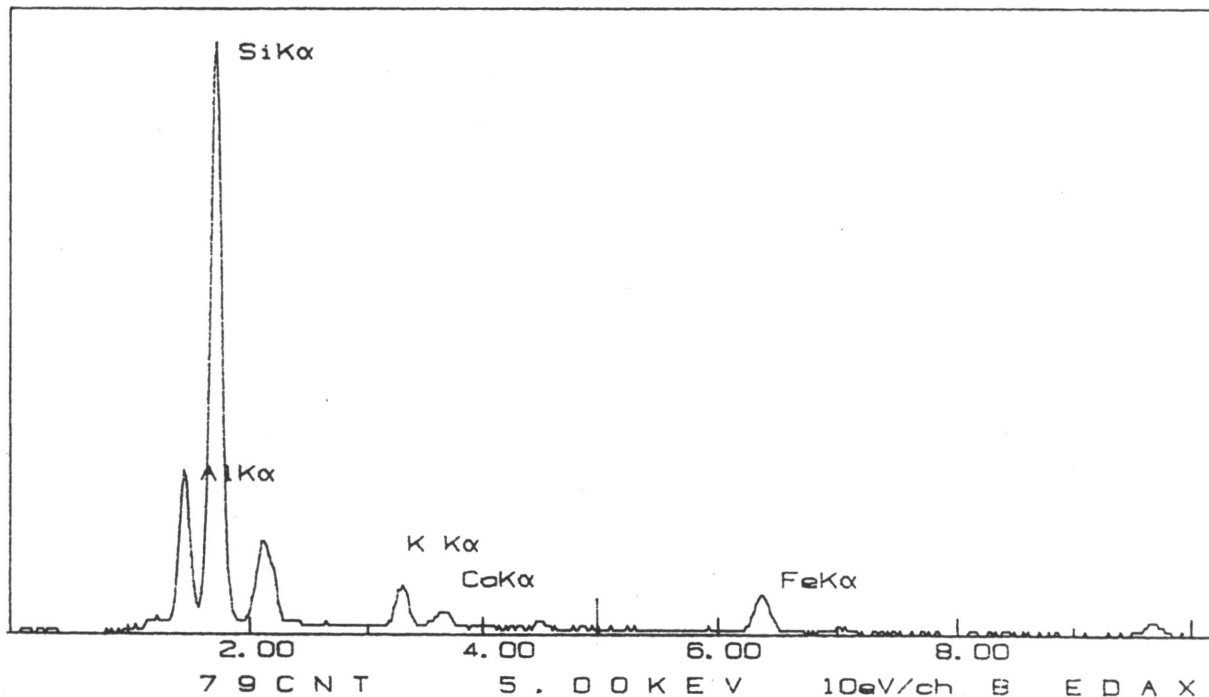


Figure 8. Microanalysis of the support in the defect zone.

02-JAN-90 20:00:46 EDAX READY
 RATE = 7CPS TIME = 82LSEC
 FS = 6145CNT PRST = 200LSEC
 A = zona sin defecto

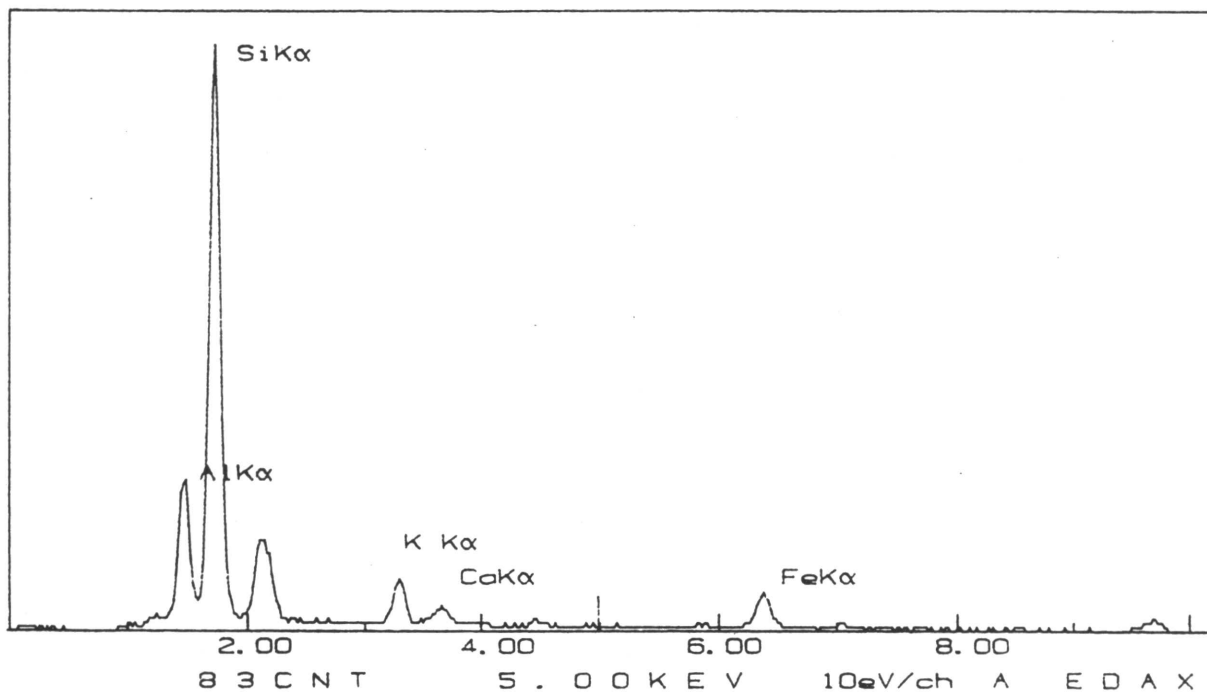


Figure 9. Microanalysis in an area of the support away from the defect.

b) Microstructure of the piece

Examining the defect zone of the sample with the electron microscope, agglomerates of spray can be seen (see Figures 10 and 11), which implies that there are small zones of low compression in the unfired piece.

c) Tests on the formulation of the paste

As mentioned previously, an excessive amount of plastic materials and/or binders would affect all the granules, altering their hardness. This would provoke, in principle, a change in the overall properties of the piece and should not be the cause of a random defect. To confirm this supposition, tests were performed on the dry mechanical resistance of pressed pieces of a standard composition, and in those pieces in which the defect was present.

Since no difference was found between the values obtained and, in the examination made using the electron microscope, the granules found away from the defect zone were perfectly deformed, the possibility that the aforementioned errors might have been produced in the formulation of the paste was discounted.

d) Experiments on the preparation of the paste.

It can be supposed that the defect most probably originates in larger sized granules. Hence, the humidity of the fraction of the paste larger than 500 micras was calculated and compared with the humidity of the rest of the spray.

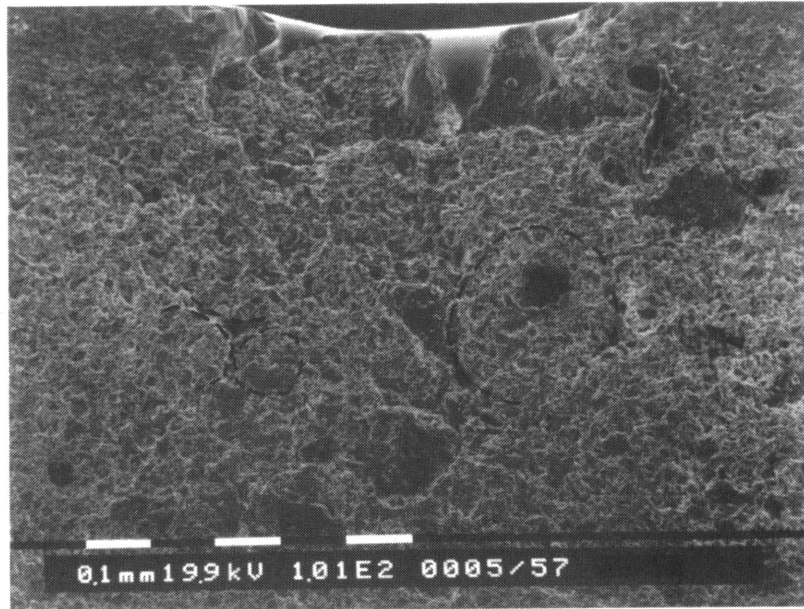


Figure 10. Defect Zone; magnified 101 times.

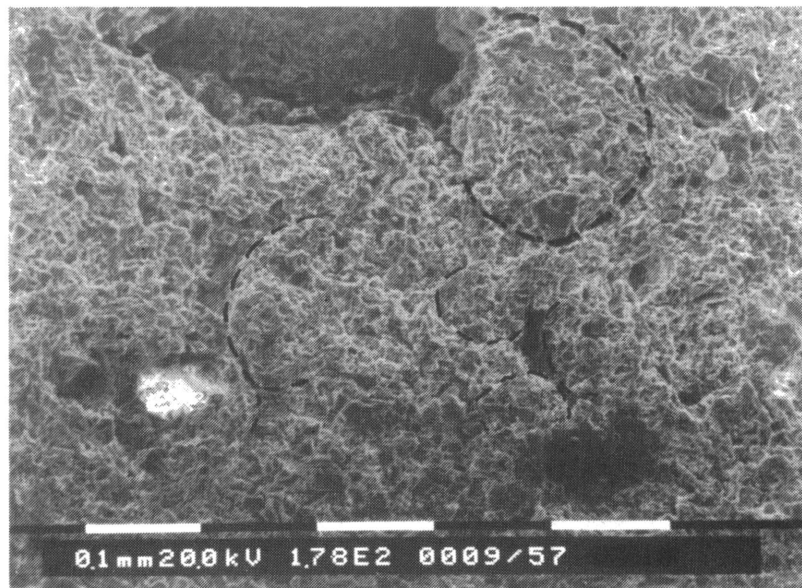


Figure 11. Defect Zone; magnified 178 times.

The granules rejected at 500 micras had a humidity considerably lower than the rest, which makes them a very probable cause of the defect. As has been studied in numerous other works, the humidity content is directly related to the compactness in both the fired and unfired piece. The relationship between the humidity of the spray (XP) and the apparent density when dry is shown in Figure 12. In this figure the displacement that the point of confluence of two sprays undergoes can be seen. (The spray with the lesser humidity content moves this point at higher pressures). The relationship between these variables is reflected in the microstructure of the pieces. Figures 13 and 14 show microphotographs of the two pieces formed at the same pressure (300 kg/cm²) using two sprays with different humidities (0.018 and 0.082 kg water/kg of dry solid respectively).

As can be seen, in the piece pressed with low humidity (Figure 13) the granules of the spray maintain their identity perfectly.

5.3 Determination of the cause of defect

In the tests performed the following results were obtained:

- There is no difference in composition between the defect zone and the rest of the support.
- There is only an appreciable microstructural change in the defective zone. (Undeformed conglomerates)

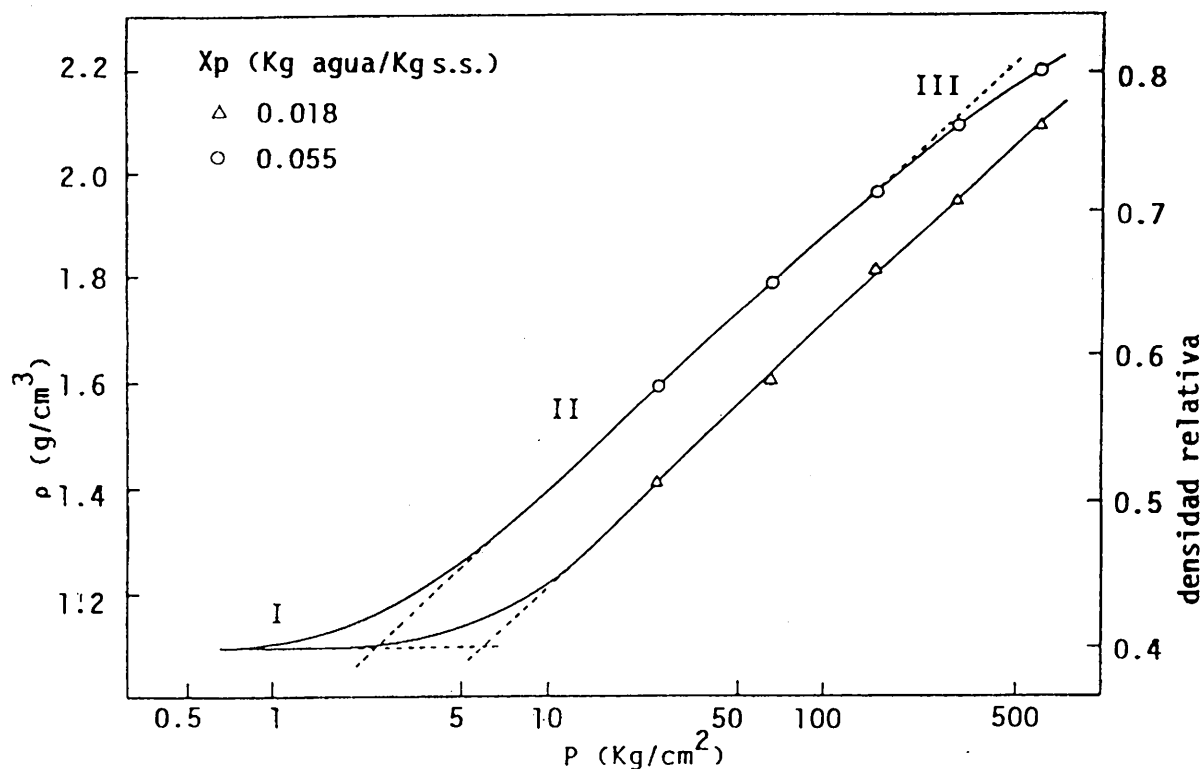


Figure 12. Spray compression diagram for two different humidities.



Figure 13. Microstructure of the pressed piece a $X_P=0.018$ kg water/kg d.s. and $P=300$ kg/cm²; magnified 101 times.

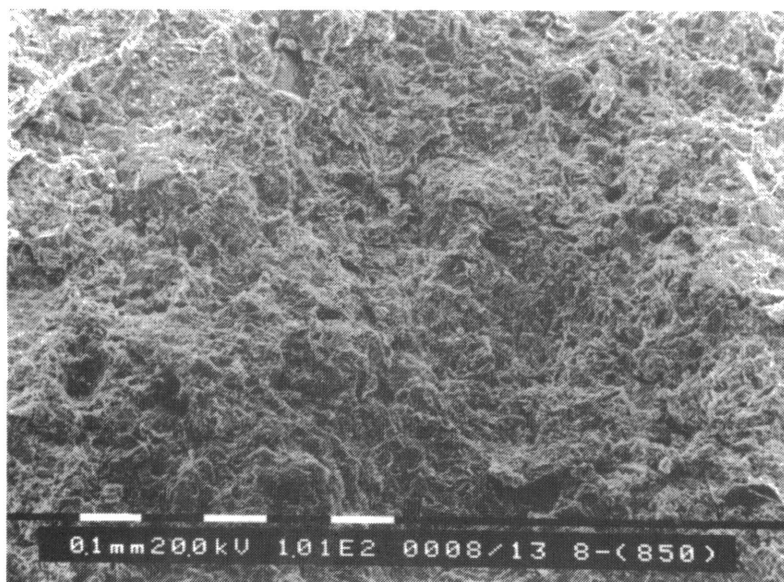


Figure 14. Microstructure of the pressed piece at $X_p=0.082$ water/kg d.s. and $P=300$ kg/cm²; magnified 101 times.

- c) There are no alterations in the mechanical resistance of the pieces.
- d) The thicker part of the spray is less humid than the rest.

Bearing in mind sections a) and c), contaminants in the paste or errors in the formulation can be discounted as the origin of the defect.

From b) and d) we can infer that the defect is probably due to large sized dry granules. These agglomerates can come from deposits produced in the atomizer itself, in the transport mechanisms used (belts, tankards,...) or in the repositories .

In the atomizer, agglomerates are usually produced by deposits of materials stuck to the wall of the atomizer, that occasionally fall into the spray.

Condensations produced whilst in transport or in repositories, usually originate in water vapour released by the atomizer, due to its humidity content and high exit temperature. This vapour condenses in the colder zones along its path, and produces an agglomerating effect on atmospheric dust and previously deposited vapour. It subsequently dries and, at a given moment, falls into the material that is being produced.

5. SOLUTIONS TO THE DEFECT

Various measures can be adopted in order to eliminate the defect or to at least reduce its effect on the quality of the finished product.

The most immediate solution is to sieve the material before feeding it in to the presses, discarding any larger sized granules or agglomerates that may have formed.

The exit temperature of the spray in the spray plant could be regulated and/or a refrigerating system that reduces evaporation adopted.

It would be advisable, on a general level, to avoid the use of low humidity spray, not even mixed with another spray of correct humidity, since the first can potentially be a nucleus of generation of the problem studied.

Finally, a careful and periodic cleaning of the equipment (silos, belts, sieves,...) would reduce the quantity of dry agglomerates in the spray.

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