ASIAN JOURNAL OF CIVIL ENGINEERING (BHRC) VOL. 16, NO. 2 (2015) PAGES 235-248



STOCHASTIC MODELS FOR ARTIFICIAL CEMENT CONTROL PARAMETERS

L. Nouri^{1*}, S. Yousfi¹, M. Saidani², H. Hadjab³ and M. Nouri⁴

¹University of Boumerdes, Faculty of Engineering, Algeria

²Coventry University, Faculty of Engineering and Computing, Priory Street, Coventry CV1

5FB, UK

³University of Science and Technology Houari Boumediene, Faculty of Civil Engineering, BP 32 Bab Ezzouar, El Alia, Algiers, Algeria

⁴University of Liège (ULg), Faculty of Sciences, Belgium

Received: 14 June 2014 Accepted: 20 October 2014

ABSTRACT

The artificial cement characterization requires the measurement of a large number of control parameters. In the present work, twenty four parameters that are monitored on a daily basis during three years of observation are investigated. Due to the large amount of data to be collected, a monthly overage is calculated. This allows the result fluctuations due to managing difficulties to be reduced.

The purpose of this study is to show that it is possible, with an acceptable approximation rate, to make a variable reduction, which is to reduce the number of control parameters, as these measurements are identified every hour at the exit of the clinker crusher and daily at the expedition.

The aim of the present investigations is to suggest a manufacturing supervision that can give improvements in the characteristics of the finished product and that can reduce, in an objective way, the number of tests. The consequences of such pertinent supervision are a great deal of saving time and lower cost.

Keywords: Manufacturing supervision; artificial cement; control parameter correlation; factorial analysis methods; variable reduction; stochastic models; crusher; and expedition.

1. INTRODUCTION

Artificial cement quality is characterized by a number of measurements made on its chemical and mineralogical composition. These measurements are completed by tests which provide guidance on the overall cement behavior as an essential material in the mortars and

^{*}E-mail address of the corresponding author: cbx086@coventry.ac.uk (L. Nouri)

concretes composition [1, 2, 3]. Various studies have demonstrated the role of each of the cement components in the final binder behavior and the interaction between some of these components as well [4, 5].

In the present work, the different correlations that link most of cement components is investigated. This investigation is done statistically; this has quickly led to a large number of potential relationships between these components. An exhaustive study appeared immediately non-rational which would not lead obviously to an exploitable outcome. Consequently, it seemed to us more judicious to consider only the most relevant correlations and to study them in a rigorous way.

Descriptive statistical methods are not suitable for such study, at least at its first stage which led us naturally to use comparative methods after a justified choice [6]. These methods are used to facilitate the correlative study between artificial Portland cement components, considered as control parameters, which are distinguished by the similarity of their statistical behavior.

The adopted working plan includes the following steps:

- measurements and tests done on ordinary artificial Portland cement components and their standardized representations;
- chemical components as mean constituents analysis and tests analysis;
- creation of the correlations between the selected control parameters in the comparative analysis.

It is worth noting that a behavior comparative study of the mineralogical components is in the present case unnecessary as analytical links exist yet between the different chemical compounds [3]. This fact does not mean that this study is useless but it is only out of the scope of this work.

The present study focuses on observed fluctuations of the artificial Portland cement composition during its dry process manufacturing at the exit of the grinder. It has focused on the cement expedition as well, this after seventy-two hours storage. This duration is fixed by management requirements. By fluctuations, we mean low amplitude variation in the order of 10%, non-controllable at the current state. These fluctuations are due to the ore composition and to the manufacturing method.

The search of correlation between 24 standard control parameters would require 264 crosses and their corresponding analysis. So, it seemed wiser to us to use the factorial analysis methods in order to reduce the crosses to the most relevant links and to retrieve simple metric relationships between the control parameters retained in this analysis, currently named stochastic models.

2. MEASUREMENTS AND ANALYSIS OF PHYSICAL AND CHEMICAL ARTIFICIAL CEMENT CHARACTERISTICS

2.1 Measurements of the physical and chemical of artificial cement characteristics

The three main measurements types used for the cement control parameters while manufacturing are:

- Measurements on the chemical composition of the finished product which variations determine the cement quality. This composition depends essentially on the ore quality and on correctional additions;
- Measurements on mineralogical composition which provide guidance on the treatment of ore in the manufacturing process;
- Physical measurements which are summed up as standardized tests. These measurements are decisive for the verification of the expected cement quality [3].

It is obvious that all these measurements are interdependent and that their dissociation is only a convenient way to ensure as close as possible the obtaining of the expected product.

In the present research, daily observations have been made in a cement factory during two whole years on current EMF II L 32. 5 class artificial Portland cement. To avoid too large dispersion due to the daily production risks, a choice had to be made on the way of gathering the results. So, the monthly arithmetic average results have been chosen arbitrarily. For the sake of better management these latter were also used as a practical choice by the plant operators.

Two different sets of measurements were undertaken on the same control parameters:

- Measurements in the final stage of manufacturing, at the exit of the clinker crusher;
- Measurements at the shipment stage of the product to the last user after a period of time of approximately seventy-two hours.

These differentiating measurements make sense as stabilization operates after cooling. This fact will be confirmed later on in the present research. In order to present clearly the control parameters, a standardization of the obtained results has been adopted. This action allowed gathering results that size is totally incompatible in the same graphics. The reading is then made very easy since it is sufficient to read the value of the magnitude seen on the graph and to multiply this value by the corresponding maximum value given at the bottom of the graph.

It has been found useful to split the representation into four different graphs that correspond to:

- chemical action on the mean compounds also called major compounds;
- chemical action on secondary compounds called minor or alkali;
- secondary physical action intended to provide guidance on the cement behavior as an isolated product;
 - compressive and bending strength measurements as defined in standard way.

As an example, one of these graphs drawn for some chemical compounds cement at crusher exit is presented (Fig. 1). The same type of graph is drawn up for all the twenty-four measured control parameters.

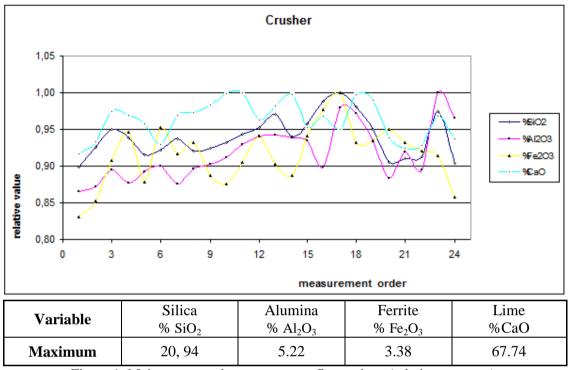


Figure 1. Main compounds measurement fluctuations (relative amounts).

2.2 Measurement comparative analysis

2.2.1 Mean components analysis

Factorial analysis is a data reduction method which retains the most relevant variables and omits redundant ones by searching correlations between all the variables. This method aims bringing together, in one or several graphs, most of the information contained in an array by focusing not on absolute values, but on the mappings between variables expressed in relative values. In this reduction method, an analogy between statistics and mechanics is used i.e. the frequency is the mass, the variance is the moment of inertia and the average is the centre of gravity. All mechanical mathematical results are then usable in statistics.

Different statistical methods have been developed on this basis. One of the more elaborate is the factorial correspondence analysis or F.C.A. This method enables an advanced calculation on variables but imposes a variables separation into ordered categories.

In our case, the variables are metric and non-ordinal. So, the most appropriate comparative method to be used is the main components analysis: the M.C.A. This choice is justified by two main reasons:

- In this analysis absolute correspondences between variables are not sought but are used as means of reducing interdependent variables research. This is done in order to focus on variables which correlation in terms of variance is the most significant;
- Other analyses impose measurement decomposition in arbitrary intervals, called categories, by using inter-categories frequencies. This method of decomposition hides fine

changes which represent the object of our study and for this reason these methods will not be used.

The knowledge of the nature of the different parameters measured on artificial Portland cement makes the use of the different methods of extracting factors proposed by J.P. Benzekri in M.C.A. [7] unnecessary. These methods are based on purely statistical analysis of the variables behavior. The present investigation seeks the variables variation in terms of their physical nature [8].

The number of factors has been limited to three; this explains the 75% of the variance rate used. In fact, the first factorial plane explains at its own 62% of the variance; a second plane has been retained only to check the absence of parallax effect. Such a representation for the first and second plan (Figs. 2 and 3) is always given as an example. Various connections between the parameters correspond to similar measurements, this allows a synthetic vision of the parameters distribution against their variance.

It must be noted that the correlations analysis of mineralogical components is unnecessary in the present investigation as proven relationships between these components and the chemical components of cement have been used in the analysis [9]. One of the methods used is based on the lime (C_aO) saturation coefficient K_S given by the following expression according to Bogue and Bogue modified methods:

$$Ks = \frac{CaO - 1.65Al_2O_3 + 0.35Fe_2O_3 + 0.7SO_3}{2.8SiO_2} \tag{1}$$

Where the formula coefficients represent parts of the lime reacting with different chemical cement compounds, their values are the results of chemical reactions giving the mineralogical compounds formation C₃A, CF and C₃S according to the following form:

$$3CaO + Al_2O_3 \rightarrow 3CaO.Al_2O_3 \text{ or } C_3A$$

The CaO and Al₂O₃ molar masses are respectively 56g and 102g. 3CaOs represent 168g which react with 102g of Al₂O₃ In mass proportions, the amount of CaO is:

CaO amount = $168/102 \text{ Al}_2\text{O}_3 = 1.65 \text{ Al}_2\text{O}_3$ amount

The same process is used for CF and C₃S by using the following reactions:

 $CaO + Fe_2O_3 \rightarrow CaO.Fe_2O_3 \text{ or } CF$

 $CaO + SO_3 \rightarrow CaSO_4$

 $3CaO + SiO_2 \rightarrow 3CaO.SiO_2 \text{ or } C_3S$

Ks numerator is the part of lime that reacts with SiO_2 giving the cement main components: C_3S and C_2S . The Ks denominator represents the amount of lime which would be used to form the C_3S alone.

By noting S1 and S2 the SiO₂ mass proportion in C₂S and in C₃S respectively and by noticing that the reactions are not total, the saturation coefficient becomes:

$$Ks = \frac{2S1 + 3S2}{3S2} \tag{2}$$

By putting S = S1 + S2 the following relations are obtained:

$$S1 = 3 S (1 - Ks)$$
 (3)

$$S2 = S (3 Ks - 2)$$
 (4)

This is only one approach of the method known since a long time. One of the method development authors is R.H. BOGUE [9] who, by using abbreviations such as:

$$CaO = C$$
; $SiO_2 = S$; $Al_2O_3 = A$ and $Fe_2O_3 = F$,

Elaborated the following equations:

$$C_3S = 4.07 \text{ C} - 7.6 \text{ S} - 6.72 \text{ A} - 1.43 \text{ F} - 2.85 (SO_3)$$
 (5)

$$C_2S = 2.87 \text{ C} - 0.754 (C_3S)$$
 (6)

$$C_3A = 2.65 A - 1.69 F$$
 (7)

$$C_4AF = 3.04 F.$$
 (8)

The composition determined in this way, underestimates the amount of C_3S and in the same time overestimates the amount of C_2S because other oxides replace lime in C_3S . The formulas (1) to (8) are shown only as indication and are not entirely developed.

By taking into account all the control parameters chosen according to the previous explanations, our present work obtained graphs similar to those represented. Only two of them have been retained. They contain 75% of the cement crusher exit information (Figs. 2 and 3). Two similar graphs have been drawn up for cement expedition but they have not been represented in order to avoid the graph burdening.

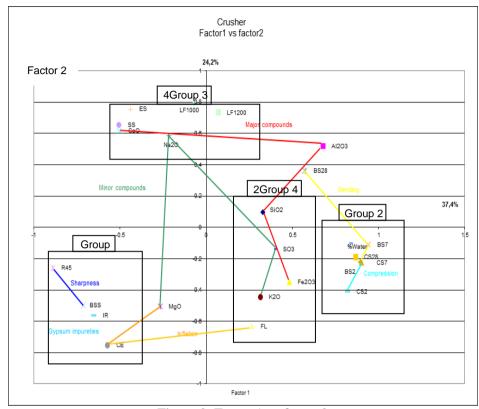


Figure 2. Factor 1 vs factor 2

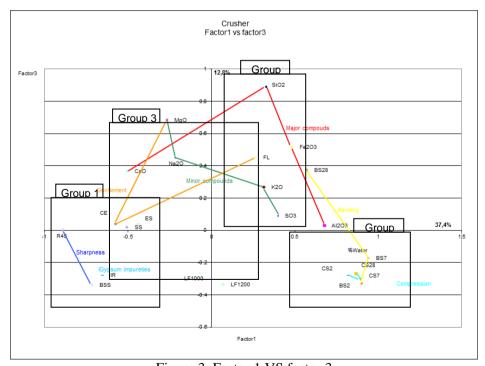


Figure 3. Factor 1 VS factor 3

The different names on the charts are the short forms of the various measurements

2.2.2 Results interpretations

Our aim is not seeking physical interpretation of the interactions between the different control variables, but rather researching the mathematical form of these interactions owing to use them in predictive purposes.

2.2.2.1 Measurements at the exit of the Crusher

Graphical results of the analysis are noteworthy because they reveal several expected groupings, which is a confirmation of the method validity for the treated problem. Four groups of control parameters appear clearly in the selected factorial planes.

```
• 1<sup>st</sup> group:
Insoluble residues (IR);
Blaine specific surface (BSS);
Residues under 45 \squarem (R45);
Le Chatelier expansion (CE).
• 2<sup>nd</sup> group:
proportion of residual water in cement;
2 days compressive strength (CS2);
7 days compressive strength (CS7);
28 days compressive strength (CS28);
2 days bending strength (BS2);
7 days bending strength (BS7);
• 3rd group:
CaO:
Na<sub>2</sub>O:
Loss to fire at 1000 °C (LF1000);
Loss to fire at 1200 °C (LF1200);
Start setting (SS);
End setting (ES).
• 4th group:
SiO_2(S);
Fe_2O_3(F);
K_2O(K);
SO_3(SO);
Free CaO (FL).
```

These groups are not chosen in a subjective manner but they stem from consideration of different projection planes to avoid any perspective or parallax effect. It is therefore appropriate to seek a metric relationship among the variables in each group by, for instance, a polynomial regression. Linear, logarithmic and exponential regressions fit into this framework which can be a merely approximation and this is the case in the present study. A more accurate study could be undertaken later.

The first and second group oppose to the second and third factorial axes. It would be interesting to check if this does not correspond to a particular relationship between these two

groups. Also, it is the case for K₂O and SO₃ on one hand, and MgO and Na₂O on the other, often viewed as having similar behaviours in the cement.

The major components are not of interest for two essential reasons. The first being the fact that these components are sufficiently studied by many authors; the second is derived from the analysis itself which showed no direct relationship for small fluctuations. This result is interesting in the sense where, in practice, it is difficult to maintain a composition perfectly constant, this result reassures us on quality and the constancy of the mechanical characteristics.

2.2.2.2 Measurements at the cement expedition

After stabilization, cement behavior differs significantly from the one at the exit of the crusher:

- CaO joins the group of main compounds (SiO₂; Al₂O₃ and Fe₂O₃);
- bending strength at 28 days is found in the vicinity of other strength measurements;
- free lime which seems to be closer to K₂O and SO₃ in 1-2 factorial plane, get away

from these two variables in the 1-3 plane and then get near to the main compounds. Indeed, the free lime is a part of this latter group. These results have been checked in the other factorials planes but are not represented in the present paper.

In order to select the most relevant relationships, the clusters represented on the factors graphs are treated, in a first approximation, by linear regressions. As an example, one graph of this type is represented (Fig. 4).

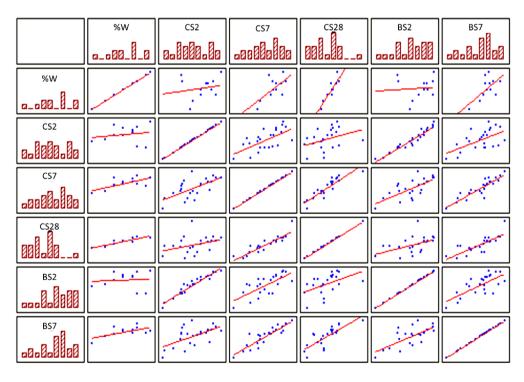


Figure 4. Linear regressions between some measurement parameters of group 2

3. CORRELATION ANALYSIS FOR THE RETAINED CONTROL PARAMETERS

3.1 Regression between retained variables using factorial analysis

It will be interesting to validate the factorial analysis results, in terms of mean compounds, by crossed polynomial regression, two by two, between the selected variables. These crosses will enable to encrypt relationships between cement parameters with significant correlation rate in the one hand, and in the other hand, to eliminate relationships which correlation rate does not confirm, in an obvious manner, the binding in the statistical sense.

However, it should be noted that some low correlation rates are retained because the cloud of points shape always presupposes a relationship when some irregular points are eliminated or when a correlation exists in a series of measurements but does not in equivalent series. These situations are applicable at the crusher exit or at the expedition.

The regression residues calculations are carried out in parallel to valid the regression choice. As it is explained earlier, only polynomials including, in an approximate way, logarithmic and exponential regressions are used. The linear regression (Fig. 5), which is a particular case, is retained only when the polynomial regression does not improve the correlation rate. Also, only the second order polynomials are used as higher degrees do not increase significantly the correlation rate. In the present paper, some chosen cases among a large number of treated crosses are presented.

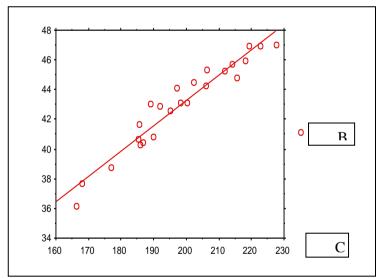


Figure 5. Regression between 2 days compressive strength (CS2); and 2 days bending strength (BS2)

Tables 1 to 4 summarize the obtained results between the various parameters measurements in an equation form. In order to judge the validity of each equation a correlation rate \mathbf{r} is adopted.

The studied control parameters (see the first and second columns of the Tables 1 to 4) are written in a short form but are easily readable as they correspond to common names used in the cement control. For instance, BS28 is the short form of the bending strength at 28 days.

The used units are conventional except for the strengths which are expressed in kilogram-force per square centimeter.

Table 1: Crusher Group1

		<u> </u>		
y	X	y = f(x)	r²	r
BS28	CS28	y = 0.183 x - 2.858	0,716	0,846
CS28	CS7	y = 0.939 x + 114.3	0,818	0,904
BS2	CS2	y = 0.171 x + 9.051	0,930	0,964
BS7	CS7	y = 0.172 x + 5.116	0,804	0,897
BS28	BS7	y = 0.993 x + 13.396	0,699	0,836
CS28	%Water	y = 17.761x + 15.934	0,820	0,906
BS28	%K2O	$y = 566.26 - 1242.13 x + 776.608 x^2$	0,510	0,714
SS	ES	y = 0.727 x - 6.797	0,912	0,955
SS	LF1200 ° C	$y = 1123.347 - 262.224 x + 17.322 x^2$	0,229	0,479
LF1200 ° C	LF1000 ° C	y = 0.718 x + 2.351	0,677	0,823
%SO3	%K2O	$y = 4.783 + 1.076 x - 4.518 x^2$	0,135	0,367

Table 2: Expedition Group1

		ı i		
y	X	$\mathbf{y} = \mathbf{f}(\mathbf{x})$	r²	r
BS28	CS28	y = 0.199 x - 10.039	0,696	0,834
CS28	CS7	y = 0.984 x + 99.669	0,783	0,885
BS2	CS2	y = 0.184 x + 6.41	0,925	0,962
BS7	CS7	y = 0.179 x + 2.79	0,664	0,815
BS28	BS7	y = 1.048 x + 9.728	0,750	0,866
CS28	%Water	y = 19,699 x - 29,702	0,340	0,583
BS28	%K2O	$y = -314.471 + 1190.802 x - 899.692 x^2$	0,630	0,794
ES	SS	y = 1.185 x + 35.712	0,952	0,976
SS	LF1200 ° C	$y = 2040.59 - 495.384 x + 31.991 x^2$	0,619	0,787
LF1200 ° C	LF1000 ° C	y = 0.939 x + 0.616	0,936	0,967
%SO3	%K2O	$y = -70.805 + 204.679 x - 143.32 x^2$	0,745	0,863

Table 3: Crusher Group2

y	X	$\mathbf{y} = \mathbf{f}(\mathbf{x})$	r²	r
CS7	IR	y = -33744 x + 373.281	0,469	0,685
CS2	IR	y = -34.39 x + 242.789	0,391	0,625
BSS	IR	y = 118.752x + 3357.787	0,048	0,219
Na2O	K2O	y = -1.271 x + 1.043	0,335	0,579
CE	R45	y = 0.045 x + 692	0,042	0,205
BS28	%Water	y = 1.483x + 41.83	0,302	0,550
Fe2O3	SiO2	y = 128 x + 58	0,318	0,564
CS28	K2O	y = -409.136 x + 719.24	0,356	0,597
BS2	IR	y = -6.15 x + 50.932	0,397	0,630
BSS	CE	y = 290.217 x + 2959.833	0,308	0,555

y	X	$\mathbf{y} = \mathbf{f}(\mathbf{x})$	r²	r
CS7	IR	y = -24.609 x + 369.579	0,386	0,621
CS2	IR	$y = 194.411 + 44.503 x - 25.229 x^2$	0,398	0,631
BSS	IR	y = 101.302x + 3569.584	0,067	0,259
Na2O	K2O	y = -0.96 x + 833	0,171	0,414
CE	R45	y = 0.104 x - 1.181	0,372	0,610
BS28	%Water	y = 0.689 x + 60.733	0,024	0,155
Fe2O3	SiO2	y = 0.057 x + 1.956	0,066	0,257
CS28	K2O	y = -348.874 x + 683.731	0,296	0,544
BS2	IR	y = -6.394 x + 52.927	0,425	0,652
BSS	CE	v = 434.977x + 3098.162	0.539	0.734

Table 4: Expedition Group2

3.2 Results Interpretation

The results are split into two groups depending on the values of the correlation rate. The first group contains the parameters which the correlation rate value is between 79% and 98%. It is reminded that some relationships have relatively low correlation rate and despite of this, they are selected. This is due to the rate rise when considering the series of equivalent measurements as they are observed at the crusher exit or at the shipping (expedition). This is the case of relationships linking:

- The bending strength at 28 days to K₂O amount;
- The early setting to the loss of fire resistance at 1200 °C;
- The Na₂O, SO₃ and K₂O content.

It appears, according to obtained results, that the measurement of compression strength at 28 days is a good predictor of bending strength at 28 days, with a consequent correlation rate. The linking relationship is:

```
y = 0.183 x - 2.858 with r = 85\%,
```

where y is the bending strength at 28 days.

and x is the compression strength at 28 days.

The relationships reached in the present research are presented in Tables 1 to 4. In the present work, a very interesting obtained relationship is that connecting the setting start to the setting end parameters at the expedition. Its corresponding equation is as follows:

```
y = 1.185 x + 35.712 \text{ with } r = 98 \%
```

where y is setting start in minutes

and x is the setting end in minutes

This result is compared with a formula proposed by Blombed in CERILH quoted by Vénuat [10] which expression is:

$$y = 1.5 x + 0.85$$
 $r = 97 \%$

where y is setting start in hours

and x is the setting end in hours

or expressed in minutes as follows:

$$y = 1.5 x + 51$$

The difference lies in the fact that the considered cement is near the 42.5 class but it is listed in the 32.5 class [3].

The second group is not part of the present study but shows the perspectives opened by this analysis.

In the present work, it is proven that the 2 days compressive strength gives no indication on the strength at 28 days. The other relationships between strength measurements can be symbolized in the following manner:



Or in more simplified way it can be said that the compressive strength at 7 days allows the prediction of the compressive strength at 28 days and the bending strength at 7 and 28 days according to the obtained equations which are presented in Tables 1 to 4.

4. CONCLUSION AND FUTHER RESEARCH

It can be concluded from the reached results that the study of the present issue in comparative statistics form is proved to be a very powerful tool which allowed a genuine analysis of the different control parameters measured on the cement composition and on the cement final behavior. This study avoided, using the well known basic methods with the tedious corresponding interpretation, the observation crossings on 24 variables which would have required 264 analyses for each the two series: grinder exit and expedition.

Thus, irrelevant study has been straightaway eliminated in order to concentrate on the bindings, in the statistical sense, capable of giving workable metric relations. A standard graphical representation of the obtained results has provided an overview of the relative variations of different measured parameters. The principal components analysis, chosen for its appropriateness for the type of the measured variables, gave a synthetic vision of the parameters behavior.

In this study, it has been given up using the correspondence factorial analysis benefits, such as the distributional equivalence, in order to avoid choosing arbitrarily categories within the variables called characters. This renouncement did not constitute a disability in the development of the present work, as the variables were treated by using numerous analytical studies and as their physical behavior was known in advance. Then, it remained only to find the simplest algebraic relations defining the variables relative variations. This goal was achieved for the studied cement, in this case the artificial ordinary cement, produced in the considered factory. It should be noted that this cement is near the real 42.5 class but it is sold under the 32.5 class label in order to meet European standard requirements [3].

Further research topics steaming from the present investigation are quite various and can be listed as follows:

- As the proportion of residual water in the cement powder seems to affect positively the compressive strength at 28 days, it would be interesting to study the limiting values of these variables;
 - K₂O influence negatively on resistance to bending at 28 days but not from an obvious

manner on resistance to compression, this only to the phase expedition cement and positively to the output of the crusher;

• The variation of the SO₃ proportion against the K₂O proportion is positive at the crusher exit and it is negative at the expedition. Also, at the expedition the nearly 86% correlation rate indicates a proven link. Consequently, it would be interesting to check if these facts are due to a behavior resulting from the treatment of the cement manufacturing process or are only due to the initially existing SO₃ and K₂O proportions in the ore [11].

The second group tables require for themselves further studies in order to confirm or to refute the established relationships.

This study concerns as stated previously, a single cement factory observed over a period of three consecutive years and it is obvious that the determined parameters can be applied only to this factory.

In the present time, we develop a wider study including twelve cement factories. This work will allow adjusting the correlation coefficients based on local settings and eventually finding a relationship that would lead to a quick calibration for each factory.

REFERENCES

- 1. F. GORISSE. Essais et contrôle des bétons, Eyrolles, Paris, (1978) 25-32.
- 2. G. DREUX. Nouveau guide du béton, Eyrolles, Paris, (1981) 16-33.
- 3. Standard E 197-1. CEN, May 2000.
- 4. CIMbéton. Les constituants des bétons et des mortiers, Tome1. Volume CT-G10, Paris, (2005) 10-29.
- 5. Mounanga P. Étude expérimentale du comportement de pâtes de ciment au très jeune âge : hydratation, retraits, propriétés thermophysiques, Doctorat thesis, Nantes, 2003.
- 6. Lagarde J. *Initiation à l'analyse des données*, Dunod 1er tr., Paris, 1983.
- 7. Benzecri JP, et al. *Pratique de l'analyse des données*, Tome 1, 2 et 3, Dunod, Paris, 1980-1982.
- 8. Neville AM. *Properties of concrete*, Pitman, London, (1981) 1-117.
- 9. Bogue RH. Chemistry of Portland Cement, Reinhold, 1955.
- 10. Venuat M. Practice of concretes and cements, Monitor, France, 1976.
- 11. Davidovits J. *Procédé supprimant la réaction agrégat-alcalin dans les bétons, et ciment obtenu par ce procédé*, Demande de brevet international N WO91/11405 W.I.P.O.