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# *A Mathematical Model for Predicting Autoclave Expansion for Iraqi Portland Cements*

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## Abstract:

***The autoclave expansion test (ASTM C151- 05) is one of the internationally used tests in detecting the unsoundness of Portland cements. The factors affecting test results were reviewed. A statistical analysis of 130 autoclave test results was done. Each result represents the average of a month production, for four Iraqi cement factories. Based on that, a mathematical model was built to predict the autoclave expansion for Iraqi Portland cements. The coefficient of determination, $R^2$, was 0.97. The model was tested with new raw data and the predictions were highly correlated to the experimental results (r = 0.92). Moreover, a t-test was carried out and it showed that there is no difference between means of experimental and predicted sets of values. The good predictions give the chance to make use of the model in saving time and money.***

***Keywords: Autoclave expansion, Fineness, Free CaO, Mathematical modeling, MgO, Portland cement, Statistical analysis, Sulphate.***

## نموذج رياضي للتنبؤ بتمدد الفحص المحمم للاسمنت البورتلاندي العراقي

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## الخلاصة :

***يعتبر فحص التمدد المحمم من الفحوص المستخدمة عالمياً وبكثرة للتحري عن عدم الثبات للسمنت البورتلاندي. في هذا البحث تم استعراض العوامل التي تؤثر في نتائج هذا الفحص واجري التحليل الإحصائي على 130 نتيجة فحص وكانت كل نتيجة فحص تمثل المعدل الحسابي لإنتاج شهر كامل لأربعة معامل للاسمنت العراقي هي كبيسة وكركوك والقائم والفلوجة. واستناداً الى ذلك فقد تم بناء نموذج رياضي للتنبؤ بالتمدد للفحص المحمم للاسمنت البورتلاندي العراقي. كانت قيمة معامل التحديد لهذا النموذج تساوي (0.97) واختبر النموذج بتطبيقه على بيانات جديدة (لم تدخل في بنائه) وكانت تنبؤاته عالية الارتباط بالقيم المختبرية المستحصلة من الفحص ( معامل الارتباط = 0.92). علاوة على ذلك ، تم إجراء اختبار - تي على النموذج الرياضي واظهر الاختبار بان لايوجد فرق بين المعدلات للنتائج المختبرية والتنبؤات المحسوبة. يعتبر التنبؤ الجيد للنموذج عاملاً مشجعاً لاستخدامه بدلاً من الفحص ألمختبري اختصاراً للوقت وتقليصاً للكلفة.***

## Introduction:

Soundness of Portland cement means that it would not undergo large change in volume after setting. Such volume changes may result from the slow hydration of certain of its constituents namely free lime, magnesia and calcium sulphate [1]. Autoclave expansion test, according to the ASTM C151 [2], is one of the internationally used tests in detecting the unsoundness of Portland cements. This test is, to some extent, complicated and needs time and professional staff. In addition to that, in Iraq, the equipment for test is not available in each laboratory.

Mathematical modeling process is a simplified representation of reality designed to fulfill a specific purpose. There are many reasons that made dealing with models preferable to dealing with real world. Often, the motivation is economic, to save money, time, or other valuable commodity [3].

Cement industry in Iraq was established since mid-forties of the last century. Iraqi cements are well complying with international standards such as ASTM C 150 [4] and BS 12-1991 [5].

This work is specified to build a statistical model for predicting autoclave expansion for Portland cements that are produced in Iraq.

## Autoclave Expansion Test:

As prescribed by the ASTM C151 [2], this test is carried out as follows:

1. At 24 h ± 30 min after molding, the specimens (25.4 × 25.4 × 250 mm) are removed from the moist atmosphere and immediately a length comparator reading for each specimen is obtained. After that specimens are placed in the autoclave (a high-pressure steam vessel) at room temperature. The autoclave shall contain enough water, at initial temperature of 20 to 28 $^{o}$C, to maintain an atmosphere of saturated steam vapor during the entire test.
2. The temperature of the autoclave is raised at a rate that will bring the gage pressure of the steam to 2 MPa in 45 to 75 min. The pressure of 2 ± 0.07 MPa will be maintained for 3 h, then the heat supply is shut off and the autoclave is cooled at such a rate that the pressure will be less than 0.07 MPa at the end of 1.5 h. Slowly, any remaining pressure is released until atmospheric pressure is attained.
3. The autoclave is opened and the specimens are placed in water at temperature above 90 $^{o}$C. The water will be cooled at a uniform rate by adding cold water so that the temperature of the water will be lowered to 23 $^{o}$C in 15 min. The temperature is maintained at 23 $^{o}$C for another 15 min.
4. One specimen at a time is removed, and a length comparator reading is obtained. The autoclave expansion is calculated by subtracting the length reading before autoclaving from that after autoclaving and reporting that as percent of effective gage length.

According to the ASTM C150 [4], the expansion must not exceed 0.8 percent for all types of Portland cement.

## Factors Affecting Autoclave Expansion:

The main factor which is governing the expansion is the free CaO and MgO contents in cement. The expansion is due to the formation of calcium hydroxide, $Ca(OH)_2$, and magnesium hydroxide, $Mg(OH)_2$, upon the delayed hydration of free CaO and MgO respectively. Chatterji [6] stated that the mechanism of expansion for both oxides is the same and its capacities for free CaO is more due to that $Ca(OH)_2$ is more soluble than $Mg(OH)_2$.

Moreover, as reported by Lea [1], it was found that there is an interrelationship between these two oxides on their effect on expansion. He quoted that cement with free CaO below 2 % will pass the autoclave test when the total MgO content is low (1 – 2) %. But with high contents of MgO, the free CaO may need to be below 1 % to get a safe situation.

Neville [7] indicated that autoclave expansion test is sensitive to MgO and free CaO but not to calcium sulphate, $CaSO_4$. In opposite to that, many researchers had pointed out that $CaSO_4$ does affect the results of the autoclave test. Lerch [8] indicated, in many cases, that the sulphate content for highest strength was corresponding to that which yields lowest autoclave expansion. Messiner *et al.* [9] had confirmed the results obtained by Lerch [8]. Samdi *et al.* [10] stated that the autoclave expansion, for mixes with different sulphate contents, increased with the increase of sulphates. Furthermore, Abdullatif [11] had suggested making use of the autoclave test to determine the optimum gypsum content of concrete. His proposal was built according to his conclusion that increasing the sulphate in cement would result in higher autoclave expansion. Al-Jabiri [12] studied the effect of both MgO and sulphate contents in cement on concrete strength development. Her results, with respect to autoclave expansion, were in agreement with that of Samdi et al. [10] and Abdullatif [11]. **(Figure .1)** shows the relationship between autoclave expansion and sulphate content of cement after Abdullatif [11] and Al-Jabiri [12]. It is noteworthy to mention here that each cement of the two shown in this figure has constant free CaO and MgO and the only variable parameter is sulphate content.

In addition to the chemical composition of cement, fineness is considered as another major factor that affecting the autoclave expansion. Lea [1] stated that the degree of unsoundness depends on the crystal size since smaller crystals tend to hydrate the more rapidly or without producing excessive internal pressure.

Czernin [13] found that with increasing finer particles of free CaO the expansion became less and more regular. Chopra *et al.* [14] showed that grinding a clinker, which has high MgO content, to higher fineness, could be adopted as an effective measure to obtain sound cement. Chatterji [6] attributed this behavior to that the larger the original oxide particles, the larger is the volume of enclosed fine hydroxide crystals and higher is the volume of possible elongated crystal growth and so also the expansion capacity.

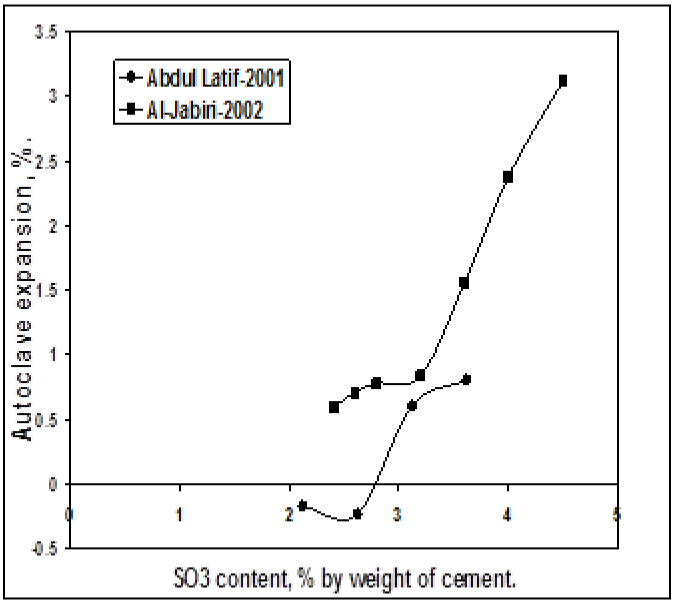


**Fig .(1) The autoclave expansion versus sulphate content of cement according to Abdullatif [11] and Al-Jabiri [12].**

## Statistical Analysis and Mathematical Modeling:

### Data Source:

The quality control (QC) records of four Iraqi factories production were reviewed. These factories were:

1. Kubaisa factory (producing Ordinary Portland cement- Type I).
2. Karkuk factory (producing Ordinary Portland cement- Type I).
3. Al-Qaim factory (producing Sulphate Resisting Portland cement- Type V).
4. Al-Falluja factory (producing White Portland cement).

The records comprised test results that required by the ASTM C150 [4] (chemical composition and physical properties).

The chosen data were for the production of the years 1995-2000. A sample consisting of 130 cases was selected for statistical analysis and each case was representing the average test result for one month production in any factory.

### Variables Selection:

The autoclave expansion (Auto) was chosen to be the dependent variable for the built model. A set of all conceivable variables, that may be considered to have an effect on the autoclave expansion, was selected. Then after, the correlations between these variables and the dependent variable were calculated and tested within the available sets of observations. (**Table .1)** shows these correlations in a matrix form. The sign (*) denotes that the variable is correlating significantly with the expansion at 1 percent significance level. Accordingly, some variables were omitted because they have no significant effect on the autoclave expansion. The final set of variables included:

1. fCaO: free lime content of cement, %.
2. MgO: magnesia content of cement, %.
3. $SO_3$: sulphate content of cement, %.
4. Fin: Fineness of cement, $m^2$/kg (Blaine method).

**Table .(1) The correlation matrix**

| Variable | Auto | fCaO | MgO | $SO_3$ | Fin | $C_3A$ | $C_4AF$ |
|---|---|---|---|---|---|---|---|
| **Auto** | 1.000 | 0.646* | 0.830* | 0.330* | -0.275* | 0.137 | -0.004 |
| **fCaO** | 0.646* | 1.000 | 0.079 | -0.107 | 0.016 | 0.029 | 0.088 |
| **MgO** | 0.830* | 0.079 | 1.000 | 0.385* | -0.158 | 0.233* | -0.165 |
| **$SO_3$** | 0.330* | -0.107 | 0.385* | 1.000 | 0.394* | 0.686* | -0.673* |
| **Fin** | -0.275* | 0.016 | -0.158 | 0.394* | 1.000 | 0.784* | -0.826* |
| **$C_3A$** | 0.137 | 0.029 | 0.233* | 0.686* | 0.784* | 1.000 | -0.959* |
| **$C_4AF$** | -0.004 | 0.088 | -0.165 | -0.673* | -0.826* | -0.959* | 1.000 |

*: Correlation is significant at the 0.01 level

The range (minimum and maximum values) of the selected variables with their means and standard deviations for the available data are shown in **(Table .2).** It is clear that the chosen data is covering wide ranges of variation for the selected variables.

**Table. (2) The descriptive statistics of the selected variables**

| Variable | Minimum | Maximum | Mean | Standard dev. |
|---|---|---|---|---|
| **Auto** | 0.03 | 0.39 | 0.18 | 0.099 |
| **fCaO** | 0.20 | 2.08 | 1.31 | 0.44 |
| **MgO** | 0.47 | 4.10 | 2.34 | 1.10 |
| **$SO_3$** | 1.84 | 2.77 | 2.38 | 0.21 |
| **Fin** | 262.00 | 485.00 | 331.71 | 53.10 |

## Mathematical Model Building:

According to above- mentioned affecting factors, the following general form was decided to represent the model:

$$Auto = A_0\left[\frac{(fCaO + MgO)^{A_1} + SO_3^{A_2}}{Fin^{A_3}}\right] \quad \textbf{(1)}$$

The reasons behind selecting such a form are:

1. Choosing a linear form would not represent the variation and behavior of the selected variables.
2. The expansion mechanism is similar and interrelated for both free lime and magnesia; meanwhile, it is different for sulphate. Moreover, free CaO and MgO are more effective on autoclave expansion than $SO_3$.
3. The current form could be used efficiently in explaining the relationships between the dependent and independent variables.

The data-sample, that collected from quality control (QC) records, was divided into two groups. The first, which contains 105 cases, was adopted to build the model. The second was selected for testing the validity of the model. The splitting was done by random numbers method. The size of each group was decided according to a recommended method (see Ref 15, p.417).

Nonlinear regression analysis was done with the aid of computer software SPSS to solve for the model coefficients. **(Table 3**) shows the values of coefficients and their 95% confidence intervals. By these coefficients, the model would possess a coefficient of determination, $R^2$, equals to 0.97 as shown in **(Table 4).**

**Table .(3) Values of coefficients and their 95% confidence intervals**

| Coefficient | Estimate | 95% Confidence Interval | |
|---|---|---|---|
| | | Lower | Upper |
| $A_0$ | 0.01515 | -0.038022 | 0.0683144 |
| $A_1$ | 2.05817 | 1.635250 | 2.481086 |
| $A_2$ | 2.0350 | 0.299689 | 3.770301 |
| $A_3$ | 0.11262 | -0.525670 | 0.750921 |

**Table .(4) Analysis of variance for the built model**

| Source of Variance | Degree of Freedom | Sum of Squares | Mean Square | F | $R^2$ |
|---|---|---|---|---|---|
| **Regression** | 4 | 4.51279 | 1.128198 | 739.80 | 0.97 |
| **Residual** | 101 | 0.15401 | 0.001525 | | |
| **Total** | 105 | 4.6668 | | | |

## Validation of the Model:

Examining for residuals (errors):

The residual is the amount which the regression equation cannot be able to explain. In performing regression analysis, the following two assumptions are made [15]:

1. The residuals should have a zero mean and a constant variance, and
2. They should follow a normal distribution.

**(Figure 2)** shows that the residuals are normally distributed and have a mean approaches to zero.

**Using new data:**

The data of the second group (consists of 25 cases) were used for testing the model. These data were not included in model building. (**Figure 3**) shows the relationship between experimental and predicted autoclave expansion. The correlation coefficient between experimental and predicted values was equal to (r = 0.92). Moreover, a t-test was carried out to detect if there is a difference between means of experimental and predicted sets of values. At 1 percent significance level, the calculated t-value was - 0.873 and the tabulated t-value was 0.391. Because of the $t_{cal}$ being less than $t_{tab}$, the hypothesis that there is no difference between means is accepted.

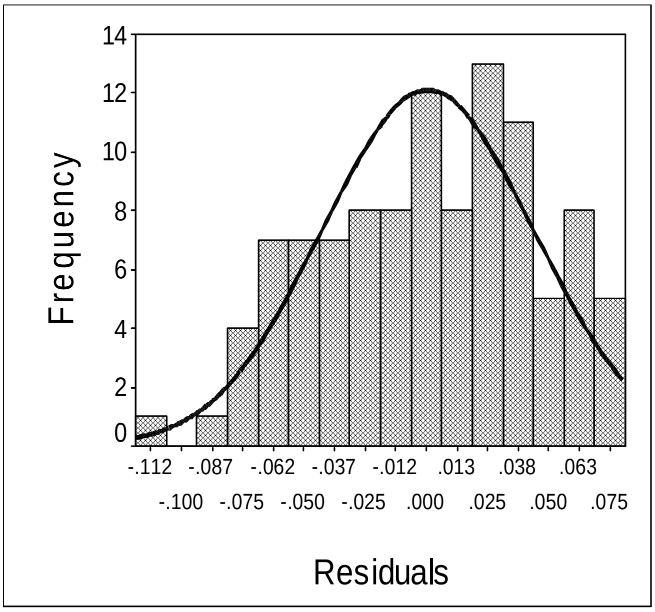


**Fig .(2) The distribution of residuals.**

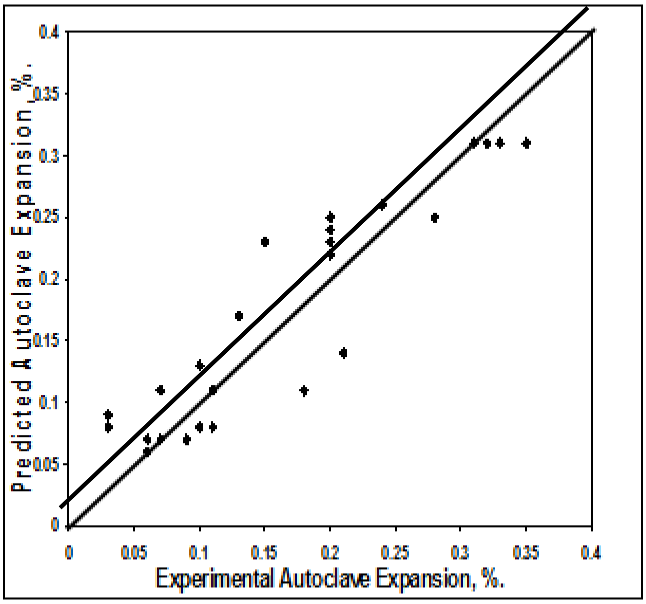


**Fig .(3) The relationship between experimental and predicted autoclave expansion (testing the model with new data - 25 cases).**

## Concluding Remarks:

The followings are the concluding remarks that could be extracted from the present work:

1. There is a possibility to predict the autoclave expansion for Portland cements to a reliable degree according to the chemical composition. This would save time and be an economical measure.
2. A mathematical model was developed and tested. This model possessed $R^2$ equals to 0.97.
3. The built model showed the ability to explain most of the known relationships between included variables.

## References:


1. **Lea, F. M. (2004).** ***The chemistry of cement and concrete*****, Elsevier Science & Technology Books, London, 1057 pp**
2. **ASTM. (2005). "Test for autoclave expansion of Portland cement – ASTM C 151-05" Philadelphia, USA.**
3. **Moscardini, A. O., and Cross, M. (1983). "Issues involved in the design of a mathematical modeling course." Proceeding of the 4th international conference of mathematical modeling in science and technology, Zurich, pp. 954-963**
4. **ASTM. (2005). "Standard specification for Portland cement – ASTM C 150-05" Philadelphia, USA.**

5. **British Standards Institution (1991). "Specification for Portland cements - BS 12-1991", UK.**
6. **Chatterji, S. (1995). "Mechanism of expansion of concrete due to the presence of dead – burnt CaO and MgO." Cement & Concrete Research, V. 25, pp. 51-56.**
7. **Neville, A. M. (1995). "Ch.1: Portland cement."** ***Properties of concrete,*** **Longman, Essex, 884 pp.**
8. **Lerch, W. (1946). "The influence of gypsum on the hydration and properties of Portland cement pastes." ASTM Proceedings, V. 46, pp. 1252-1292.**
9. **Meissner, H. S., Cook, H. K., and Gonnerman, H. F. (1950). "The optimum gypsum content of Portland cement." ASTM Bulletin, V. 139, pp. 39-45.**
10. **Samdi, M. M., Haddad, R. H., and Akour, A. M. (1999). "Potential use of phosphogypsum in concrete." Cement and Concrete Research, V. 29, pp. 1419-1425.**
11. **Abdullatif, A. M. (2001). "Mathematical model for optimum gypsum content in concrete"** ***PhD thesis,*** **University of Baghdad, Iraq, 152 pp.**
12. **Al-Jabiri, T. M. (2002). "Concrete strength development related to MgO and $SO_3$ contents in concrete."** ***MSc thesis,*** **University of Baghdad, Iraq, 81 pp.**
13. **Czernin, W. (1980).** ***Cement chemistry and physics for civil engineering*****. $2^{nd}$ English Edition, George Godwin Ltd., London.**
14. **Chopra, S. K., Narang, K. C., Ghosh, S. P., and Sharma, K. M. (1980). "Studies on unsoundness of clinker with below 3.5 percent MgO content." Proceedings of the $7^{th}$ International Conference on Chemistry of Cement, Paris, V. 3, Communications, VII, pp. 51-56.**
15. **Draper, N. R., and Smith, H. (1981),** ***Applied regression analysis,*** **John Wiley, New York, 709 pp.**