

## The Effect of Alkalis on The Properties of Portland Cement

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### ABSTRACT.

Portland cement consists of major oxides which include CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> as well as minor oxides which include: SO<sub>3</sub>, MgO, Na<sub>2</sub>O and K<sub>2</sub>O, the last two oxides are called alkalis oxides.

The research aims to study the effect of alkalis oxide ( Na<sub>2</sub>O+ K<sub>2</sub>O) on some physical properties of ordinary Portland Iraqi cement (type I) and sulfate - resisting Portland Iraqi cement (type V) provided from (Taslooja Factory) are used in the experimental work. The physical properties of the two types above, which are used in the experimental work, are initial and final setting time, soundness and compressive strength at (3, 7 and 28)days.

The results show that the values of physical properties of type I and type V increase when the alkalis percentage increases up to 0.6 percent, while the value of the physical properties of the two types of cement mentioned above begins to reduce even when the percentage of alkalis still increases.

Through the use of the ordinary Portland cement (type I) and the sulfate resisting Portland cement (type V), it is found that there is a little difference in the value of the physical properties.

**Keywords: Alkali, Percentage, Portland cement, Tests and Physical Properties**

### 1. INTRODUCTION.

Portland cement is essentially a calcium silicate cement, which is produced by firing to partial fusion, at a temperature of approximately 1500°C, a well-homogenized and finely ground mixture of limestone or chalk (calcium carbonate) and an appropriate quantity of clay or shale. The composition is commonly fine tuned by the addition of sand and/or iron oxide.

Cement making is essentially a chemical process industry and has much in common with the manufacture of so-called heavy chemicals such as sodium hydroxide and calcium chloride. Close control of the chemistry of the product is essential if cement with consistent properties is to be produced. This control applies not only to the principal oxides which are present but also to impurities, which can have a marked influence on both the manufacturing process and cement properties.

Cement consists mainly of four oxides: CaO (lime), SiO<sub>2</sub> (silica), Al<sub>2</sub>O<sub>3</sub> (alumina) and Fe<sub>2</sub>O<sub>3</sub> (iron oxide). In order to simplify the description of chemical composition, a form of shorthand is used by cement chemists in which the four oxides are referred to respectively as C, S, A and F [1].

In addition to the main compounds, there exist minor compounds, such as MgO, TiO<sub>2</sub>, Mn<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, and Na<sub>2</sub>O...etc; they usually not to be more than a few per cent of the mass of cement. Two of the minor compounds are of interest: the oxides of sodium and potassium, Na<sub>2</sub>O and K<sub>2</sub>O, known as the alkalis (although other alkalis also exist in cement). Approximate limits of alkalis in Portland cement 0.3–1.2 [1]. They have been found to react with some aggregates, the products of the alkali–aggregate reaction causing disintegration of the concrete, and have also been observed to affect the rate of the gain of strength of cement[2].

Burrows and Jawed [2] concluded that the effects of alkalis on various properties of cement paste, mortar, and concrete, including early hydration and setting, bleeding, strength development and ultimate strength, drying shrinkage, susceptibility to cracking, micro-texture of cement hydrates, and durability, have been a subject of concern since many decades.

Stark [3] concluded that significant proportions of alkalis in clinker can cause a quick setting, reduce the ultimate strength of concrete, and increase expansion under water and shrinkage under drying conditions.

## **2. TESTS ON CEMENT.**

A number of tests are performed in the cement plant laboratory to ensure that the cement is of the desired quality and that it conforms to the requirements of the relevant national standards. To examine the properties of a cement to be used for some special purpose such as chemical composition, fineness tests, setting times, soundness tests and strength tests, will now be briefly described [1,4] .

### **2.1.Consistence of Standard Paste.**

For the determination of the initial setting time, the final setting time, and soundness tests, neat cement paste of a standard consistence has to be used. Therefore, it is necessary to determine for any given cement the water content which will produce a paste of standard consistence. Consistence is determined by the Vicat apparatus, which measures the depth of penetration of a 10 mm diameter plunger under its own weight.

### **2.2.Setting Time.**

This is the term used to describe the stiffening of the cement paste. Setting refers to a change from a fluid to a rigid state. Setting is mainly caused by a selective hydration of C3A and C3S and is accompanied by temperature rises in the cement paste; initial set corresponds to a rapid rise and final set corresponds to the peak temperature. Initial and final sets should be distinguished from false set which sometimes occurs within a few minutes of mixing with water. No heat is evolved in a false set and the concrete can be re-mixed without adding water. Flash set has previously been mentioned and is characterized by the liberation of heat. For the determination of initial set, the Vicat apparatus is again used, this time with a 1 mm diameter needle, acting under a prescribed weight on a paste of standard consistence. When the needle penetrates to a point 5 mm from the bottom of a special mould, initial set is said to occur (time being measured from adding the mixing water to the cement). A minimum time of 45 min is prescribed by (I.Q.S) for Portland cements but ( BS EN 197–1), a minimum time of 45 min for cements of strength classes 52.5 MPa and 62.5 MPa whereas 60 minutes applies to strength classes of 32.5 MPa and R and 42.5 MPa.

A similar procedure is specified by ASTM C 191 except that a smaller depth of penetration is required; a minimum setting time of 60 min is prescribed for Portland cements (ASTM C 150–05) .

Final set is determined by a needle with a metal attachment hollowed out so as to leave a circular cutting edge 5 mm in diameter and set 0.5 mm behind the tip of the needle. Final set is said to have occurred when the needle makes an impression on the paste surface but the cutting edge fails to do so. British Standards prescribe the final setting time as a maximum of 10 hours for Portland cements, which is the same as that of the Iraqi Standards but American Standards are prescribed the initial and final setting times are approximately related:

$$\begin{aligned} \text{final time (min.)} &= 90 + 1.2 [\text{initial time (min.)}] && (1-1) \\ &(\text{except for high alumina cement}) \end{aligned}$$

### **2.3. Soundness.**

It is essential that the cement paste, once it has set, does not undergo a large change in volume. The expansion may occur due to reactions of free lime, magnesia and calcium sulfate, and cements exhibiting this type of expansion are classified as unsound. Free lime is present in the clinker and it hydrates very slowly occupying a larger volume than the original free calcium oxide. Free lime cannot be determined by chemical analysis of cement because it is not possible to distinguish between unreacted CaO and Ca(OH)<sub>2</sub> produced by a partial hydration of the silicates when the cement is exposed to the atmosphere. Magnesia reacts with water in a manner similar to CaO, but only the crystalline form is deleteriously reactive so that unsoundness occurs. Calcium sulfate is the third compound liable to cause expansion through the formation of calcium sulfoaluminate (ettringite) from excess gypsum. Le Chatelier's accelerated test is prescribed by BS EN 196-3: 1995 for detecting unsoundness due to free lime only.

### **2.4. Strength.**

Strength tests are not made on neat cement paste because of difficulties in obtaining good specimens and in testing with a consequent large variability of test results. The British Standard method for testing the compressive strength of cement BS EN 196-1: 2005 specifies a mortar prism test. ASTM C 109-05 prescribes a cement-sand mix with proportions of 1:2.75 and a water/cement ratio of 0.485, using standard sand (ASTM C 778-06) for making 50 mm cubes and are cured in saturated water at 23 °C until they are tested.

## **3. ALKALI COMPOUNDS.**

The alkalis, sodium and potassium (Na<sub>2</sub>O, K<sub>2</sub>O), in Portland cement clinker are derived mainly from the clay components present in the raw mix and coal; their total amount, expressed as Na<sub>2</sub>O<sub>e</sub> equivalent (Na<sub>2</sub>O + 0.658K<sub>2</sub>O), contain no more than 0.60 percent equivalent alkali. The sulfates in a cement kiln generally originate from fuel. Depending on the amount of sulfate available, alkali presence is known to have a significant influence on the early hydration reactions of the cement. When sufficient sulfate is not present in the kiln system, the alkalis are preferentially taken up by C<sub>3</sub>A and C<sub>2</sub>S, which may then be modified to compositions of the type NC<sub>8</sub>A<sub>3</sub> and KC<sub>23</sub>S<sub>12</sub>, respectively [2,5].

The alkalis in the cement in an amount sufficient to cause excessive expansion except that if such materials are present in injurious amounts, the aggregate may be used with a cement containing less than 0.6 percent alkalis or with the addition of a material that has been shown to prevent harmful expansion due to the alkali-aggregate reaction.

## **4. EFFECTS OF ALKALIS ON STRENGTH DEVELOPMENT AND ULTIMATE STRENGTH.**

Odler and Wonnemann [6] observed that the alkalis incorporated into the cement clinker did not affect the compressive strength, whereas an external addition of alkali sulfate considerably reduces the strength at any age up to 28 days. Mullick [7] observed that the higher the alkali increase, the lower the compressive strength of cement pastes; however, intermediate alkali additions increased the modulus of rupture whereas smaller and larger additions decreased it.

Concrete was presenting the lowest 7-day compressive strength of all ASTM Type I cements investigated, which is coherent with a lower early rate of hydration. Vivian [3] subjected to flexure tests mortar specimens containing alkali-silica reactive and nonreactive aggregates. The mortars were made with a cement containing 0.59% Na<sub>2</sub>O<sub>eq</sub>, and NaOH additions of 0%, 0.5%, 2%, and 4.5% (by mass of cement), which correspond to total alkali contents of 0.59%, 0.98%, 2.14%, and 4.08% Na<sub>2</sub>O<sub>e</sub>, respectively (by mass of cement). It must be

mentioned that all mortars with a lower alkali content were made with a water-to-cement (w/c) ratio of 0.50 or 0.52, whereas a higher ratio (0.60) was used for the 4.08% Na<sub>2</sub>O<sub>eq</sub> mixture. The researcher thinks that when the amount of alkalis is greater than (0.6%) because the alkalinity of the solution will be greater, so the hydration of cement will be slower, and that could explain the decrease in strength by itself [3]. Shayan and Ivanusec [2] studied the changes in the mechanical properties (compressive strength and modulus of rupture) and micro structural characteristics of cement pastes and mortars of various alkali contents. The authors found that the higher the alkali content to the level (1.0, 2.0 and about 3% Na<sub>2</sub>O<sub>eq</sub>), the lower the compressive strength and modulus of rupture at any age (i.e., 7, 28, and 90 days). When alkali ions were added to water mixture, the setting times are shorter and the later strengths are lower. However, 0.5% of Na<sub>2</sub>O<sub>e</sub> addition gave high early compressive strengths (40% more than developed by reference specimens). In addition, the final compressive strength developed by mortar specimens (90 days) with Na<sub>2</sub>O<sub>3</sub> is so 20% smaller than reference specimens [6, 8].

## **5. EFFECTS OF ALKALIS ON MICRO-TEXTURE.**

According to Jawed and Skalny [2], “when alkali-containing cement is mixed with water, the alkali metal ions readily go into the liquid phase of the hydrating system and influence the rate of cement hydration and the morphology of the hydration products. This, in turn, affects the strength and other engineering properties of the concrete. ; Thaumaturgo report from the literature that the microstructure of CSH becomes coarser and heterogeneous in alkali solutions and the lower strength development at later age is due to this heterogeneous structure [3]. Mullick [7] reported that in the presence of high alkali contents, the products of hydration tend to be gelatinous rather than crystalline and observed that high-alkali cement pastes had a less dense microstructure compared to low-alkali cement past.

## **6. EXPERIMENTAL WORK.**

### **6.1. Materials.**

#### **6.1.1. Cement:**

Ordinary Portland cement (OPC) (type I) and Sulfate Resisting Portland cement (SRPC) (type V). This cement is manufactured in Iraq by Taslooja factory and commercially known (Taslooja). **Table (1) and Table (2)** show the chemical and physical properties for (type I) and (type V) cement respectively. These properties of cement comply with Iraqi Standard Specification I.Q.S. No.5, 1984 requirement [9].

#### **6.1.2. Sand:**

Natural siliceous sand of Al-Ukaider region with maximum size 4.75mm and fineness modulus, specific gravity and absorption of 2.98, 2.7 and 1.5% respectively is used in this work. **Table (3)** shows the grading of sand and complies with Standard Specification I.Q.S.No.45, 1984 requirement [10].

### **6.2. Mix Design.**

Several mixtures were tested in order to study the effect of percentage of alkali content on properties of Ordinary Portland cement (type I) and Sulfate Resistance Portland cement (type V).

### **6.3. Testing of Hydraulic Cement.**

#### **6.3.1. Setting Time:**

The Vicat needle is used as test method to determine the initial and final setting time of hydraulic cement, according to ASTM C191-01 [11].

#### **6.3.2. Compressive Strength:**

According to ASTM C109/C109M-02 [12], the mortar used consists of (1 part) of cement and (2.75 parts) of sand are mixed at (0.485) water/ cement ratio. For testing compressive strength for cement mortar, 3 cube of size (50X50X50) mm for each mixture were cast and are compacted by tamping in two layers. The cubes are cured one day in the moulds and stripped and immersed water test.

#### **6.3.3. Soundness:**

This test method determination of the autoclave expansion of Portland cement and is conducted according to ASTM : C 151 – 00 [13].

## **7. RESULTS AND DISCUSSION.**

The results obtained from the experimental work are analyzed and studied. This study includes the effect of alkali on (initial and final) setting time , compressive strength at (3, 7, 28) days and soundness of Ordinary Portland cement type (I) compared with Sulfate Resistance Portland cement type (V). The results are analyzed by using statistic software. Where the relationship between results is selected from the best coefficient of correlation (R).

### **7.1. Result of Setting Time.**

The results of initial and final setting time for cement type (I) and type(V) are shown in **Tables (4) and(5)** respectively while **fig. (1) and (2)** show the relation between alkali content and initial setting time and final setting time respectively. The initial and final setting time for SRPC is (6.8% and 14%) respectively which is lower than of OPC at the optimum ratio. It shows that the microstructure of the cement will be changed and become weaker the percentages of alkalis become greater than (0.6%).

### **7.2. Result of Soundness.**

The results of Soundness for cement type(I) and type(V) are shown in **Table (6)** while **fig. (3)** shows the relation between alkali content and soundness. The soundness for SRPC is (11.1%) which is lower than of OPC at the optimum ratio. It shows that the microstructure of the cement will be changed and become weaker the percentages of alkalis become greater than (0.6%).

### **7.3. Result of Compressive Strength.**

The results of compressive strength at(3,7and 28) days for cement type(I) and type(V) are shown in **Tables (7) and(8)** respectively while **figs. (4) ,(5) and(6)** show the relation between alkali content and compressive strength. The compressive strength of SRPC at (3 ,7 and 28) days is (9% ,7.5% and 3.6%) respectively which is lower than of OPC at the optimum ratio and it shows that the microstructure of the cement will be changed and become weaker the percentages of alkalis become greater than (0.6%).

#### **7. 4.Rate of Development of Compressive Strength Cement Mortar.**

It is well known that the compressive strength of cement mortar increases with age, while it is being cured in water due to the hydration of cement. The following **fig. (7)** shows the relative gain of all strengths for cement mortar concerning the alkali content.

#### **8. CONCLUSION.**

- 1- The alkalis content of cement generally affects of the mechanical properties of cement.
- 2- There is an optimum alkali content which gives the highest compressive strength at 3,7 and 28 days and it's about (0.6%).
- 3- The alkali content affects some physical properties such as initial and final setting time and soundness.
- 4- There is a little difference in the value of optimum alkali content of the two types of cement: Ordinary Portland cement and Sulfate Resistance.
- 5- There is a small difference in the obtained values of physical properties when the type of cement is changed from O.P.C to S.R.C as follows:
  - \* The initial and final setting time for SRPC is (6.8% and 14%) respectively which is lower than of OPC at the optimum ratio.
  - \* The soundness for SRPC is (11.1%) which is lower than of OPC at the optimum ratio.
  - \* The compressive strength of SRPC at (3 ,7 and 28) days is (9% ,7.5% and 3.6%) respectively which is lower than of OPC at the optimum ratio.

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**Table (1):** chemical and physical properties of (Taslooja) for Ordinary Portland Cement Type (I).

Chemical Composition		IQS 5:1984	
Oxides		%	
Calcium oxide	CaO	64.01	
Silicon oxides	SiO <sub>2</sub>	20.05	
Aluminum oxides	Al <sub>2</sub> O <sub>3</sub>	5.15	
Ferric oxides	Fe <sub>2</sub> O <sub>3</sub>	3.27	
Magnesium oxides	MgO	2.8	5.00 max.
Sulfur trioxides	SO <sub>3</sub>	2.6	2.80 max.
Loss on Ignition	L.O.I	2.01	4.00 max.
Insoluble residue	I.R	1.01	1.50 max.
Lime saturation factor	L.S.F	0.96	0.66 -1.02
C3A		8.10	
Physical Properties		Test Result	IQS 5:1984
Fineness: specific surface, Blaine (cm <sup>2</sup> /gm)		270	230 min
Soundness		0.1	0.8
Setting time, Vicat's method:-			
Initial (min)		125	45 min.
Final (hrs:min)		3:5	10 max.

**Table (2):** chemical and physical properties of (Taslooja) for Sulfate- Resisting Portland Cement Type (V).

Chemical Composition		IQS 5:1984 Limits	
Oxides		%	
Calcium oxide	CaO	60.92	
Silicon oxides	SiO <sub>2</sub>	21.88	
Aluminum oxides	Al <sub>2</sub> O <sub>3</sub>	3.96	
Ferric oxides	Fe <sub>2</sub> O <sub>3</sub>	4.28	
Magnesium oxides	MgO	3.15	5.00 max.
Sulfur trioxides	SO <sub>3</sub>	2.46	2.50 max.
Loss on Ignition	L.O.I	2.09	4.00 max.
Insoluble residue	I.R	0.60	1.50 max.
Lime saturation factor	L.S.F	0.86	0.66 -1.02
C3A		3.26	3.5 max
Physical Properties		Test Result	IQS 5:1984
Fineness: specific surface, Blaine (cm <sup>2</sup> /gm)		288	250 min
Soundness		0.17	0.8 max
Setting time, Vicat's method:-			
Initial (min)		66	45 min.
Final (hrs:min)		3:26	10 max.
Compressive strength of cement			
3 days		24.10	15 min.
7 days		36.20	23 min.
28 days		52.00	-----

**Where:** Tests of cement were made in the National Center for Construction Laboratories and Research.

**Table (3):** grading of fine aggregate.

Sieve Size (mm)	% Passing by Weigh	
	%Fine Aggregate Passing	Limits of Iraqi Specification No.45/1984 for Zone2
10	100	100
4.75	94.50	90-100
2.36	87.00	75-100
1.18	76.80	55-90
0.6	45.00	35-59
0.3	12.20	8-30
0.15	0.05	0-10
<b>Fineness of Modulus = 2.96</b>		

**Where:** Tests of cement were made in the National Center for Construction Laboratories and Research.

**Table (4):** initial and final setting time for OPC Type(I) cement.

%Alkali Content	Initial Setting Time (min)	Final Setting Time (hr)
0.25	91	2.4
0.30	100	2.6
0.35	120	3.0
0.40	135	3.2
0.45	146	3.9
0.50	155	4.2
0.55	169	4.7
0.60	175	5.0
0.65	165	4.5
0.70	152	4.1

**Table (5):** initial and final setting time for SRPC Type(V) cement.

%Alkali Content	Initial Setting Time (min)	Final Setting Time (hr)
0.25	80	2.38
0.30	96	2.5
0.35	118	2.9
0.40	127	3.13
0.45	138	3.4
0.50	144	3.7
0.55	156	4.0
0.60	163	4.3
0.65	150	3.85
0.70	146	3.5



**Table (6):** soundness for OPC Type(I) and SRPC Type (V) cement.

<b>%Alkali Content</b>	<b>%Soundness for OPC (Type I)</b>	<b>%Soundness for SRPC (Type V)</b>
0.25	0.05	0.02
0.30	0.07	0.04
0.35	0.09	0.05
0.40	0.11	0.07
0.45	0.13	0.10
0.50	0.14	0.11
0.55	0.17	0.14
0.60	0.18	0.16
0.65	0.15	0.13
0.70	0.12	0.09

**Table (7):** compressive strength(MPa) for OPC Type(I) cement.

<b>%Alkali Content</b>	<b>Compressive Strength (MPa) 3 days</b>	<b>Compressive Strength (MPa) 7 days</b>	<b>Compressive Strength (MPa) 28 days</b>
0.25	20	26	38
0.30	23	29	40
0.35	26	34	42
0.40	29	38	45
0.45	33	41	49
0.50	39	43	50
0.55	41	50	52
0.60	44	53	55
0.65	42	47	52
0.70	37	42	51

**Table (8):** compressive strength(MPa) for SRPC Type(V) cement.

<b>%Alkali Content</b>	<b>Compressive Strength (MPa) 3 days</b>	<b>Compressive Strength (MPa) 7 days</b>	<b>Compressive Strength (MPa) 28 days</b>
0.25	17	23	37
0.30	19	25	38
0.35	21	30	41
0.40	27	35	43
0.45	29	37	47
0.50	34	40	48
0.55	36	45	51
0.60	40	49	53
0.65	38	43	50
0.70	33	39	49

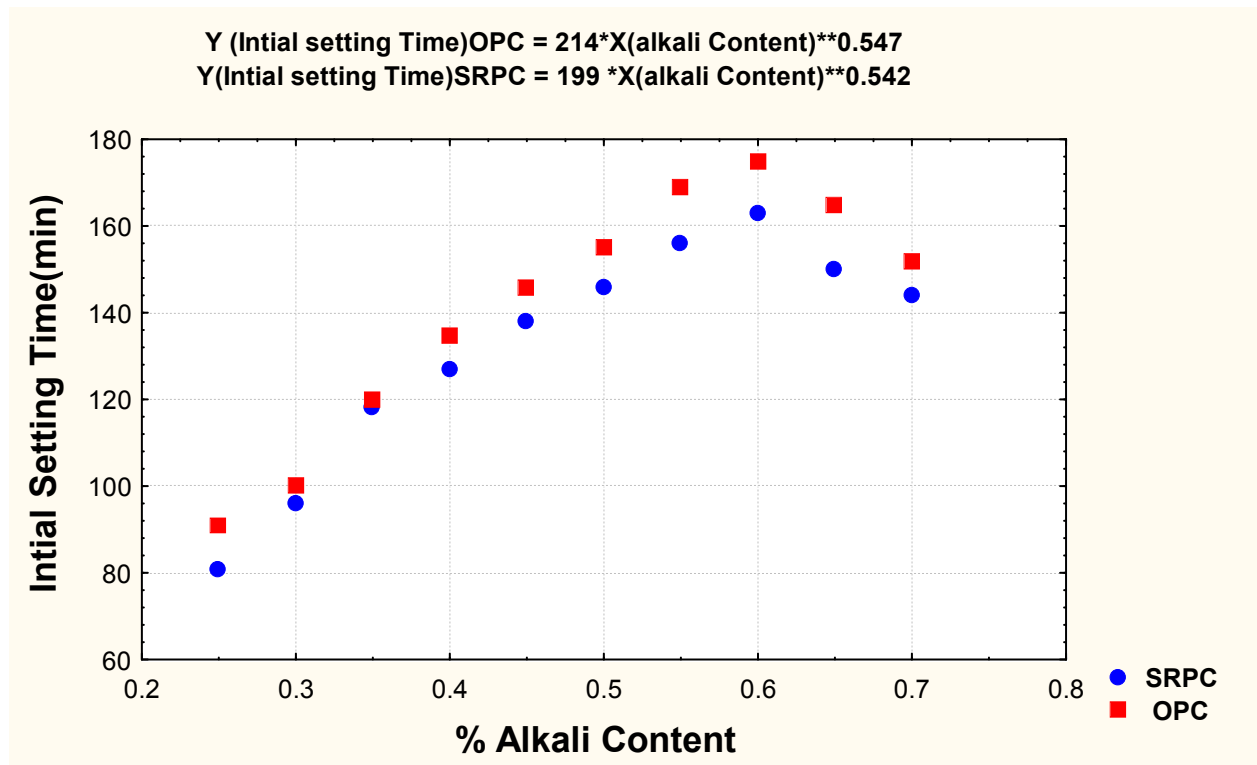


Figure (1): initial setting time for SRPC and OPC versus alkali content

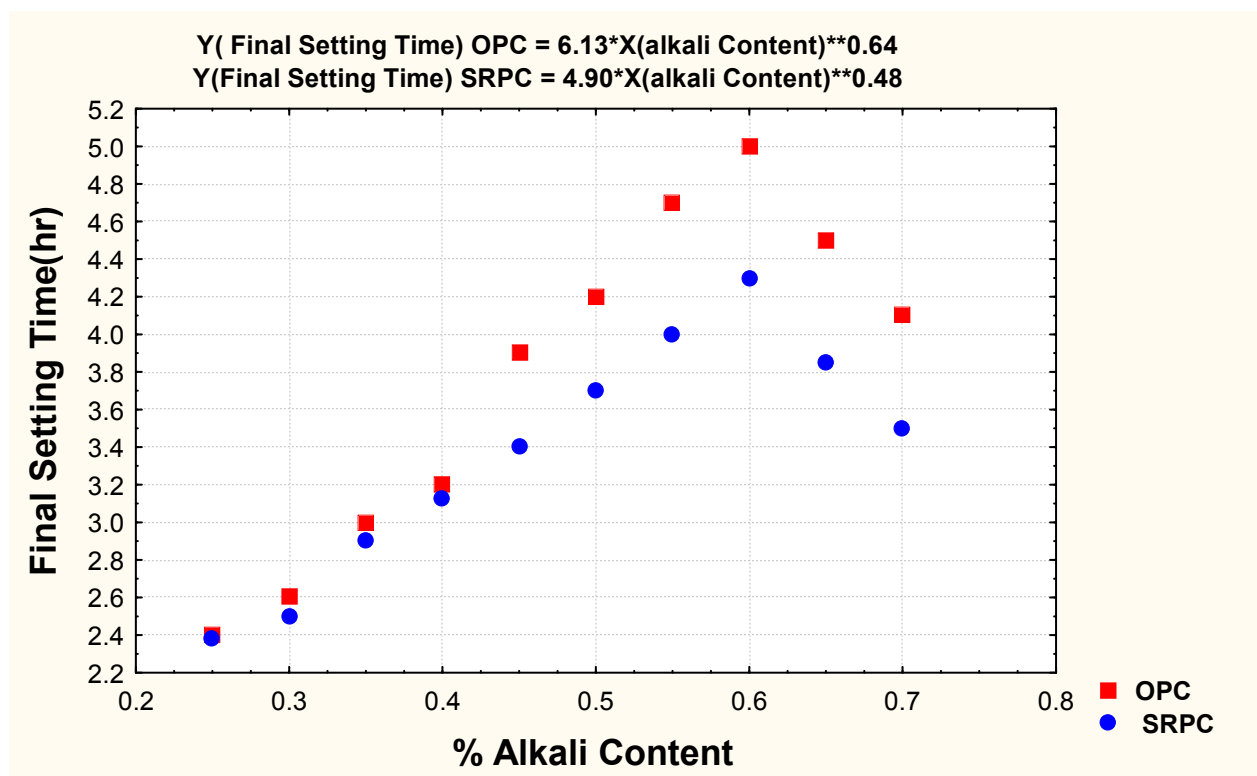


Figure (2): final setting time for SRPC and OPC versus alkali content.

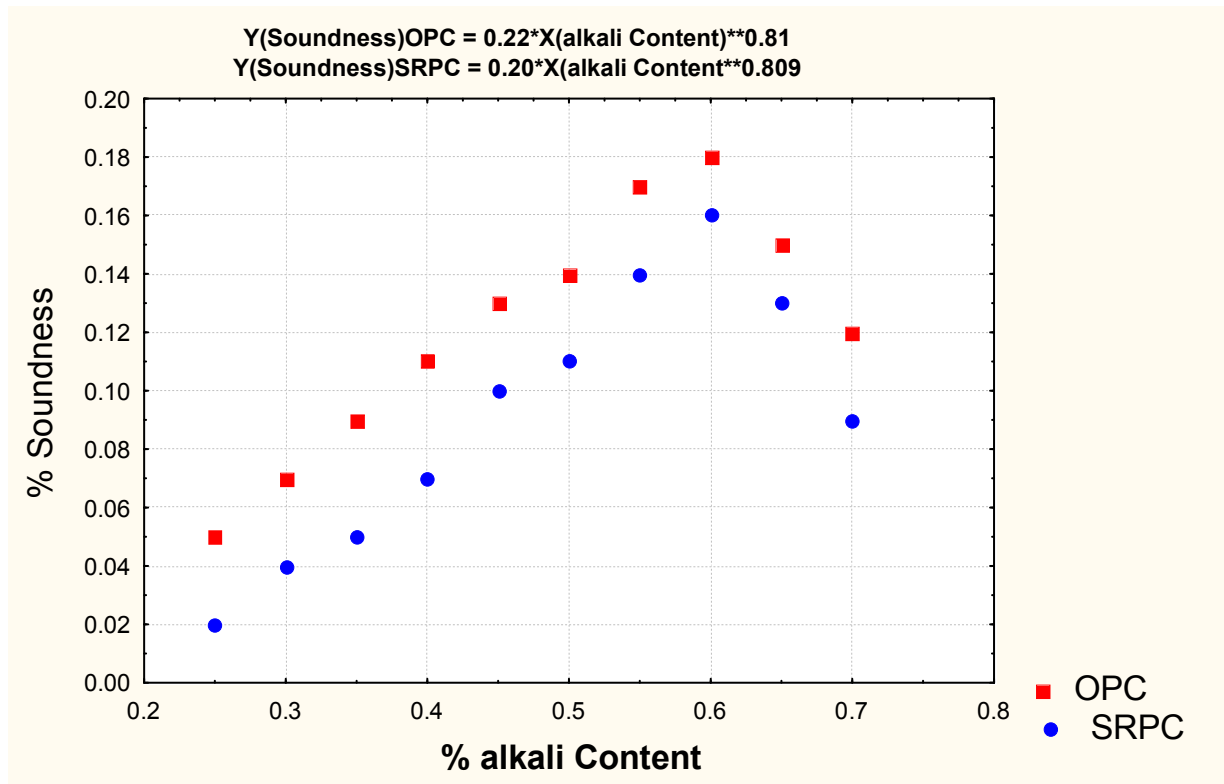
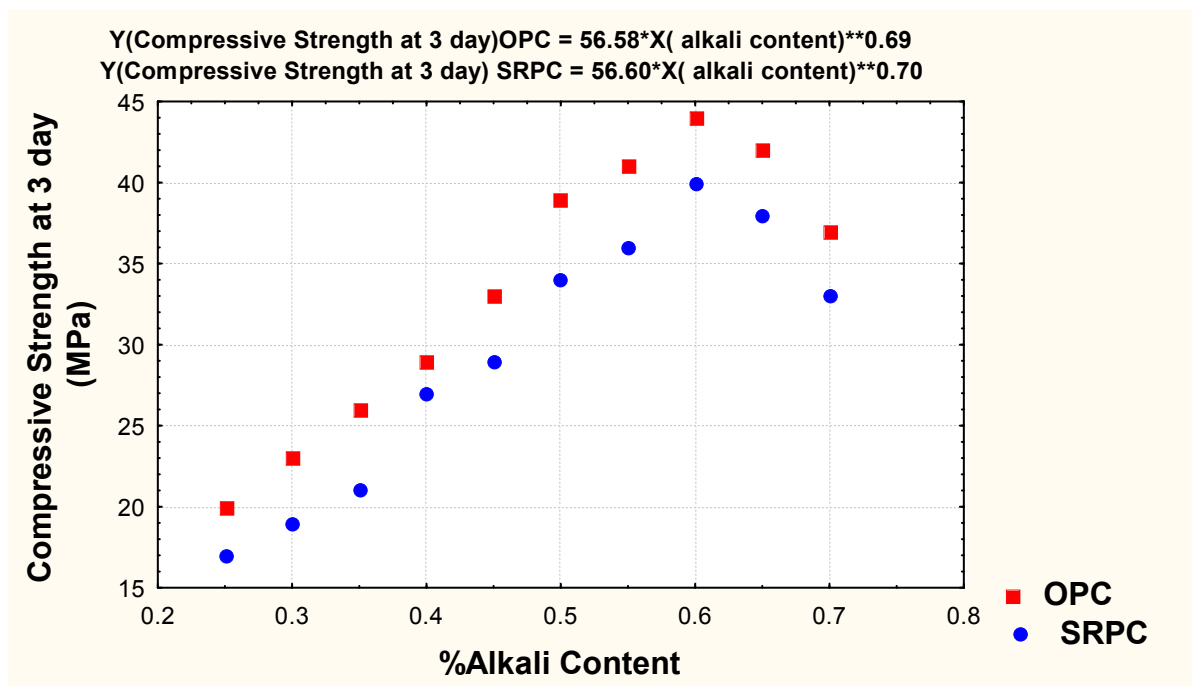


Figure (3): soundness for SRPC and OPC versus alkali content.



Figure(4): compressive strength (MPa) at 3 days for SRPC and OPC versus alkali content.

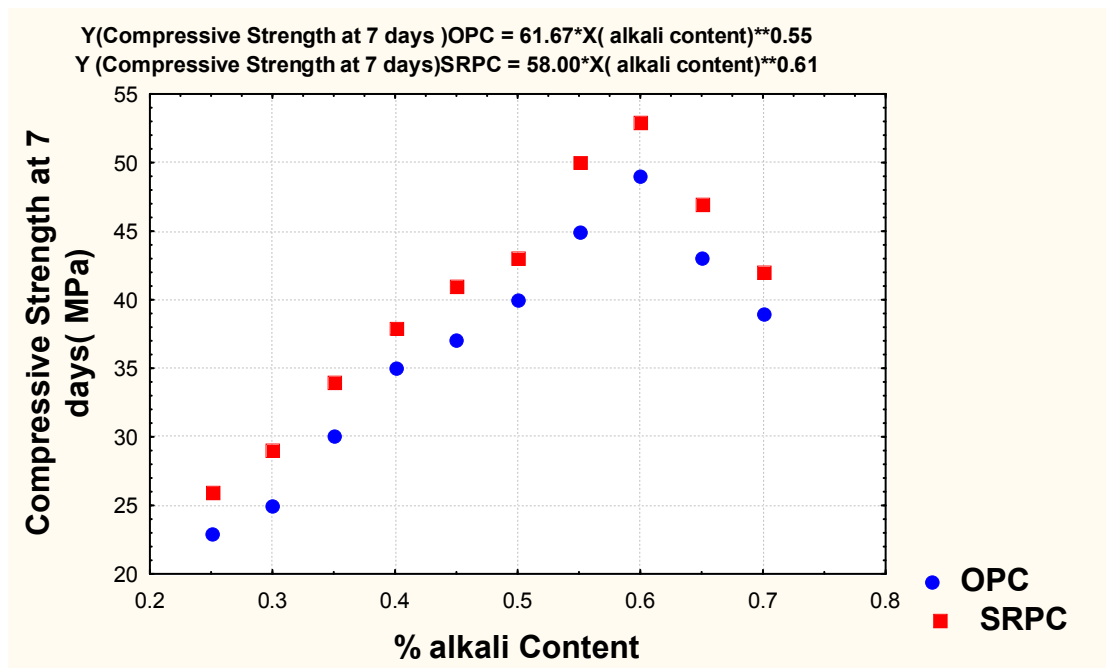


Figure (5): compressive strength (MPa) at 7 days for SRPC and OPC versus alkali content.

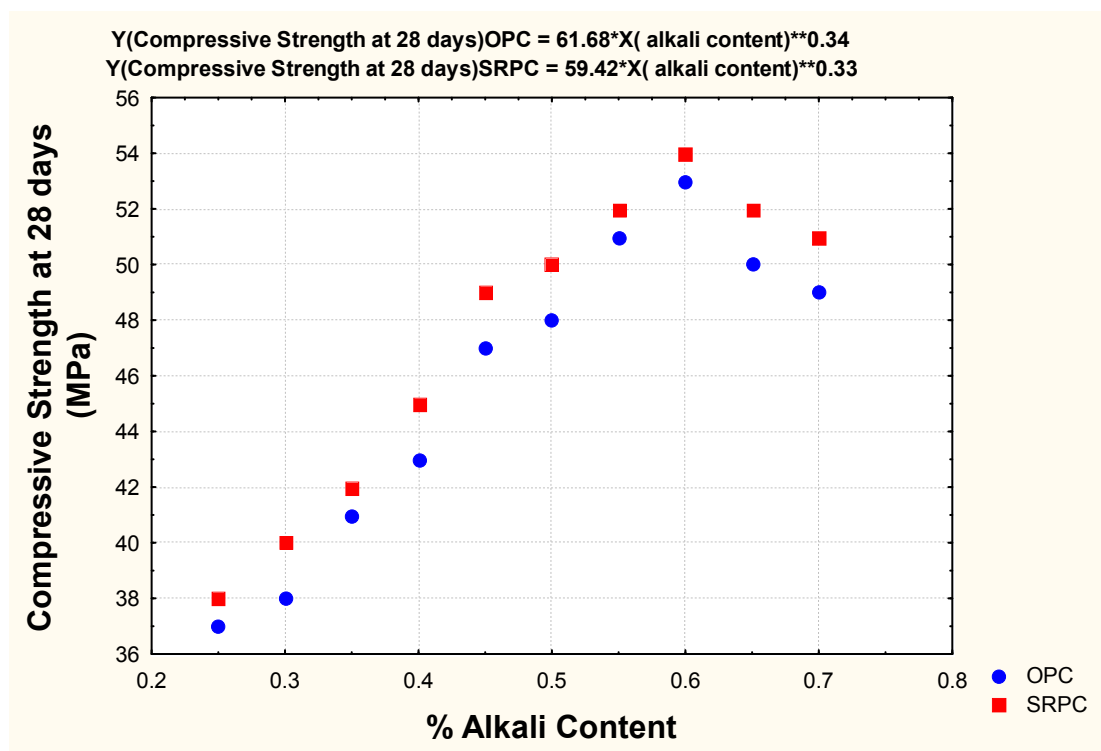


Figure (6): compressive strength (MPa) at 28 days for SRPC and OPC versus alkali content.

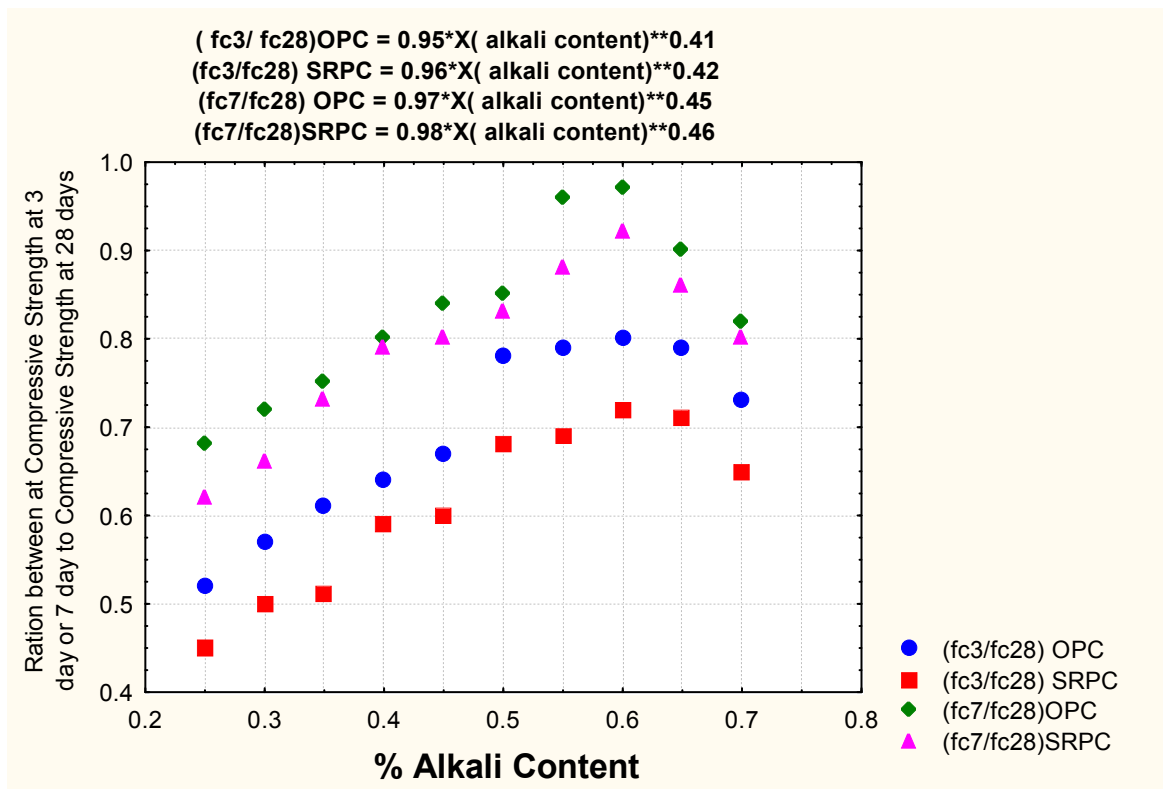


Figure (7): the relative gain of all strengths versus the alkali content.

## تأثير القلويات على خواص السمنت البورتلندي العراقي

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كلية الهندسة-جامعة المستنصرية

### الخلاصة.

يتكون السمنت من الأكاسيد الرئيسية وهي (أكسيد الكالسيوم، أكسيد السليكون، أكسيد الألمنيوم وأكسيد الحديد) والأكاسيد الثانوية و المتمثلة بثالث أكسيد الكبريت، أكسيد المغنيسيوم و أكسيدي الصوديوم والبوتاسيوم والذان يسميان بالأكاسيد القلوية. وهناك أكاسيد ثانوية أخرى بالإضافة إلى الأكاسيد المذكورة في أعلاه. يهدف هذا البحث إلى دراسة تأثير الأكاسيد القلوية ( $K_2O$ ,  $Na_2O$ ) على خواص السمنت البورتلندي العراقي حيث سيتم إجراء تجارب مختبرية على نوعين من أنواع السمنت البورتلندي العراقي المجهز من معمل سمنت طاسلوجه وهما السمنت البورتلندي الاعتيادي Type I و السمنت البورتلندي المقاوم للأملاح Type V , وتحديد وقت التماسك الابتدائي والنهائي و مقاومة الانضغاط و فحص الثبات حيث تم التوصل إلى ان الخواص الفيزيائية التي تم دراستها للسمنت تزداد مع زيادة محتوى السمنت من القلويات حتى نسبة معينه أي عند نسبة (0.6%) تقريبا يحدث انخفاض في نتائج فحص زمن التماسك الابتدائي و النهائي، فحص الثبات وفحص مقاومة الانضغاط) وكذلك لوحظ بأن يوجد اختلاف بسيط ما بين نتائج فحص السمنت البورتلندي الاعتيادي Type I و السمنت البورتلندي المقاوم للأملاح Type V حيث كانت نسبة القلويات المثلى تقريبا متشابهة لكلا النوعين، بالنسبة إلى فحص زمن التجمد الابتدائي والنهائي للسمنت المقاوم للأملاح يقل بمقدار (6.8%) و (14%) على التوالي عن السمنت البورتلندي الاعتيادي وفحص الثبات للسمنت المقاوم للأملاح يقل بمقدار (11.1%) عن السمنت البورتلندي الاعتيادي و فحص مقاومة الانضغاط للسمنت المقاوم للأملاح يقل بمقدار (9% ، 7.5% و 3.6%) على التوالي عن السمنت البورتلندي الاعتيادي و تم استنباط المعادلات الرياضية باستعمال برنامج إحصائي (statistic software) لتخمين خواص السمنت الفيزيائية من خلال محتواة من القلويات.

الكلمات الرئيسية: القلويات، نسبة، السمنت البورتلندي، الفحوصات، الخواص الفيزيائية.