## استهلال

# قال تعالى:

# بسم الله الرحمن الرحيم

أشكُر َ نِعِم َ يَكُ التي أنع مَت عَلي وَ عَلي وَ الرِد َي وَ أَن أَعِم لَ صارِلحآ وَ أَشكُر وَ أَدخ لني بر حم يَ يك في ع باد ك الصال حين )

صدق الله العظيم سورة النمل الآية (19)

### **DEDICATION**

To the most wonderful parents in the world my parents for their endless love, support and encouragement

To my brother and sisters

To my friends

I am trying to say thank you

## **Acknowledgements**

First I thank Allah for giving me the strength to do this study.

I would like to express my immense gratitude and appreciation to my wonderful supervisor **Dr. Elmugdad AhmedAli**for his help, suggestions and close supervision throughout the study.

Finally all my thanks go to everyone who supported and helped me to accomplish this study.

#### **Abstract**

Cement free lime value generally indicates quality of raw materials, the completeness of the clinkering reaction and the quality of the burning practices hence the quality of the cement. A high free lime clinker is usually due to in-homogeneity and coarseness of cement raw mix or to improper burning and cooling condition in the kiln. Excess free lime results in undesirable effects such as volume expansion, increased setting time or reduced strength; hence it is critical to measure free lime content to ensure the quality of cement.

The aim of this research was to determine the percentage of free lime in different Sudanese cement samples (Berber cement and Atbara cement) by using methods that depend on extraction with ethylene glycol using different methods and also to study the effect of extraction temperature, extraction time and the concentration of hydrochloric acid on the results obtained by extraction with ethylene glycol.

The results obtained by all methods showed that the samples containing free lime percentage agreed with accepted values (not more than to 2% max according to ASTM). Also it is found that the percentage of free lime increased withincreasing extraction temperature (50, 70 and 100°C), extraction time (5, 10 and 20 minutes) and hydrochloric acid concentration (0.05, 0.1 and 0.2 M).

#### مستلخص البحث

تشير قيمة كمية الجير الحر في التحليل الكيميائي للأسمنت إلى جودة المواد الخام، إكتمال عملية المعالجة الحرارية في فرن الحرق، وجودة عمليات الحرق مما يضمن جودة النتج النهائي. تعود الزيادة في نسبة الجير الحر في عينات الكلنكر إلى عدم تجانس وعدم نعومة مخلوط المواد الخام أو الحرق الغير جيد أو ظروف التبريد السيئة في الفرن. ويؤدي الفائض من الجير الحر في الأسمنت إلى تمدد حجم الجسم الأسمنتي، زيادة في زمن التصلب وضعف في القوة وبذلك يكون قياس كمية الجير الحر مؤشر للجودة.

الهدف من هذا البحث تعيين نسبة الجير الحر في عينات مختلفة من الأسمنت السوداني (أسمنت بربر، أسمنت عطبرة) بطرق تعتمد على الإستخلاص بواسطة مذيب الإثيلين غلايكول وكذلك دراسة أثر حرارة الإستخلاص، زمن الإستخلاص وتركيز حمض الكلور على النتائج المتحصل عليها بالإستخلاص بمذيب الإثيلين غلايكول.

كانت النتائج المتحصل عليها بكل الطرق متفقة مع النسب المحددة في الطرق القياسية الأمريكية  $4 ext{ K}$  لاختبار المواد (في حدود  $2 ext{ M}$ ).

ووجد ان نسبه الجير الحر تزيد بزيادة حرارة الإستخلاص (50،70 و 100 درجة مئوية)، زمن الاستخلاص (5، 10 و 15 دقيقة) وتركيز حمض الكلور (0.05، 0.1 و 0.2 مولار).

### **Table of contents**

	Title	Page
	الآية	I
	Dedication	II
	Acknowledgement	III
	Abstract (English)	IV
	Abstract (Arabic)	V
	Table of contents	VI
	List of tables	VII
	List of figures	VIII
	List of abbreviation	IX
	Chapter one	
1	Introduction	1
1.1	Cement	1
1.1.1	Cement definition and history	1
1.1.2	Cement classification	1
1.2	Portland cement	3
1.2.1	Introduction	3
1.2.2	History of Portland cement	4
1.2.3	Different types of Portland cement	5
1.3	Chemical composition of Portland cement	7
1.3.1	Major constituents	7
1.3.2	Minor Constituents	11
1.4	Manufacture of Portland cement	13
1.4.1	Overview	13
1.4.2	Raw materials	14
1.4.3	Production process	15
1.5	Clinker compositional parameters	23
1.6	Quality control parameters	25
1.6.1	Physical properties	25

1 ( )		27
1.6.2	Chemical analysis 27 Cement setting and hardening 28	
1.7	Cement setting and hardening	
1.8	Free lime in cement	30
1.81	Sources of free lime	30
1.8.2	Effect of free lime in cement	
1.8.3	Determination of free lime in cement	32
	Chapter 2	
		<b>.</b>
2	Experimental and results	34
2.1	Equipments	34
2.2		2.4
2.2	Reagents	34
2.3	Materials	34
2.4	Standardization of HCl 0.05, 0.1 and 0.2 M	35
2.5	Free lime determination	36
2.5.1	Method (1)	36
2.5.2	Method (2)	37
2.5.3	Method (3)	39
2.6	Effect of extraction temperature	39
2.7	Effect of extraction time	40
2.8	Effect of concentration of HCl	41
	Chapter Three	
3	Discussion and conclusion	45
	References	47

### **List of Tables**

Table No	Title	page
Table 1:1	Approximate Oxide Composition Limits of Ordinary Portland cement	7
Table 1:2	Properties of the major constituents of Portland cements	10
Table 2:1	vol. of HCl 0.05 M	35
Table 2:2	vol. of HCl 0.1 M	35
Table 2:3	vol. of HCl 0.2 M	36
Table 2:4	free lime percentage calculated by method (1)	
Table 2:5	free lime percentage calculated by method (2)	38
Table 2:6	free lime percentage calculated by method (3)  39	
Table 2:7	effect of ethylene glycol temperature on CaO dissolution 40	
Table 2:8	CaO dissolution in ethylene glycol with time	40
Table 2:9	effect of HCl concentration	41
Table 3:1	Free lime percentage calculated by the three methods	44
Table 3:2	calculated free lime percentage at different temperature degrees	44
Table 3:3	calculated free lime percentage at different times	45
Table 3:4	free lime percentages calculated by using different concentration	46

## List of figures

Figure No	Title	Page
Figure 1.1	Flow chart for wet process of cement manufacturing	16
Figure 1.2	Flow chart for dry process of cement manufacturing	17
Figure 1.3	General layout of a rotary	19
Figure 1.4	Schematic outline of reactions in the typical dry process rotary cement kiln	22
Figure 1.5	A flow diagram of Portland cement production	23
Figure 1.6	Degree of hydration with time	30

### **List of Abbreviations**

A*	Aluminum oxide or alumina (Al <sub>2</sub> O <sub>3</sub> )
AR	Alumina Ratio
ASTM	American Standards for testing materials
C*	Calcium oxide or lime (CaO)
C <sub>2</sub> S*	Dicalcium silicate (2CaO•SiO <sub>2</sub> )
EDTA	ethylene diamine tetraacetic acid
IR	Insoluble residue
F*	Iron oxide or rust (Fe <sub>2</sub> O <sub>3</sub> )
LCF	Lime Combination Factor
LSF	Lime Saturation Factor
LOI	Loss on ignition
LHPC	Low-Heat Portland Cement
M*	Magnesium oxide, periclase or magnesia (MgO)
M.wt	molecular weight
M	Morality
OPC	Ordinary Portland cement
K*	Potassium oxide (K <sub>2</sub> O)
RHPC	Rapid Hardening Portland cement
SR	Silica Ratio
S*	Silicon dioxide or silica(SiO <sub>2</sub> )
N*	Sodium oxide (Na <sub>2</sub> O)
S	Sulfur trioxide or sulfuric anhydride(SO <sub>3</sub> )

SRPC	Sulfate Resisting Portland cement
C <sub>4</sub> AF*	Tetracalcium aluminoferrite(4CaO•Al <sub>2</sub> O <sub>3</sub> •FeO <sub>3</sub> )
C <sub>3</sub> A*	Tricalcium aluminate(3CaO•Al <sub>2</sub> O <sub>3</sub> )
C <sub>3</sub> S*	Tricalcium silicate (3CaO•SiO <sub>2</sub> )
V	Volume
W	weight
W-	weight of sample
XRD	X-ray diffraction
XRF	X-ray fluorescence
Mpa	$1 \text{Nmm}^{-1} = 10 \text{ bar} = 9.87 \text{atm} = 7500 \text{ tor} =$
	$145.0 \text{ Ib in}^{-2} = 10.198 \text{ kg cm}^{-2}$

## \* Cement notation