# Sudan University of Science and Technology

# College of Graduate Studies

# The Use of Steel Slag in Manufacturing of Portland cement

# استخدام خبث الحديد في انتاج الاسمنت البورتلاندي

**A Thesis Submitted in Partial Fulfillment of the requirement for the Degree of Master of Chemical Engineering**

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# Abstract

The main objective of this research is to determine the effectiveness of local Electric Arc Furnace Slag (EAFS) as a replacement material in cement mortar and to determine the degree of its pozzolanicity.

The properties of steel slag blended cement have been studied at chemical and physical laboratories of Nile cement Industry in accordance with the international standards (ASTM and BS). Chemical analysis shows that locally produced steel slag contains a high amount of iron due to a purification process in steel manufacturing compared with the standard (ACI 233).

In studying the performance of concretes, two concrete mixes have been prepared for class C-50 with cement blended with steel slag in separate mixes with 5% replacement. Different test results together with the literature review were used to analyze the effects of using steel slag in cement and concrete production.

Compressive strength test of mortar samples at 2, 7 and 28 days with different percentages of electric arc furnace slag (0%, 5%, 10%, 15%, and 20%) had been conducted

The investigation of this thesis has revealed that compressive strengths of cement pastes from 5% steel slag blended Portland cement is comparable with the control one, that is 100% ordinary Portland cement. For 10, 15 and 20% replacement ranges, through the reduction in compressive strength is observed, blended types of cement at these percents satisfy the standard compressive strength limits for high early strength of class of 42.5 MPa as per the EN 197-1 standard. According to the test results, it was found that the degree of pozzolanicity is 95.8 % during 28 days which point out to a good quality electric arc furnace slag and a promising pozzolanic supplementary material in concrete.

Observation on the test results also indicated that the effects of blending steel slag on the properties of cement such as consistency, setting times, insoluble residue, and soundness remain within the acceptable ranges of many standards.

The investigation also indicated that the replacement of cement steel slag at 5% range, in concrete production, results in comparable compressive strength as of concrete specimens without steel slag with slight slump reduction.

Finally, the study proved that using the local steel slag as a pozzolanic material is very efficient in the cement industry.

# المستخلص

الهدف الرئيسي من هذا البحث هو تحديد فعالية خبث افران القوس الكهربائي المحلي عند استخدامه كمواد بديلة في صناعة الاسمنت وكذلك تحديد درجة البوزلانية له.

تمت دراسة خواص عينات الأسمنت المنتج من خبث الصلب في المختبرات الكيميائية والفيزيائية لمصنع أسمنت النيل وفقاً للمعايير الدولية . يبين التحليل الكيميائي أن خبث الصلب المنتج محليًا يحتوي على كمية عالية من اكسيد الحديد ويعود ذلك لعملية تنقية في تصنيع الصلب.

في دراسة أداء الخرسانات ، تم إعداد عينتين من الخرسانة من النوع C-50 مع خلط الأسمنت بخبث الحديد في خلطات منفصلة باحلال الخبث بنسبة 5٪.

تم إجراء اختبار مقاومة الانضغاط لعينات المونة الاسمنتية في أيام (2 , 7 , 28 ) بنسب مختلفة من خبث فرن القوس الكهربائي (0٪ , 5٪ , 10٪ , 15٪ , 20٪. وقد أوضحت نتائج هذه الدراسة أن مقاومة الضغط لعينات الأسمنت المخلوط مع خبث الحديد بنسبة 5٪ متقاربة جدا عند مقارنتها مع عنصر التحكم ، وهو الاسمنت البورتلاندي العادي 0٪ . اما بالنسبة لمجالات الاستبدال 10 ،15 ،20٪ ، لوحظ أن مقاومة الضغط للأسمنت المخلوط عند هذه النسب المئوية يقل تدريجيا عن عينة الاسمنت البورتلاندي العادي الا انه ايضا يفوق حدود مقاومة الانضغاط القياسية لقوة عالية مبكرة من فئة 42.5 ميجا باسكال.

وفقا لنتائج الاختبار ، فقد وجد أن درجة البوزلانية هي 95.8 ٪ خلال 28 يوما ، مما يشير إلى ان خبث فرن القوس الكهربائي المستخدم في الدراسة من النوعية الجيدة ويتحوي على المواد التكميلية البوزلانية الموعودة في الخرسانة.

كما أشارت نتائج الدراسة إلى أن آثار خلط خبث الحديد على خواص الأسمنت مثل :كثافة العجينة الاسمنتية زمن التصلب ،البقايا غير القابلة للذوبان و الصلادة تظل ضمن النطاقات المقبولة للعديد من المعايير المحلية والعالمية. كما اتضح أن استخدام الاسمنت المخلوط بالخبث في إنتاج الخرسانة ، ينتج عنه قوة ضغط مماثلة لنتائج العينات الخرسانية بدون خبث الحديد.

وأخيراً أثبتت الدراسة أن استخدام خبث الحديد المحلي كمواد بزولانية فعال جداً في صناعة الأسمنت والخرسانة .

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## Symbols for variables

| | |
|---|---|
| B | Blain value |
| $B_F$ | Blain factor |
| $d_1$ | Different in distance before boiling |
| $d_2$ | Different in distance after boiling |
| $m_1$ | Mass of material passing 1.18 mm sieve |
| $m_2$ | Mass of material passing 75 µm sieve |
| S | Silt content |
| SA | Strength activity index |
| $S_c$ | Strength of control sample at 28 Days |
| $S_r$ | Strength of 5% replacement at 28 Days |
| t | Average time |
| $T_F$ | Temperature factor |

## Symbols for units

| | |
|---|---|
| atm | atmosphere |
| $^oC$ | Centigrade |
| $^oF$ | Fahrenheit |
| g | Gram |
| N | Newton |
| µc | Micrometer |

## Acronyms and abbreviations

| | |
|---|---|
| ACI | American Concrete Institute |
| ASTM | American Society for Testing Materials |
| BOF | Basic Oxygen Furnace |
| BS | British Standard |
| CN0 | Control concrete sample prepared without steel slag. |
| CN5 | Concrete sample with cement blended with 5% steel slag |
| EAF | Electric Arc Furnace |
| EN | Euro Norm |
| Fig | Figure |
| LF | Ladle furnace |
| NOPC | Control cement prepared by Nile OPC without steel slag |
| NS05 | Nile OPC cement blended with 5% steel slag |
| NS10 | Nile OPC cement blended with 10% steel slag |
| NS15 | Nile OPC cement blended with 15% steel slag |
| NS20 | Nile OPC cement blended with 20% steel slag. |
| OHF | Open Hearth Furnace |
| OPC | Ordinary Portland cement |
| XRD | X Ray Diffraction |

## Chemicals and materials

| | |
|---|---|
| $Al_2O_3$ | Aluminum Oxide |
| $C_3A$ | Tricalcium Aluminate |

| | |
|---|---|
| $C_4AF$ | Tetra calcium Alumni Ferrite |
| $C_2S$ | Dicalcium Silicate |
| $C_3S$ | Tricalcium Silicate |
| $CaCO_3$ | Calcium Carbonate |
| $CaO$ | Calcium Oxide |
| $Ca(OH)_2$ | Calcium Hydroxide |
| $CO_2$ | Carbon Dioxide |
| $Fe_2O_3$ | Iron Oxide |
| FL | Free lime |
| LOI | Loss on ignition |
| $MgO$ | Magnesium Oxide |
| $MnO$ | Manganese Oxide |
| $SiO_2$ | Silicon dioxide |
| $SO_3$ | Sulfur trioxide |

# Chapter One

# Introduction

## 1.1 Introduction

In recent years in Sudan, different industrial plants had started mass manufacturing and mass production of structural steel such as steel reinforcement, steel beams and other structural steel sections. Some of these plants use scrap materials to manufacture steel products. So, electric arc furnace slag (EAFS) is produced as by-product of steel melting process. And this locally produced EAFS is considered as an environmental hazard and it could be investigated for the potential of been used as an artificial pozzolana or an aggregate material in the construction industry.

## 1.2 General

The protection of the environment from pollution is a key to the extent of urbanization and the progress of any nation indicators, the state that achieve a balance between raising the industrial development rates that produce many pollutants and the protection of the environment from pollution at the same time is considered to have succeeded in applying the slogan advocated by the United Nations, a technology clean production, began the developed world organizations and international companies in recent years, serious attempts to reduce the negative impact of the industry on the environment, including secreted from pollutants and solid and gaseous and liquid waste.

The golden rule in the field of preservation of the environment from the risk of waste is to reduce waste, and the most important step in that reduced somewhere origin before that produces. Before farewell to address them or to retrieve it, and reduce waste will be based on the awareness of preserving the environment to rationalize the use of resources arises optimal use of natural, which takes into account the principles of preservation of the environment, or that the use of such waste in other industries and is the most important action in the absence of the possibility of preventing the emergence of this waste completely.

Solid wastes are known as solid and semi-solid non-hazardous materials which generated from residential, commercial and industrial areas. It must be collected and carried forward and treated and disposed as useless as well as they cause pollution of the environment in which they arise, and with the large increase in the industry globally and locally increases the challenge to deal with industrial waste from these industries are a problem, and on the grounds that the mining industry, which has grown is growing rapidly in the twentieth century, one of the most damaging industries environment first for two reasons is what exhaust of the natural resources of raw materials, fuel, etc., and the second is produced from remnants of harmful gaseous and loaded with solid plankton that no less reciprocating, in addition to the failure of the liquid or solid waste in very large quantities to be a major cause of pollution of surface water or groundwater in places assembled or disposed of environmentally healthy.

## 1.3 Problem Statement

Topic selection was based on the problem recently facing Rabak cement factory, and it’s a lack of sufficient quantities of Pozzolana (from natural or industrial resources). So searches were done to find an alternative option that is similar in terms of chemical and physical composition and properties, in addition to be available and inexpensive in production, processing and deportation.

On the other hand, there are huge quantities of steel slag resulting from scrap smelting operations as by-products with polluting materials that has to be processed, recycled and utilized in other industries.

## 1.4 Objectives of Research

The main objectives of this thesis are:

1) To study the possibility of using of steel slag resulting from the steel industry as an additive material for the manufacture of Portland cement and its effect on the physical and chemical properties of Portland cement paste and hardened mortar.
2) To study the effect of percentage replacement of cement by steel slag on different properties of concrete.

## 1.5 Importance of Research

The importance of research lies through the gains achieved which including the environmental and economic terms, as shown below:

**First: Environmental Terms:**

1. The safe disposal of accumulated amounts of slag as a by-product (waste) of the Iron Smelting Industry.
2. Work to stop the arrival of slag components to the soil surrounding the landfill and prevent pollution.
3. Contribute to the reduction of carbon dioxide emissions from the cement industry.
4. Preserve the natural Pozzolana quarries.

**Second: Economical Terms:**

1. Provide an important product (ordinary Portland cement) at a lower cost as slag good additive.
2. Provide a new product (compound cement specifications as resistant sulphate cement) which is a win-win situation for additional cement factory.
3. Provide the cost of dealing with contaminated soil and water.
4. Provide slag processing costs, where the slag commodity has a price in the developed countries and not waste constitutes a burden on the environment as it is to have.

## 1.6 Structure of the Research

The thesis has six chapters that discuss various aspects of cement and steel slag related with relevance of the thesis. Chapter one explains the background and the objectives of the research. Chapter two is literature review which provides a general understanding of previous studies and theories related to the research.

Chapter three discusses the properties of materials used in the investigation, Chapter four deals with the experimental program that was used in the research, Chapter five is about the analysis and discussion of the results obtained from the study, and the last chapter draws conclusions from the research and provides recommendations. Other data are presented in the Appendices.

# Chapter Two

# Literature Review

## 2.1 Introduction

The use of such industrial by-product or waste material having desirable qualities can result in saving of energy and conventional materials. With increase in population, the demand for construction of residential and public buildings is also increasing (Rojas and Rojas, 2004). The iron and steel industry produces extremely large amounts of slag as by-product of the iron making and steelmaking processes . As useful recycled materials, iron and steel making slag are mainly used in fields related to civil engineering, for example, in cement, roadbed material, and concrete aggregate (Leung and Wong, 2010).

The American Society for Testing and Materials (ASTM) defines Steel Slag as a non-metallic product, consisting essentially of calcium silicates and ferrites combined with fused oxides of iron, aluminum, manganese, calcium and magnesium that are developed simultaneously with steel in basic oxygen, electric arc, or open-hearth furnaces. Steel slag can be produced by a variety of processes that include Open-Hearth Furnace (OHF), Basic Oxygen Furnace (BOF), and an Electric Arc Furnace (EAF). Due to the amount of time the OHF takes to produce the steel, most of them have been closed since the early 1990's and have been replaced by BOF and EAF processes.

Electric arc furnace slag is produced during the manufacture of crude steel by the electric arc furnace (EAF) process. In this process steel scrap with additions of fluxes (e.g., limestone and/or dolomite) are heated to a liquid state by means of an electric current. During the melting process, the fluxes combine with nonmetallic scrap components and steel incompatible elements to form the liquid slag

(Nwaubani and Muntasser, 2012). As the slag has a lower density than steel, it floats on top of the molten bath of steel. The liquid slag is tapped at temperatures around 1600°C and allowed to slowly air-cool forming crystalline slag (Ansu and Elson, 2013). Blast furnace slag is formed with the molten steel by the melting of iron ore or pellets with lime or dolomite, coke or fly ash. The fluxing agent such as lime combines with the silicates and other elements, which results in the production of the slag. The slag not only helps to remove impurities from the smelted steel, but it also helps to protect the lining of the furnace. There are three types of blast furnace slag that can be produced: air cooled, expanded, and granulated slag. Steel slag is produced in a similar manner to blast furnace slag and can be separated into categories depending on the process used to form the steel (Netinger, et al, 2011).

Since the difference between electric arc furnace slag and blast furnace slag is in the technology of production rather than the chemical composition and behavior in mortar mixes, same international standards are used for both types. According to ASTM C989, Slag is classified into three grades according to its performance in the “slag activity test”. The three grades are Grade 80, Grade 100 and Grade 120 (Tsakiridis, et al, 2008).

The basic components of Slag comprise generally CaO (30-48%), MgO (28-45%), $Al_2O_3$ (5-18%), and $SiO_2$ (1-18%), which are in principle the same as that of Portland cement. Other minor components including $Fe_2O_3$, MnO, and $SO_3$ are also present in slag. The compositions do not change very much so long as the sources of iron ore, coke and flux are consistent (Lacobescua, et al, 2011). The high cost of slag disposal besides their negative impact on the environment and the lack of natural aggregate resources in many regions led the utilization of steel slag in various applications. There are also a few types of researches that have been performed regarding the utilization of steel slag in concrete (Wanga, et al, 2011).

In Sudan, many industrial plants consume scrap raw material to manufacture of reinforced concrete, steel beams and other structural steel sections. These plants annually produce huge tones of electric arc furnace steel slag. This locally produced (EAFS) has been an environmental hazard and it could be investigated for the potential used as an artificial pozzolanic or aggregate material in the construction Industry.

This chapter reviews the work done by previous investigators regarding the manufacture of iron and steel slag and their properties.

## 2.2 Steel Slag

After the iron ore is smelted in the blast furnace, the pig iron that is produced from the process can then is used in a second process to produce steel. It is this second process that produces the steel slag. Steel slag is formed as a fluxing agent like lime or dolomite reacts with the molten iron ore or molten scrap metal. The fluxing agent reacts with the molten steel and the impurities from the mixture form the slag. After removing the entrained metals from the slag, the contents of the slag consist mainly of oxides and silicates (Tufan, 2014).

### 2.2.1 Basic oxygen furnace slag

Basic oxygen furnace (BOF) slag is produced when the hot molten iron from the blast furnace process is combined with scrap metals and a fluxing agent like lime or dolomite. Oxygen is then blown into the chamber and through the molten iron, which lowers the carbon content. This results in a reaction that produces a high quality steel product and the by-product steel slag. The BOF process was developed in 1948 by Robert Durrer, but was not commercialized until 1952. Basic oxygen furnace slag can also be referred to as converter slag.

### 2.2.2 Electric arc furnace slag

An electric arc furnace uses cold steel or scrap steel to produce a new alloy steel from the recycled steel. The scrap steel is placed into the furnace and then the graphite electrodes are lowered into place. The contents placed in the furnace have to be carefully monitored in order to control the chemical composition of the mixture (Mahmoud, 2012).

Chemical elements are added to the mixture to help remove impurities from the molten steel. The amount of chemicals added to the mixture will vary from furnace to furnace. Some of the elements that are added to the mixture are magnesium, aluminum, and silicon. An electrical current is then passed through the electrodes, and an arc is produced, which melts the steel scrap. Oxygen is then blown in to the furnace in order to produce a low carbon steel. Once the process is complete, the molten steel can be used to cast new steel or it can be poured into a separate ladle for further processing. The impurities that have been removed from the molten steel form the by-product, which is known as steel slag. Electric arc furnace slag can also be known as black slag (due to its color), acid slag, and oxidizing slag (Fronk, 2012).

### 2.2.3 Secondary slag

Secondary slag are produced from the further refining of the EAF process in which the molten steel is poured from the EAF to a ladle and the steel is processed again. This secondary refining process can produce special high quality steels such as stainless steel. This process leads to Argon Oxygen Decarburization slag. Argon Oxygen Decarburization is a process that is used to refine the molten steel to produce stainless steel. Prior to its use the amount of chromium in the steel needed to be increased after the steel had been decarburized, since the decarburization caused the oxidation of the chromium.

The decarburized process is accomplished by subsurface blowing with the Argon / Oxygen gas mixture, which allows the steel to be decarburized without the oxidation of the chromium .Ladle furnace slag break down very rapidly after being exposed to the outdoors and weather, usually within a week, unless the Argon Oxygen Decarburization process is used in the processing of the steel.

The breaking down of LF slag is called slaking and is due to the expansion of the free lime and Pericles, which react with the moisture and water to produce hydroxides. The volume of the slag essentially doubles and turns to dust that can be blown away by the wind [16].

## 2.3 Current uses and applications of steel slag

While the use of slag dates back many centuries, the slag industry continues to find new applications. At one time slag was hauled away as a waste material and deposited in a landfill. In more recent years, steel slag has found use as an aggregate in asphalt pavements, in the production of cement, agricultural uses, railroad ballast and as an aggregate as a base or sub base.

### 2.3.1 Bituminous / Asphalt paving

The use of steel slag in bituminous pavements has been well documented. EAF slag sand was used in the Illinois Tollway in 1994. This pavement mixture termed F-4 consisted of 36% slag sand, which provided a high skid resistance that is needed near the toll plazas. EAF slag was also used in the surface layer of I-94 in which the asphalt layer consisted of 85% slag (Fronk and Brad, 2012).

EAF slag was also used in the asphalt pavement in the construction of Colorado's Glenwood Canyon corridor on 1-70. The slag provided the durability needed for this stretch of road and when used in a 25% by weight mixture it provided a 20% increase in stability of the pavement.

### 2.3.2 Manufacture of Portland cement

In 1999, Texas Industries, Inc. and Chaparral Steel introduced a new process of adding chunks of steel slag to the kiln for the production of cement. This process was named the Cemstar process. Portland cement clinker is similar to steel slag in its composition, which in turn means that the fuel cost to convert it to cement is low.

The addition of the steel slag to the process of manufacturing cement has shown an increase in production of 15% without increasing the emissions from the manufacturing process. This not only saves money in fuel cost, but also reduces the amount of $CO_2$ produced from the processing. The emissions of $CO_2$ have been reduced by 5 to 10% per ton of $CO_2$ per ton of clinker. The addition of the steel slag has also reduced the emissions of $NO_x$ by 25 to 45% on a pound per short ton of clinker.

### 2.3.3 Agriculture

According to the National Slag Association, steel slag has been used in agriculture as a liming agent for over a hundred years. It has been used as an agricultural amendment in Alabama, Illinois, Indiana, Kentucky, Maryland, Ohio, Pennsylvania, and West Virginia. The chemical composition of the steel slag allows the slag to react with the soil and this reaction can reduce the pH of the soil (Anderson, 1984). The steel slag is used as a fertilizer in soils for corn, rice, and soybeans. Some beneficial chemical components of the slag that can add valuable nutrients to the soil for plant life include Fe, P, S, Mn, Mo, and amounts of calcium silicate. The calcium in the slag reacts with the water and forms $Ca(OH)_2$, which then reacts with the soils acidity rapidly. The silicates in the slag will provide a long term buffering of the soil for the pH since it will react more slowly (Teresa, et al, 2012).

### 2.3.4 Other applications

Steel slag has been used in applications for controlling erosion on the banks of lakes and rivers. It has also been used to adjust the pH of water coming from mines. Steel slag has even been used in the construction of reefs and sea walls. Steel slag has also been used as railroad ballast. Its higher density allows for more resistance to movement on the lateral directions on turns in the railroad and its higher density also makes it less susceptible to being washed away. It is used in this application not only because it has a high density, but also because it has a low electrical conductivity.

Steel slag is also used as an aggregate for roadway bases and sub bases. Steel slag can be found as a fill for landowners that need a stable and dense material to fill in low and uneven lands.

One final application is by a company called Roxul, which is using the steel slag and a basalt rock to make mineral wool insulation for systems furniture panels. This material is reported to be able to withstand temperatures up to 21500 F ($1177^0$C).

## 2.4 Chemical composition and properties of steel slag

The chemicals and minerals found in the steel slag are leftover components from the production of the steel. The slag produced from the production of the steel contains the impurities that have been removed from the molten steel. The actual chemical compounds found in the slag are dependent on the type of steel production plant and the type of flux used in the process. For example, a ladle slag will have different ranges of chemical composition than that of a slag from an EAF steel plant, which will have different ranges in composition from a BOF plant.

The chemical properties of steel slag produced at different plants using the same process, such as an EAF will probably have different chemical compositions from one another. This is because the scrap metal being refined is different and the types and amounts of fluxing agents will be different. The chemical composition of the steel slag at any particular plant will not usually have a large variation from day to day or year to year (Pacheco, et al, 2006).

### 2.4.1 Determining the chemical and mineralogical composition

The chemical and mineralogical analysis of steel slag can be accomplished by the following methods: X-ray fluorescence method, X-ray powder diffraction, Electron probe micro analysis and X-ray diffraction method (XRD). The chemical composition of the steel slag is provided in many studies. The methods for determining the chemical and mineralogical composition of the slag were not always stated. The most common analysis used in the studies was XRD, and for this reason it will be described in the following section. The method for determining the content of free lime by the ethylene glycol method is also discussed.

#### 2.4.1.1 X-Ray diffraction testing:

XRD is used in order to determine the chemical and mineralogical composition of a rock, soil or slag aggregate. X-ray diffraction is the most widely used method for the determination of fine-grained soil minerals and the study of their crystalline structure.

As the X-rays penetrate the crystal, small portions of them are absorbed by the atoms, which cause them to oscillate. This results in some waves that are in phase, which can then be detected as waves that have resulted from a reflection of the incident beam. The direction of the parallel planes of the crystal structure, which are relative to the direction of the incident beam, at which the radiations are in phase depends on the wavelength of the X-rays and the spacing between atomic planes (Mitchell, et al, 2005).Since no two minerals have the same spacing of the inter atomic planes in three dimensions, this method can be used to identify the mineral. This particular method cannot detect the presence of the crystalline CaO but it can detect the presence of hydrated lime, which can show that free lime was present in the material prior to treatment.

The presence of MgO in steel slag can be detected by X-ray diffraction using the main reflection peaks located at 78°, 62.3°, and 42.91°. The most important peak of $2\theta$ is located at 78°, and if this peak is not present, then MgO content is less than 20% and is difficult to differentiate from the baseline. The presence of some minerals in the steel slag can also make it difficult to detect MgO because minerals such as Larnite overlap and hide the reflection peaks of MgO (Alexander, 2015).

#### 2.4.1.2 Ethylene glycol test

This test is generally used to determine the types of minerals that are contained within a sample. The ethylene glycol and glycerol is applied to the mineral, and some of it is absorbed on the surface of the mineral. The amount of glycol retained can be used to determine the type of clay mineral, since different clay minerals have different specific surface areas (idem).

The ethylene glycol test is currently being used to determine the presence of free lime in steel slag. In Germany, samples of the molten steel slag are taken while it is still in the converter (Motz, et al, 2001). Once the slag has been analyzed, the slag is then poured into different slag pits depending on the content of the free lime.

One of the reasons the sample is taken at this point in the processing of the steel slag is that the ethylene glycol test does not differentiate between hydrated lime (Calcium Hydroxide) and free lime. Once the steel slag has been aged and allowed to come into contact with moisture, additional testing is required on the slag to find the free lime content.

The slag now needs to be tested by thermo-gravimetric analysis, which is able to find the amount of $Ca(OH)_2$ by the weight loss of the sample at a temperature of 842 to 1022 °F (450 to 550 °C).

## 2.5 Treatment options: Techniques for the stabilization of steel slag

The expansion is an important aspect to the possible use of steel slag in cement and concrete, and so is the proper treatment of the slag to eliminate the expansion. The aging of steel slag by stockpiles outdoors and spraying has been used by the slag industry for some time now and is generally accepted for reducing the expansion.

The treatment of the steel slag prior to cooling is also a possibility. This accomplished by the addition of silica sand and the blowing of oxygen into the slag while it is still in its molten state. Some other new methods that have recently been developed are quick quenching of the slag, steam aging, and autoclave aging of the slag. One final possible treatment of the slag is to chemically treat the slag to remove the chemical components that are responsible for the expansion (idem).

### 2.5.1 Aging by spraying with water:

The process of cooling the slag outside in areas that are covered or not covered has been used by slag companies for treating the slag aggregate since sometime in the early 1980’s. The slag is brought outside in the slag pot and then it is poured into a slag pit and allowed to cool. Depending on the company and the intended use of the slag, the slag pits can be sprayed with water.

The spraying of water initially helps to cool the hot molten slag faster and help to fracture the slag as it is cooled. This type of cooling is considered a slow cooling process. Once the slag has cooled completely, the continued spraying of the slag with water accelerates the aging process of the slag by the hydration of the free lime and Pericles. The slag remains outdoors, exposed to the natural elements, and is occasionally turned over to insure all the slag is aged properly. The slag should remain outside for a period of 90 days prior to being used as an aggregate in unbound applications such as a road base.

According to the National slag Association, tests have indicated that aging in stockpiles after the processing and the use of coarser aggregate sizes tends to limit the potential expansion of the steel slag. It is also noted that aging the slag in large piles or pieces is not very effective, since the steel slag needs to be directly exposed to the weathering or it can remain expansive for a long period of time. The frequency of the overturning of the piles of slag is not discussed.

### 2.5.2 The Addition of silica sand combined with oxygen blowing

This process takes place in a separate slag ladle so the process does not affect the quality of the steel. The molten slag is poured into a separate ladle where dry sand and oxygen are injected into the molten slag. The $CaO$ / $SiO_2$ ratio is reduced, and the free lime is chemically bound and thus the free lime content is reduced.

This process can also be used to chemically bind the free magnesium as well. The addition of the siliceous material into the molten slag reduces the free MgO content by the hydration of the Pericles during the high hydrothermal condition, which then reduces the expansion by reducing the amount of free MgO content. The free MgO content of the slag produced from this process is not given but it was stated that the reaction works the same for the free MgO.

### 2.5.3 Quick quenching methods

The instant chilled slag process is comprised of four basic stages. The first stages consist of the cooling of the molten slag on shallow plates, which results in a thickness of the slag of approximately 4 inches (100 mm). The molten slag is cooled for approximately 4 minutes before it moves on to the next stage.

The second stage of the cooling process consists of continuously spraying the slag with water for approximately 20 minutes until a temperature of 932°F (500°C) is reached. The slag is then transported to the 3rd cooling stage, which sprays the slag for an additional 4 minutes and cools the slag to a temperature of 392°F (200°C). The slag is finally placed into a pool of water and cooled to a temperature of 140 °F (60°C) and the slag is now ready for further processing such as magnetic screening. This process of treating and cooling the slag produces slag with a free lime content ranging from 2 – 4%. No Pericles exists in the slag and the particle sizes of the slag ranges from 1.2 to 2 inches (30–50 mm) (Motgomery, et al, 1991).

Another process of rapidly cooling the slag is by quickly quenching the molten slag in water. This is accomplished by allowing the liquid slag to leak out from the converter into a slag pot that contains water, which produces a granulated slag. This process of granulating the slag produces a slag with a lower free CaO content then if the slag was cooled by air. The steel slag used in this study for cooling the slag rapidly had a CaO of 40.15%, MgO content of 10.56%, and free lime content of 1.4%. There was no detection of free MgO. There was no expansion testing done in the study (Gumieri, et al, 2004).

### 2.5.4 Other treatment processes

Some other treatment processes using some form of water that are mentioned in different references are not described in detail. This may be because they are relatively new and the processes are still being perfected, or it might be because these processes are patented and proprietary to a certain company. One of these methods is the hot water aging process. Hot water aging is accomplished by submerging the steel slag aggregate into hot water at a temperature of 176 ± 37 °F (80 ± 3 °C) for 1 to 3 days. Another aging process is the aging or processing of the slag accomplished by exposing the aggregate to 212 °F (100 °C) steam for a period of over 3 days under 1 atm of pressure. Treatment of the steel slag in an autoclave device has also been mentioned in literature, but the details were not discussed. Aging the slag by steam at 212 °F (100 °C) is accomplished without pressure, and experimental processing times range from 3 hours to days in steam is another example. A final method of steam processing covers the slag with large tents and steams the slag in the tents for a period of a few days (Moon, et al, 2002).

## 2.6 Portland cement

Portland cement was developed from natural cements made in Britain in the early part of the nineteenth century, and its name is derived from its similarity to Portland stone, a type of building stone that was quarried on the Isle of Portland in Dorset, England. It is the chief ingredient in cement paste and the binding agent in Portland cement concrete. It is a hydraulic cement that, when combined with water, hardens into a solid mass. Interspersed in an aggregate matrix it forms Portland cement concrete. As a material, Portland cement has been used for well over 175 years and, from an empirical perspective, its behavior is well understood (Hakan, 2006).

The patent for Portland cement was obtained in 1824 by Joseph Aspdin. Chemically, however, Portland cement is a complex substance whose mechanisms and interactions have yet to be fully defined.

The Portland Cement Association provides the following precise definitions: Hydraulic cement: Hydraulic binder, i.e. a finely ground inorganic material, which, when mixed with water, forms a paste which sets and hardens by means of hydration reactions and processes and which, after hardening, retains its strength and stability even under water. Portland cement: Hydraulic cement composed primarily of hydraulic calcium silicates.

As the use of Portland cement was increased for making concrete, engineers called for consistently higher standard material for use in major works. Association of Engineers, Consumers and Cement manufacturers has been established to specify standards of cement. The German standard specification for Portland cement was drawn in 1877.The British standard specification was first drawn up in 1904.The ASTM specification was issued in 1904 (Steven and Michelle, 2001).

### 2.6.1 Manufacturing process of Portland cement

Portland cement is produced by grinding cement clinker in association with gypsum to specified fineness depending on the requirements of the cement consumers. Cement clinker is produced on large scale by heating finely ground raw materials (Calcareous and Argillaceous materials) at very high temperature up to 1450 $^{o}$C in rotary kilns.

Raw mixture preparation and raw mix blending, formation and grinding of clinker are the fundamental stages in the production of Portland cement.

#### 2.6.1.1 Raw mix preparation and blending

The raw materials for Portland cement production are a mixture (as fine powder in the 'dry process' or in the form of slurry in the 'wet process') of minerals containing calcium oxide, silicon oxide, aluminum oxide, ferric oxide and magnesium oxide. The raw materials are usually quarried from local rock, which in some places has already practically the desired composition and in other places requires the addition of clay and limestone, as well as iron ore, bauxite or recycled materials. The individual raw materials are first crushed, typically to below 50 mm. In many plants, some or all of the raw materials are then roughly blended in a "pre-homogenization pile". The raw materials are next ground together in a raw mill. Silos of individual raw materials are arranged over the feed conveyor belt.

Accurately controlled proportions of each material are delivered onto the belt by weigh feeders. Passing into the raw mill, the mixture is ground to raw mix. The fineness of raw mix is specified in terms of the size of the largest particles, and is usually controlled so that there are less than 5-15% by mass of particles exceeding 90 μm in diameter. It is important that the raw mix contains no large particles in order to complete the chemical reactions in the kiln, and to ensure the mix is chemically homogenous. In the case of a dry process, the raw mill also dries the raw materials, usually by passing hot exhaust gases from the kiln through the mill, so that the raw mix emerges as a fine powder. This is conveyed to the blending system by conveyor belt or by a powder pump. In the case of wet process, water is added to the raw mill feed, and the mill product is slurry with moisture content of 25-45% by mass. This slurry is conveyed to the blending system by conventional liquid pumps (Hakan, 2006).

The raw mix is formulated to a very tight chemical specification. Typically, the content of individual components in the raw mix must be controlled within 0.1% or better. Calcium and silicon are present in order to form the strength producing calcium silicates. Aluminum and iron are used in order to produce liquid ("flux") in the kiln burning zone. The liquid acts as a solvent for the silicate forming reactions, and allows these to occur at an economically low temperature. Insufficient aluminum and iron lead to difficult burning of the clinker, while excessive amounts lead to low strength due to dilution of the silicates by aluminates and ferrites.

Very small changes in calcium content lead to large changes in the ratio of alite to belite in the clinker, and to corresponding changes in the cement's strength growth characteristics. The relative amounts of each oxide are therefore kept constant in order to maintain steady conditions in the kiln, and to maintain constant product properties. In practice, the raw mix is controlled by frequent chemical analysis (hourly by X-Ray fluorescence analysis, or every three minutes by prompt gamma neutron activation analysis). The analysis data is used to make automatic adjustments to raw material feed rates. Remaining chemical variation is minimized by passing the raw mix through a blending system that homogenizes up to a day's supply of raw mix (idem).

### 2.6.1.2 Formation and grinding of clinker

The raw mixture is heated in a cement kiln, with temperatures increasing over the length of the cylinder up to a peak temperature of 1400-1450 °C. A complex succession of chemical reactions take place as the temperature rises. The peak temperature is regulated so that the product contains sintered but not fused lumps. Sintering consists of the melting of 25-30% of the mass of the material. The resulting liquid draws the remaining solid particles together by surface tension, and acts as a solvent for the final chemical reaction in which alite is formed. Too low a temperature causes insufficient sintering and incomplete reaction, but too high a temperature results in a molten mass or glass, destruction of the kiln lining, and waste of fuel. When all goes to plan, the resulting material is clinker.

On cooling, it is conveyed to storage. Some effort is usually made to blend the clinker, because although the chemistry of the raw mix may have been tightly controlled, the kiln process potentially introduces new sources of chemical variability. The clinker can be stored for a number of years before use. Prolonged exposure to water decreases the reactivity of cement produced from weathered clinker (Steven and Michelle, 2001).

In order to achieve the desired setting qualities in the finished product, a quantity (2-8%, but typically 5%) of calcium sulfate (usually gypsum or anhydrite) is added to the clinker and the mixture is finely ground to form the finished cement powder. This is achieved in a cement mill. The grinding process is controlled to obtain a powder with a broad particle size range, in which typically 15% by mass consists of particles below 5 μm diameter, and 5% of particles above 45 μm. The measure of fineness usually used is the "specific surface", which is the total particle surface area of a unit mass of cement. The rate of initial reaction (up to 24 hours) of the cement on addition of water is directly proportional to the specific surface. Typical values are 320-380 $m^2$/kg for general purpose cements, and 450-650 $m^2$/kg for "rapid hardening" cements.

### 2.6.1.3 Environmental concerns in cement production

Many of the aspects of cement making process are potentially environmentally damaging, although these risks can be minimized. Cement manufacturing is an energy intensive process. The enthalpy of formation of clinker from calcium carbonate and clay minerals is about 1500 to 1700 kJ/kg. However, because of heat loss during production, actual values can be much higher. The high energy requirements and the release of significant amounts of carbon dioxide make cement production a concern for global warming.

Carbon dioxide is produced during the calcinations phase of the manufacturing process and also as a result of burning fossil fuels. Opportunity to reduce emissions through increased energy efficiency is only possible on the latter of the $CO_2$ emissions. Approximately **1** ton $CO_2$ is generated for making **1** ton of clinker (Hakan, 2006).

### 2.6.2 Types and standards of cement

There are different standards for classification of Portland cement. The two major standards are the ASTM C150 standard, used primarily in the U.S., and European EN-197 standard.

### 2.6.2.1 American standard

Eight types of cement are covered in ASTM C 150 standard. These types and brief descriptions of their uses are listed in Table 2.1.

Table 2.1: Portland cement types and their uses as ASTM C 150

| Cement Type | Usage |
|---|---|
| I | General purpose cement, when there are no extenuating Conditions |
| II | Aids in providing moderate resistance to sulfate attack |
| III | When high-early strength is required |
| IV | When a low heat of hydration is desired |
| V | When high sulfate resistance is required |
| VI | A type I cement containing an integral air-entraining agent |
| IIA | A type II cement containing an integral air-entraining agent |
| IIIA | A type III cement containing an integral air-entraining agent |

### 2.6.2.2 European standard

EN 197-1 defines 5 classes of common cement that comprise Portland cement as a main constituent. These classes differ from the ASTM classes.

I. Portland cement: comprising Portland cement and up to 5% of minor additional constituents

II. Portland composite cement: Portland cement and up to 35% of other single constituents

III. Blast furnace cement: Portland cement and higher percentages of blast furnace slag

IV. Pozzolanic cement: Portland cement and up to 55% of pozzolanic constituents

V. Composite cement: Portland cement, blast furnace slag and pozzolana or fly ash.

- Constituents that are permitted in Portland composite cements are blast furnace slag, silica fume, natural and industrial pozzolana, siliceous and calcareous fly ash, burnt shale and limestone.

## 2.6.3 Properties of Portland cement

### 2.6.3.1 Chemical properties

It is a Portland cement's chemical properties that determine most of its physical properties and how it cures. Therefore, a basic understanding of Portland cement chemistry can help one understand how and why it behaves as it does.

### 1. Chemical composition

The composition of Portland cement distinguishes one type of cement from another. The phase compositions in Portland cement are denoted as tricalcium silicate ($C_3S$), dicalcium silicate ($C_2S$), tricalcium aluminate ($C_3A$), and tetracalcium aluminoferrite ($C_4AF$). The actual components are often complex chemical crystalline and amorphous structures, denoted by cement chemists as "alite" ($C_3S$), "belite" ($C_2S$), and various forms of aluminates. The behavior of each type of cement depends on the content of these components.

Table 2.2: Main constituents in a typical Portland cement

| Chemical Name | Chemical Formula | Shorthand Notation | Percent by weight % |
|---|---|---|---|
| **Tricalcium Silicate** | $3CaOSiO_2$ | $C_3S$ | 50 |
| **Dicalcium Silicate** | $2CaOSiO_2$ | $C_2S$ | 25 |
| **Tricalcium Aluminate** | $3CaOAl_2O_3$ | $C_3A$ | 12 |
| **Tetracalcium Aluminoferrite** | $4CaOAl_2O_3Fe_2O_3$ | $C_4AF$ | 8 |
| **Gypsum** | $CaSO_4H_2O$ | $CSH_2$ | 3.5 |

- Tricalcium silicate ($C_3S$) hydrates and hardens rapidly and is largely responsible for initial set and early strength. Portland cements with higher percentages of $C_3S$ will exhibit higher early strength.
- Dicalcium silicate ($C_2S$) hydrates and hardens slowly and is largely responsible for strength increases beyond one week .
- Tricalcium aluminate ($C_3A$) hydrates and hardens the quickest. It liberates a large amount of heat almost immediately and contributes somewhat to early strength. Gypsum is added to Portland cement to retard $C_3A$ hydration. Without gypsum, $C_3A$ hydration would cause Portland cement to set almost immediately after adding water [28].
- Tetracalcium aluminoferrite ($C_4AF$) hydrates rapidly but contributes very little to strength. Its presence allows lower kiln temperatures in Portland cement manufacturing. Most Portland cement color effects are due to $C_4AF$ .

## 2. Loss on ignition

Loss on ignition is calculated by heating up a cement sample to (900-1000$^{o}$C) (1650 - 1830°F) until a constant weight is obtained. The weight loss of the sample due to heating is then determined. A high loss on ignition can indicate pre-hydration and carbonation, which may be caused by improper and prolonged storage or adulteration during transport or transfer. Maximum loss on ignition permitted by BS 12:1991 and ASTM C150-94 is 3% and 4% respectively. But with cement containing calcareous filler, 5% of the mass of the cement nucleus is allowed by EN 197-1992.

The standard loss on ignition test is contained in EN 196-2 No.7:1994 Standard Test Methods for Determination of Loss on Ignition in European standard and ASTM C 114: Chemical Analysis of Hydraulic Cement in American standard the loss in mass on ignition shall not exceed 4% for Portland cement.

## 3. Insoluble residue

Insoluble residue which is determined by treating cement with hydrochloric acid is a measure of adulteration of cement, largely arising from impurities in gypsum. BS 12 1991 limits the insoluble residue to 1.5% content of a filler, the standard insoluble residue test is contained in EN 196-2 no,9;1994 standard test methods for determination of insoluble residue and EN 196-2 no 10 1994 standard test methods for determination of insoluble residue.

### 2.6.3.2 Physical properties

Portland cements are commonly characterized by their physical properties for quality controlling purposes. Their physical properties can be used to classify and compare Portland cements. EN and ASTM standards have specified certain physical requirements for each type of cement.

#### I. Fineness

Fineness is defined depending upon the method of measurement. It may be defined as sieve diameter, the width of the minimum square aperture through which particle pass, or surface diameter, diameter of sphere having the same surface as the surface of particle. It is a general practice to describe the fineness of cement by a single parameter, the specific surface area.

Fineness of Portland cement has great effects on hydration rate and thus the setting time, and the rate of strength gain. As an example, the smaller is the particle size, the greater the surface area-to-volume ratio. This causes more area available for water-cement interaction. The finer particles mainly affect the early strength of the cement (2 days) while the larger particles dominate the strength after this time. The effects of greater fineness on strength are generally seen during the first seven or twenty eight days.

There are, however, several disadvantages associated with high fineness. In fine cement, more gypsum is required for proper retardation because increased fineness makes more tricalcium aluminate available for early hydration. Grinding clinker to a high fineness requires more energy, increasing the production cost, and a higher early rate of hydration causes a higher early rate of heat liberation. If not properly dissipated, this heat may cause cracking especially in mass concrete construction. The reaction of fine cement with alkali-reactive aggregate is stronger.

Fineness, which has considerable effects on cement strength and hydration rate, is accepted as a vital parameter by European and American Standards. The Wagner Turbid meter and the Blaine air permeability test for measuring cement fineness is required by the American Society for Testing Materials (ASTM).Another test to determine the fineness is Sieve Analysis. The fineness of cement is measured by sieving it on standard sieves.

According to European standard there are two methods to measure cement fineness as designated on EN 196-6,no.3,1989: Standard Test Method for Fineness of Portland Cement by the sieving method and EN 196-6,No.4,1989: Standard Test Method for Fineness of Portland Cement by air permeability method or blain method . Blaine fineness of modern cement ranges from 3,000 to 5,000 $cm^2/g$ (300 to 500 $m^2/kg$) .

### II. Consistency of cement paste

For determination of the initial and final setting times and for the Le-Chatelier soundness test, neat cement paste of standard consistency has to be used. It is, therefore, necessary to determine for any given cement the water content of the paste which will produce the desired consistency. Consistency is measured by Vicat apparatus; the content of a standard paste is expressed as a percentage by mass of the dry cement. The European standard for consistency test is indicated on EN 196-3, No.5:1994 .The usual range of water-cement ratio for normal consistency is between 26% and 33%.

### III. Soundness

When referring to Portland cement, "soundness" refers to the ability of a hardened cement paste to retain its volume after setting without delayed destructive expansion. This destructive expansion is caused by excessive amounts of free lime

(CaO) or magnesia (MgO). Most Portland cement specifications limit magnesia content and expansion.

The typical expansion test places a small sample of cement paste into an autoclave (a high pressure steam vessel). The autoclave is slowly brought to 2.03 MPa (295 psi) and then kept at that pressure for 3 hours. The autoclave is then slowly brought back to room temperature and atmospheric pressure. The change in specimen length due to its time in the autoclave is measured and reported as a maximum autoclave expansion of 0.80 percent for all Portland cement types .

- The standard autoclave expansion tests are: ASTM C 151: Autoclave Expansion of Portland cement in American standard and EN 196-3, No.7 1994: Standard percentage. ASTM C 150, Standard Specification for Portland cement specifies a Test Method for Soundness Test of Portland Cement in European standard.
- According to the Sudanese standard, the expansion of Portland cement shall not exceed 10mm. If this is not satisfied, an additional test shall be made using aerated portion of the same sample. The maximum expansion of such aerated sub sample shall not exceed 5mm.

## IV. Setting time

The stiffening of a cement paste is called setting. The time starting from the mixing of cement and water until the cement paste sets is called the setting time. Cement paste setting time is affected by a number of items including: cement fineness, water-cement ratio, chemical content (especially gypsum content) and admixtures. Setting tests are used to characterize how a particular cement paste sets. For construction purposes, the initial set must not be too soon and the final set must not be too late. Additionally, setting times can give some indication of whether or not cement is undergoing normal hydration. Normally, two setting times are defined: Initial set occurs when the paste begins to stiffen considerably;

final set occurs when the cement has hardened to the point at which it can sustain some load.

These particular times are just arbitrary points used to characterize cement; they do not have any fundamental chemical significance. Both common setting time tests, the Vicat needle and the Gillmore needle, define initial set and final set based on the time at which a needle of particular size and weight either penetrates a cement paste sample to a given depth or fails to penetrate a cement paste sample. The Vicat needle test is more common and tends to give shorter times than the Gillmore needle test. Table 2.2 shows ASTM C 150 specified setting times.

Table 2.3: ASTM C 150 specified setting times by test method

| **Test method** | **Setting type** | **Time specification (minutes)** |
|---|---|---|
| **Vicat** | Initial | 45 |
| | Final | 375 |
| **Gillmore** | Initial | 60 |
| | Final | 600 |

- The standard setting time tests are: ASTM C 191: Time of Setting of Hydraulic Cement by Vicat Needle, ASTM C 266: Time of Setting of Hydraulic-Cement Paste by Gillmore Needles in American standard and EN 196-3.No.6.2, 1994: Standard Test Method for Determination of Initial Setting Time of Cement by Vicat Needle and EN 196-3, No 6.3, 1994: Standard Test Method for Determination of Final Setting Time of Cement by Vicat Neddle in European standard.
- Sudanese standard recommends that the initial setting time for cement not to be less than 45 minutes and the final setting time not to exceed 10 hours.

## V. Strength

Cement paste strength is typically defined in three ways: compressive, tensile and flexural. These strengths can be affected by a number of items including: water-cement ratio, cement-fine aggregate ratio, type and grading of fine aggregate, manner of mixing and molding specimens, curing conditions, size and shape of specimen, moisture content at time of test, loading conditions and age. Since cement gains strength over time, the time at which strength test is to be conducted must be specified. Typically times are 1 day (for high early strength cement), 3 days, 7 days, 28 days and 90 days.

### First: Compressive strength

The most common strength test, compressive strength, is carried out on a 50 mm (2-inch) cement mortar test specimen in American standard. The test specimen is subjected to a compressive load (usually from a hydraulic machine) until failure. Table 2.4 shows ASTM C 150 compressive strength specifications.

Table 2.4: Portland cement mortar compressive strength specification (MPa)

| **Curing time (days)** | **Portland cement type** | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | **I** | **IA** | **II** | **IIA** | **III** | **IIIA** | **IV** | **V** |
| **1** | _ | _ | _ | _ | 12.4 | 10.0 | _ | _ |
| **3** | 12.4 | 10.0 | 10.3 | 8.3 | 24.1 | 19.3 | _ | 8.3 |
| **7** | 19.3 | 15.5 | 17.2 | 13.8 | _ | _ | 6.9 | 15.2 |
| **28** | _ | _ | _ | _ | _ | _ | 17.2 | 20.7 |

Note: type II and IIA requirements can be lowered if either an optional heat of hydration or chemical limit on the sum of C3S and C3A is specified According to European standard, the test is carried out on prismatic test specimen which is 40mmx40mmx160mm cement mortar that is subjected to a compressive load until failure.

The standard cement mortar compressive strength test is described in EN 196-1, No 4.8,1994: Standard Test Method for Compressive Strength of Cement Mortars in European standard and ASTM C 109: Compressive Strength of Hydraulic Cement Mortars (Using 50-mm or 2-in Cube Specimens) and ASTM C 349: Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) in American standard.

### Second: Tensile strength

Although still specified by ASTM, the direct tension test does not provide any useful insight into the concrete-making properties of cements. It persists as a specified test because in the early years of cement manufacturing, it was the most common test since it was difficult to find machines that could compress a cement sample to failure.

### Third: Flexural strength

Flexural strength (actually a measure of tensile strength in bending) is carried out on cement mortar beam that is loaded at its center point until failure. The standard cement mortar flexural strength test is described in: ASTM C 348: Flexural Strength of Hydraulic Cement Mortars in American standard and EN 196-1, No4.7: Standard Test Method for Flexural Strength of Cement Mortars in European standard.

## VI. Heat of hydration

The heat of hydration is the heat generated when water and Portland cement react. Hydration begins at the surface of the cement particles. Therefore, the total surface area of cement represents the material available for hydration. That is, the early rate of hydration depends on the fineness of the cement particles. However, at later stages, the effect of surface area diminishes and, consequently, fineness exercise no influence on the total heat of hydration.

Heat of hydration is also influenced by the proportion of $C_3S$ and $C_3A$ in the cement, water-cement ratio, fineness and curing temperature. As each one of these factors is increased, heat of hydration increases. In large mass concrete structures such as gravity dams, hydration heat is produced significantly faster than it can be dissipated (especially in the centre of large concrete masses), which can create high temperatures in the centre of these large concrete masses that, in turn, may cause undesirable stresses as the concrete cools to ambient temperature. Conversely, the heat of hydration can help maintain favorable curing temperatures during winter.

# Chapter Three

# Materials

## 3.1 Introduction

In this chapter, the materials used for the investigation are described with respect to their sources and relevant physical and chemical properties. All laboratory investigations on the materials used in studying the properties of steel slag blended cements are carried out in Nile cement factory at the quality control department.

## 3.2 Materials used in studying the properties of steel slag blended cement

### 3.2.1 Clinker

For the production of laboratory cements, clinker from Nile cement Factory was chosen. For this test purpose, the sample of the clinker was collected from the clinker silo which was collected after passing the full clinkerization process by controlling the raw materials in such a way that their chemical composition were within the norm of the factory.

To have consistent product and to keep the quality of cement, the factory used to control the raw material mix by chemical analysis and computerized control system. For this purpose, from experience, it sets its norm chemical range for each raw material such that the clinker is expected to be consistent and be within the standard. It corrects any deviation, of the raw material chemical composition from the range, by adjusting the raw material proportion and or using corrective raw materials. The clinker used for the test was also produced from raw materials whose chemical composition was within the norm of the factory.

The sample of the clinker particles of size between 0.8 cm to 1.5 cm, having blackish gray color and intermediate hardness r was tested for its chemical and mineralogical composition and the test results are shown in Table 3.1 and 3.2 below.

Table 3.1: Chemical composition of clinker used for the test

| Component | Percentage (%) |
|---|---|
| **CaO** | 64.40 |
| **$SiO_2$** | 21.46 |
| **$Al_2O_3$** | 04.85 |
| **$Fe_2O_3$** | 04.79 |
| **MgO** | 03.63 |
| **$SO_3$** | 00.50 |
| **L.O.I** | 00.79 |
| **F. L** | 02.42 |

Table 3.2: Mineralogical composition of clinker used for the test

| Mineralogical composition | Test result (%) |
|---|---|
| **$C_3S$** | 48.37 |
| **$C_2S$** | 25.04 |
| **$C_3A$** | 04.75 |
| **$C_4AF$** | 14.96 |

### 3.2.2 Gypsum

The gypsum used for the test was also taken from the source of Nile cement factory. Before it was used in the Portland cement production, the gypsum was crushed in raw material grinding mill and dried in the lab. The particle size in between10 mm to 15mm of density 1.2 g/cm$^3$

The chemical composition of the gypsum was tested to check its conformity to the norm of the factory. The test results are shown in Table 3.3 below.

Table 3.3: Chemical composition of gypsum used for the test

| **Component** | **Percentage %** |
|---|---|
| **CaO** | 33.60 |
| **$SiO_2$** | 05.60 |
| **$Al_2O_3$** | 00.26 |
| **$Fe_2O_3$** | 00.40 |
| **MgO** | 01.01 |
| **$SO_3$** | 41.93 |

### 3.2.3 Steel Slag

Steel slag used in tests is a byproduct of steel smelting from scrap in Giad Steel Industry in Khartoum with bulk density of 1.102 g/cm3 and specific density of 1.65 g/cm3.

Table 3.4: Chemical composition of steel slag used for the test

| Component | Percentage % |
|---|---|
| **CaO** | 30.80 |
| **$SiO_2$** | 23.42 |
| **$Al_2O_3$** | 11.09 |
| **$Fe_2O_3$** | 18.55 |
| **MgO** | 07.26 |
| **$SO_3$** | 00.46 |
| **L.O.I** | 02.46 |
| **F.L** | 00.07 |

### 3.2.4 Water

Throughout the investigation, tap water supplied for drinking consumption at Nile Factory was used for curing the hardened mortar samples. For all physical and chemical analysis distilled water was used.

### 3.2.5 Chemicals

For determination of the chemical and mineralogical composition as well as sulphate and insoluble residue, different chemicals were used as per the specified method of testing cement based on the European standard EN 196.2.

### 3.2.6 Sand

Locally available sand was used in tests as fine aggregate, obtained from west of Kosti town. Firstly it's sieved, washed and dried to be ready for using.

## 3.3. Materials used in studying concrete properties

### 3.3.1 Cement

For all concrete specimens casted for the investigation, cement of Nile Ordinary Portland, which was manufactured according to Sudanese standard SD-164/2013 and European standard EN-197-1-2000, was used. It’s typical chemical and mineralogical compositions are shown in Table 3.5 and 3.6 below:

Table 3.5: Chemical composition of cement used for the test

| **Component** | **Percentage (%)** |
|---|---|
| **CaO** | 63.16 |
| **$SiO_2$** | 20.80 |
| **$Al_2O_3$** | 04.61 |
| **$Fe_2O_3$** | 04.67 |
| **MgO** | 03.53 |
| **$SO_3$** | 02.16 |
| **L.O.I** | 01.90 |
| **F. L** | 02.32 |

Table 3.6: Mineralogical composition of cement used for the test

| **Mineralogical composition** | **Test result (%)** |
|---|---|
| **$C_3S$** | 45.33 |
| **$C_2S$** | 25.51 |
| **$C_3A$** | 04.57 |
| **$C_4AF$** | 14.03 |

### 3.3.2 Aggregates

Throughout the experiment, river sand and basaltic crushed stone from local market, with the following physical characters, were used as fine and coarse aggregate respectively.

#### 3.3.2.1 Silt content of fine aggregate

The presence of dust, loam and clay materials with sand decreases the bond between the materials to be bound together thereby decreases the strength of concrete besides decreasing the quality of concrete. Accordingly, the sand for the experiment was tested for silt content and was found to have 13% silt content. Therefore, the sand, before used in all tests, was washed until clear water came out.

#### 3.3.2.2 Gradation of fine and coarse aggregate

Aggregate grain size distribution or gradation is one of the properties of aggregates which influence the quality of concrete. Therefore, fine aggregate and coarse aggregate with gradation satisfying the grading requirement of B.S standard as shown in Table 3.7 and 3.8 respectively were used throughout the experiment.

Table 3.7: Gradation of fine aggregate used for the test

| Sieve size | Weight Retained (gm) | Percentage retained | Cumulative coarser (%) | Cumulative passing (%) |
|---|---|---|---|---|
| 9.50 mm | 0 | 0 | 0 | 100 |
| 4.75 mm | 17 | 02.53 | 02.53 | 97.47 |
| 2.36 mm | 30 | 04.48 | 07.01 | 92.99 |
| 1.18 mm | 95 | 14.16 | 21.17 | 78.83 |
| 600 µm | 158 | 23.55 | 44.72 | 55.28 |
| 300 µm | 186 | 27.72 | 72.44 | 27.56 |
| 150 µm | 126 | 18.78 | 91.22 | 08.78 |
| pan | 53 | 08.78 | 100 | - |

Table 3.8: Gradation of coarse aggregate used for the test

| Sieve size | Weight Retained (gm) | Percentage retained | Cumulative coarser (%) | Cumulative passing (%) |
|---|---|---|---|---|
|  | 0 | 0 | 0 | 100 |
| 37.5 mm | 0 | 0 | 0 | 100 |
| 25 mm | 901 | 11.33 | 11.33 | 88.67 |
| 19.5 mm | 1495 | 18.79 | 30.12 | 69.88 |
| 12.5 mm | 3144 | 39.53 | 69.65 | 30.35 |
| 9.5 mm | 1379 | 17.34 | 86.99 | 13.01 |
| 4.75 mm | 927 | 11.65 | 98.64 | 01.36 |
| pan | 107 | 01.36 | 100 | - |

### 3.3.2.3 Unit weight, specific gravity and absorption capacity of aggregates

As unit weight, specific gravity and absorption capacity affect the type and quality of concrete and as such parameters are input for mix designing, these parameters were determined for both fine and coarse aggregate. The determined values are shown in Table 3.9.

Table 3.9: Physical test results of aggregates used for the test

| **Type of aggregate** | **unit weight kg/cm$^3$** | **specific gravity g/cm$^3$** | **absorption capacity %** |
|---|---|---|---|
| Fine | 1.691 | 2.57 | 1.83 |
| Coarse | 1.345 | 2.70 | 1.15 |

### 3.3.3 Water

Tap water suitable for drinking is usually good enough for cement mortar. The water should be free of all organic matter and certain chemicals such as alkaline and sulfate salts.

### 3.3.4 Chemicals

Water reducing admixture SP was used during mixing for high strength concrete samples as per the manufacturer’s instruction.

# Chapter four

# Experimental program

## 4.1 Introduction

The main objectives of the experimental program are to study the effects of using steel slag as an additive material for the manufacture of Portland cement, specifically on the physical and chemical properties of cement paste and hardened mortar. And also aim to study the effects of using steel slag as a replacement material on the performance of concrete. To achieve these objectives, two major experiments were designed.

The first experiment (experiment one) was done to determine the effects of replacing part of Ordinary Portland cement clinker with steel slag on various properties of cement such as water requirement or normal consistency, setting time, soundness, compressive strength, and fineness.

Following experiment one, another experiment (experiment two) was performed to determine the effects of replacing part of cement with steel slag on concrete performance such as workability, strength, and permeability.

## 4.2 Experiment one

Experiment one, which consists of preparation of steel slag, determination of fineness of steel slag, choosing type of cement and fineness of cement for the experiment, production of cement, preparation of blended cements, different physical and chemical tests of cement and preparation and test of mortar and cement pastes, was designed to determine the effects of replacing part of Ordinary Portland cement with steel slag on various properties of cement.

### 4.2.1 Preparing of the testing cement

The First step in the experimental program is how to prepare the testing cement with specified properties as shown below:

#### 4.2.1.1 Preparation of steel slag

The steel slag originally was discharged from the EAF in slurry form and was cooled by atmospheric air to form blocks of different sizes:

1. Coarse :(2.5”- 8”) 64-203 mm
2. Intermediate: (5/8” - 2.5”) 16-64 mm
3. Fine (Minus 5/8”) 0-16 mm

The interest of the researcher was to use the steel slag after crashing to fine size (90 µc) to be suitable for testing.

#### 4.2.1.2 Determination of the fineness of steel slag

After grinding, the fineness of steel slag sample was tested for fineness by Blaine air permeability apparatus. And it was found that the testing sample had a Blaine fineness of 3120 $cm^2$/gm.

#### 4.2.1.3 Production of cement for the experiment

For producing cement for the research, clinker passing the normal controlling step and composed of raw materials with chemical composition within the range of the norm of Nile cement factory was taken and ground with 4% gypsum which fulfills the norm of chemical composition range of the factory. The clinker and gypsum was ground in a mill to fineness of 3113 $cm^2$/gm.

### 4.2.2 Preparation of blended cements

After Ordinary Portland cement was produced to the required fineness in the mill, and after the steel slag was prepared as described in section **4.2.1**, group of blended cements were prepared by mixing Steel Slag with 5, 10, 15, 20% addition of the mass of Ordinary cement. This blending was done in dry condition by rotary mixer such that the distribution of the filler will be homogenous and uniform. The blended cements (5 in number) ,were prepared by blending Nile OPC with steel slag of Blaine fineness 3120 $cm^2/gm$ from 5 to 20 % ranges as shown below in Table 4.1.

Table 4.1: Proportion of blending of OPC with Steel Slag

| **Sample Number** | **Code** | **Clinker %** | **Gypsum %** | **Steel Slag %** |
|---|---|---|---|---|
| 1 | NOPC | 96 | 4 | 0 |
| 2 | NS05 | 91 | 4 | 5 |
| 3 | NS10 | 86 | 4 | 10 |
| 4 | NS15 | 81 | 4 | 15 |
| 5 | NS20 | 76 | 4 | 20 |

- Note: The description of the blending code is:

NOPC: indicates control cement prepared by Nile OPC without steel slag.

NS05: indicates Nile OPC cement blended with 5% steel slag.

NS10: indicates Nile OPC cement blended with 10% steel slag.

NS15: indicates Nile OPC cement blended with 15% steel slag.

NS20: indicates Nile OPC cement blended with 20% steel slag.

### 4.2.3 Determinations of chemical properties of blended cement

The chemical compositions of cement samples were determined by using the traditional method that dependent on chemical titrations. The testing procedures based on British Standards (BS12: 1992) have been followed for chemical analysis of Portland cement. Amount of total $SiO_2$, $Al_2O_3$, $Fe_2O_3$, lime content, MgO, $SO_3$, free lime and L.O.I were determined.

### 4.2.4 Determination of physical properties of blended cement

#### 4.2.4.1 Fineness

Fineness is defined depending upon the method of measurement. It may be defined as sieve diameter, the width of the minimum square aperture through which particle pass, or surface diameter, diameter of sphere having the same surface as the surface of particle.

According to European standard there are two methods to measure cement fineness as designated on EN 196-6,no.3,1989: Standard Test Method for Fineness of Portland cement by the sieving method and EN 196-6,No.4,1989: Standard Test Method for Fineness of Portland Cement by air permeability method or Blaine method. Blaine fineness of modern cement ranges from 3,000 to 5,000 $cm^2/g$ approximately not less than 2250 $cm^2/g$.

#### 1. Blaine air permeability test

Blaine Air Permeability Apparatus measures the specific surface area of fine materials in square centimeters per gram of test sample. A quantity of air is drawn through a bed of definite porosity. The pore volume in the bed is a function of the size of particles, and determines the rate of air flow.

The apparatus is supplied complete with stainless steel test cell, plunger, perforated disk, calibrated U-tube manometer, rubber aspirator and bulb, all mounted on a sturdy wooden panel and base. Besides, manometer fluid, package of filter paper, and a wood block for holding cell during filling are included.

The principle of the test depends on impelled air penetrate into compressed cement barriers within known dimensions (permeability cell). Taken a known weight of the sample of cement and it is estimated with 2.9005 gm and placed inside the cell between two filtering papers by using funnel then cement pressure within the cell using a plunger, then placed it on the top of manometric tube after filling the internal aspects of the tube and also the outer edges of the cell with wax to prevent the passage of air between the blanks.

Air is pull up from inside the cell by Blower, which in turn raises the level of the liquid with the tube and then begins to go down gradually depending on the force of air pushing it from the top and record how long it takes the liquid by using a stop watch and to confirm the experiment repeated three times.

Necessary before starting the test to measure the room temperature by Thermometer because it affects on the viscosity of the manometer liquid Therefore, we find that there is a coefficient for each temperature starting from 28 till 32.

- To set the specific surface of cement expressed by total area by $cm^2$ / gm:

$$\textbf{Blaine} = (\sqrt{t}) * T_F * B_F \longrightarrow \text{equation (4.1)}$$

- t: average time
- $B_F$: Blaine factor = 5.6274
- $T_F$: Temperature factors:

28°C …………… 73.6 , 30°C …………… 73.4

32°C …………... 73.2 , 34°C …………… 73.0

### 2. Sieves method

Weigh 20 g of dry cement put on measuring sieve 170 ( 90 µc ) for 15 minutes if the sieving process conducted by hand then weighed cement remaining on the sieve. Fineness estimated as percentage ratio (1 to 2 %). Taken into account when conducting the test not to lose any amount of cement and the use of a brush to clean the bowl and sieve.

### 4.2.4.2 Soundness test

Soundness of cement is determined by Le-Chatelier method, it consists of a small split cylinder of spring brass or other suitable metal. It is 30 mm in diameter and 30 mm high. On either side of the split are attached two indicator arms 165 mm long with pointed ends. Take 200 gm of cement and mix with suitable amount of water to make cement paste with standard consistency then fill into the mould. The mould is covered on the top with another glass plate, place a small weight on this covering glass sheet and immediately submerge the whole assembly in water at a temperature of 19 ± 1 $C^o$ and keep it there for 24 hrs.

Measure the distance separating the indicator points to the nearest 0.5mm ($d_1$). Then submerge the mould again in water at the temperature prescribed above. Bring the water to boiling point in 25 to 30 minutes and keep it boiling for one hour. Remove the mould from the water, allow it to cool and measure the distance between the indicator points ($d_2$). The value of ($d_2 - d_1$) represents the expansion of cement it should be not more than 10 mm.

The permissible expansion is 10mm according to the standards specification of cement.

### 4.2.4.3 Standard consistency test

For finding out initial setting time, final setting time and soundness of cement, and strength a parameter known as standard consistency has to be used. The standard consistency of a cement paste is defined as that consistency which will permit a Vicat plunger having 10 mm diameter and 50 mm length to penetrate to a depth of 33-35 mm from the top of the mould. The apparatus is called Vicat Apparatus.

The following procedures are adopted to find out standard consistency. Take about 500 gm of cement and prepare a paste with a weighed quantity of water (say 24 per cent by weight of cement) for the first trial. The paste must be prepared in a standard manner and filled into the Vicat mould within 3-5 minutes. After completely filling the mould, shake the mould to expel air. A standard plunger is attached and brought down to touch the surface of the paste in the test block and quickly released allowing it to sink into the paste by its own weight. Take the penetration of the plunger. Conduct a 2nd trial (with 25 per cent of water) and find out the depth of penetration of plunger. Similarly, conduct trials with higher and higher water/cement ratios till such time the plunger penetrates for a depth of 33-35 mm from the top. That particular percentage of water which allows the plunger to penetrate only to a depth of 33-35 mm from the top is known as the percentage of water required to produce a cement paste of standard consistency.

### 4.2.4.4 Setting time test

Prepare a cement paste by mixing (400 g) of cement with water required to give a paste of standard consistency. Start a stop-watch, the moment water is added to the cement. Then fill the Vicat mould completely with the cement paste, the mould resting on a non-porous plate and smooth off the surface of the paste making it level with the top of the mould.

**a) Initial setting time:**

Place the test block under the rod bearing the needle. Lower the needle gently in order to make contact with the surface of the cement paste and release quickly, allowing it to penetrate the test block. Repeat the procedure till the needle fails to penetrate the test block to a point 5.0 ± 0.5mm measured from the bottom of the mould. The time period elapsing between the time, water is added to the cement and the time, the needle fails to penetrate the test block by 5.0 ± 0.5mm measured from the bottom of the mould, is the initial setting time.

**b) Final setting time:**

Replace the above needle by the one with an annular attachment. The cement should be considered as finally set when, upon applying the needle gently to the surface of the test block, the needle makes an impression therein, while the attachment fails to do so. The period elapsing between the time, water is added to the cement and the time, the needle makes an impression on the surface of the test block, while the attachment fails to do so, is the final setting time.

### 4.2.4.5 Compressive strength

The compressive strength of hardened cement is the most important of all properties. Therefore, it is not surprising that the cement is always tested for its strength at the laboratory before the cement is used in important works. Strength tests are not made on neat cement paste because of difficulties of excessive shrinkage and subsequent cracking of neat cement. Strength of cement is indirectly found on cement sand mortar in specific proportions.

The standard sand is used for finding the strength of cement. Take 555 gm of standard sand, 185 gm of cement (i.e., ratio of cement to sand is 1:3) mix them with a trowel for one minute, then add 74 gm of water of mix the three ingredients thoroughly until the mixture is of uniform color. The time of mixing should not be less than 3 minutes nor more than 4 minutes. Immediately after mixing, the mortar is filled into a cube mould of size $50cm^2$. Compact the mortar either by hand compaction in a standard specified manner or on the vibrating equipment (12000 rpm) for 2 minutes. Keep the compacted cube in the mould at a temperature of 19C° ± 1C° and at least 90 per cent relative humidity for 24 hours. Where the facility of standard temperature and humidity room is not available, the cube may be kept under wet conditions to simulate 90 per cent relative humidity.

After 24 hours the cubes are removed from the mould and immersed in clean fresh water until taken out for testing. Three cubes are tested for compressive strength at the periods mentioned. The compressive strength shall be the average of the strengths of the three cubes for each period.

## 4.3 Experiment two

Experiment two, which consists of determination of the physical parameters of fine and coarse aggregate, mix designing, concrete specimens preparation and tests of fresh and hardened concrete cubes and beams, was designed to determine the effects of replacing part of cement and sand with marble waste powder on concrete performance.

### 4.3.1 Determination of physical parameters of aggregates (fine and coarse)

Aggregates are important constituents in concrete. The mere fact that aggregates occupy 70-80 percent of the volume of concrete, their impact on various characteristics and properties of concrete is undoubtedly considerable. The depth and range of studies that are required to be made in respect of aggregates to understand their widely varying effects and influence on the properties of concrete cannot be underrated. As aggregates are natural materials their proprieties vary in different extent that concretes of different aggregates vary accordingly.

Different physical parameters of aggregates are required to be within a certain limit by different standards so that the concrete from that aggregate will give the intended performance. Some physical parameters are also important as an input in mix designing. Therefore, the following properties of aggregates were tested and determined.

1. Gradation of fine and coarse aggregates by sieving with procedures as indicated in construction materials laboratory manual. From this test the gradation of both coarse and fine aggregates were found to be within the standard requirement of B.S
2. Silt content of fine aggregate: for determination of silt content by mass, a sample of fine aggregate was taken and it was dried in an oven at 105 $^{o}$C for 24 hours. It was then sieved on a 1.18 mm sieve. The material passing was weighed and its mass was recorded as $\mathbf{m_1}$.It was then thoroughly washed on a 75μm until clear water came out, and again dried in the oven at 105 $^{o}$C for another 24 hours. Its final mass was then recorded as $\mathbf{m_2}$.The silt content $\mathbf{S_{content}}$ was then calculated as

$$\mathbf{S_{content} = (m_1 - m_2) / m_1 * 100} \longrightarrow \qquad \text{equation (4.2)}$$

By this test, the silt content was found to be 13% which is above the limit recommended by B.S standard; therefore the sand was washed before use.

3. The specific gravity and absorption capacity of fine and coarse aggregates were determined by methods and procedures indicated in construction materials laboratory manual as they are inputs for mix designing. The unit weights of the aggregates were also determined.

### 4.3.2 Mix designing and trial mix preparation

Mix design can be defined as the process of selecting suitable ingredients of concrete and determining their relative proportions with the object of producing concrete of certain minimum strength and durability as economically as possible. For this experiment, design mix is done using DOE method for the class of concrete (C-50).The proportion of the materials as per the design is given in Table 4.2 below:

Table 4.2: The proportion of the materials

| **Type of Material** | **Quantity kg** |
|---|---|
| Cement | 09.90 |
| Sand | 16.28 |
| Coarse aggregate | 22.66 |
| Water | 04.85 |

### 4.3.3 Concrete Specimens preparation

For studying the effects of replacing parts of cement by steel slag, a group of concrete specimens were prepared as shown in table 4.3.

Table 4.3: Proportion of concrete specimens prepared by cement blended with steel slag

| **Sample Number** | **Code** | **Cement kg** | **Coarse Aggregate kg** | **Fine Aggregate kg** | **Water kg** | **Admixture gm** | **w/c** |
|---|---|---|---|---|---|---|---|
| 1 | CN0 | 9.9 | 22.66 | 16.28 | 4.85 | 99 | 0.48 |
| 2 | CN5 | 9.9 | 22.66 | 16.28 | 4.85 | 99 | 0.48 |

- Note:

CN0: indicates control concrete sample prepared with cement without steel slag.
CN5: indicates concrete sample prepared with cement blended with 5% steel slag.
w/c : water per cement ratio.

For each mix, mixing was done homogenously to respective cement or sand. Then coarse aggregate, fine aggregate and cement was put to the mixer one over the other. These materials were mixed dry by vertical rotary mixer for about 1 minute. After dry mix, water was added. The wet mix was mixed for more than 2 minutes. During each mix slump test was taken.

After the mix was done, the fresh concrete was then poured in to the mould and compacted in three layers by hand compaction. Hand compaction is done with the help of steel tamping rod.

### 4.3.4 Curing

After the concrete was cast it was left for a time in open air .Then it was trimmed, and finished inside the mold .After finishing the specimens were left in the mold for 24 hours after which they were released from the mold and immersed in water pond for curing until test period.

# Chapter Five

# Experimental Results and Discussions

## 5.1 Introduction

In this section, the test results on the physical and chemical properties of steel slag blended cements (test results of experiment 1), the test results on the performance of concrete made with steel slag blended cement (test results of experiment 2) are presented, analyzed and discussed.

## 5.2 Test results and discussions on experiment one

In this part the test results on the physical and chemical properties of steel slag blended cements are presented, analyzed and discussed.

### 5.2.1 Suitability of steel slag as additive in cement production

To check the suitability of the steel slag as blending material in cement production, the research began by studying the physical and chemical properties of the steel slag.

Physically, as tested in the lab, the fineness value of the steel slag was 3120 $cm^2/g$, comparable with the fineness of ordinary Portland cement (3000-5000 $cm^2/g$).

Chemical analysis was carried out at the chemical laboratory of Nile cement industry. The chemical composition of steel slag was tested in accordance to (ASTM C 311 and ACI 233) and the results are shown in Table 3.4 and figure 5.1.

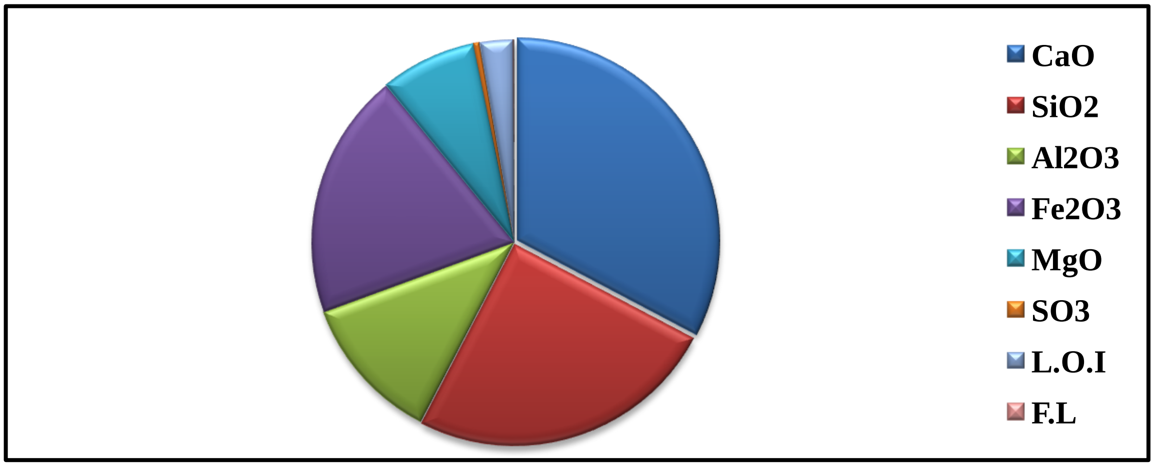

Fig 5.1: chart of chemical composition of steel slag

The limiting values according to BS 6699: 1992 are the maximum insoluble residue of 1.5 percent, maximum magnesia content of 14 percent, maximum loss on ignition of 3 percent. In addition, the maximum lime/silica ratio is 1.4 and minimum chemical modulus [(CaO) + (MgO)]/ ($SiO_2$), is 1.0.

According to BS 146:1991 and BS 4246:1991, at least two- thirds of the total mass of slag must consist of the sum of CaO, MgO, and $SiO_2$. Also, the ratio of the mass of CaO plus MgO to the mass of $SiO_2$ must exceed 1.0, this ratio assures a high alkalinity, without which the slag would be hydraulically inactive.

However, when taking into account the second and the third criterion (BS 146:1991,BS 4246:1991 and BS 6699:1992) we find that the sum of CaO, MgO, and $SiO_2$ is 61.48 less than the minimum, whereas the ratio of [(CaO) + (MgO)]/ ($SiO_2$) is 1.6 which exceeds the minimum. In addition, the lime/ silica ratio is 1.32close to the standard. Also, the value of LOI is 2.46<3% whereas $SO_3$ 00.46<4%, they clearly satisfy the requirements of BS 6699:1992.

### 5.2.2 Fineness of cement pastes

To see the effects of steel slag on the cement chosen for the test, the fineness of all steel slag blended cements were determined by Blaine air permeability apparatus and sieve. The test results are shown in Table 5.1 below.

Table 5.1: Fineness of steel slag blended cements

| **Sample number** | **Code** | **By Blaine ($cm^2/g$ )** | **By 45 µc sieve (%)** | **By 90 µc sieve (%)** |
|---|---|---|---|---|
| 1 | NOPC | 3500 | 25.5 | 2.6 |
| 2 | NS05 | 3411 | 27.9 | 2.8 |
| 3 | NS10 | 3237 | 29.1 | 2.9 |
| 4 | NS15 | 3150 | 29.8 | 3.0 |
| 5 | NS20 | 3006 | 32.3 | 3.2 |

From these tests, the increment of retained particles on sieving, with 45 µc sieve, shows that the steel slag has certain percentage of grains coarser than ordinary cement. Sieving on 90 µc also ascertains the presence of more grains greater than size 90 µc in the steel slag than in cement. In addition to Blaine test shows decrease of Blaine with increment of percentage of steel slag in all cases.

### 5.2.3 Consistency of cement pastes

Normal consistency tests, for the blended cements, were conducted, by Vicat apparatus, to observe the changes in water requirement of pastes due to steel slag additives.

Table 5.2: Normal consistency of steel slag blended cement pastes

| **Sample number** | **Code** | **Consistency (%)** |
|---|---|---|
| 1 | NOPC | 30 |
| 2 | NS05 | 29 |
| 3 | NS10 | 29 |
| 4 | NS15 | 29 |
| 5 | NS20 | 29 |

The test results, as shown in table 5.2, reveal a slight decrease in water requirement for all cement with steel slag addition when compared to the control cement. This is attributed to the increment of fineness of blended cements. But the increment of the percentage of steel slag addition from 5 to 20% didn’t show remarkable change on the water requirement.

In all cases of blending, the normal consistencies of the pastes are within the range of the normal consistency for normal cement which is between 26-33%

### 5.2.4 Setting Time

Tests for setting time were conducted to compare the setting time of the blended cements with standards and also with the control paste. The test results are shown in table 5.3.

Table 5.3: Initial and final setting time of steel slag blended cement pastes

| **Sample number** | **Code** | **Initial ( minute )** | **Final ( minute)** |
|---|---|---|---|
| **1** | NOPC | 125 | 190 |
| **2** | NS05 | 115 | 175 |
| **3** | NS10 | 115 | 170 |
| **4** | NS15 | 110 | 170 |
| **5** | NS20 | 110 | 165 |

The results show that the addition of Steel Slag has reduction effect on initial setting time up to 20%. The addition of Steel Slag also reduces final setting time compared to the paste with cement of 0% Steel Slag addition (the control).

EN 197-1:2000 limits the initial setting time for composite Portland cement not to be less than 45 minutes. Comparing test results with standards, blended cements by addition of steel slag satisfy the requirement of European and ASTM.

### 5.2.5 Soundness

When referring to Portland cement, "soundness" refers to the ability of a hardened cement paste to retain its volume after setting without delayed destructive expansion. This destructive expansion is caused by excessive amounts of free lime (CaO) or magnesia (MgO). Most Portland cement specifications limit magnesia content and expansion. According to the EN 196-1 standard, the expansion of Portland cement shall not exceed 10mm .In the research Le-Chatlier expansion tests were conducted for soundness test. The results are shown in Table 5.4 below.

Table 5.4: Soundness of steel slag blended cement pastes

| Sample number | Code | Soundness (mm) |
|---|---|---|
| **1** | NOPC | 1 |
| **2** | NS05 | 1 |
| **3** | NS10 | 1 |
| **4** | NS15 | 1 |
| **5** | NS20 | 1 |

The result revealed that the addition of marble waste powder on Ordinary Portland cement has no remarkable effects on the soundness of cement pastes. During the investigation, sound produced by colliding dried pastes were observed and was uniform and thin light which indicates that there is no problem in expansion or no sign of cracking.

The absence of expansion in marble waste powder blended cement like the OPC cement reveals that the amount of free lime and MgO is controlled in clinkerization step and the free lime and MgO in the additive are little in quantity to affect late expansion.

### 5.2.6 Compressive strength of hardened mortars

Different standards set minimum strength for different purpose cement as standard controlling parameters. In this research, mortars were prepared from Nile OPC cement and Steel Slag blended cements in accordance with EN 196-1, Methods of testing cement, for examining the effects of the Steel Slag on the compressive strengths of mortars.

The compressive strength of the mortars were tested on 2,7 and 28 days. The detail test results are given in the Appendices. But for discussion, the summarized and averaged test results are given in Table 5.5 below.

Table 5.5: Summarized compressive strengths of test mortars for two, seven and twenty eight days

| **Sample number** | **Code** | **Compressive Strength ( N/mm$^2$ )** | | |
|---|---|---|---|---|
| | | **2 days** | **7 days** | **28 days** |
| 1 | NOPC | 19.6 | 30.0 | 43.0 |
| 2 | NS5 | 16.8 | 31.4 | 46.0 |
| 3 | NS10 | 13.6 | 29.0 | 45.8 |
| 4 | NS15 | 18.4 | 32.2 | 44.0 |
| 5 | NS20 | 20.2 | 32.0 | 43.0 |

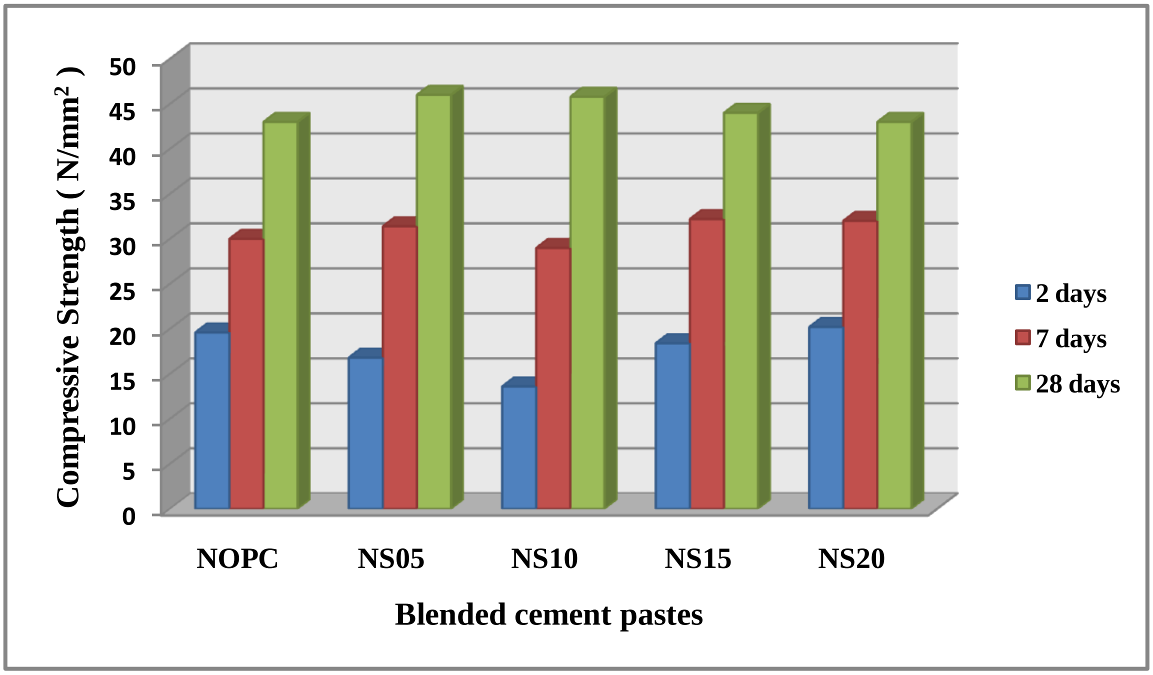

Fig 5.2: Histogram of compressive strength of test mortars for two, seven and twenty eight days

As shown in figure (5.2), the effects of steel slag addition to clinker on two days compressive strengths are different. The addition of 5%, 10% and 15% of steel slag decreases the compressive strength, however, the addition of 20% cause increase the compressive strength.

On the other hand, the test results on the seven days compressive strength showed the similar effect on different types of cement mortars, therefore, all additions of steel slag cause decrease the compressive strength compared with the control one.

The test results on the 28days compressive strength test showed different effects degree. The addition of 5%,10% of steel slag decrease the compressive strength compared to the control one and more addition of steel slag up to 20 % led still more decreases in strength, however, all results are in the range of standard value 42.5.

According to EN 197-1 the compressive strengths of the blended cements at two, seven and 28 days satisfy the EN 197-1 compressive strength limit for cement requirement of high early strength class 42.5 MPa.

### 5.2.7 Strength activity index test

The Strength Activity Index SA is calculated as follows:

$$\mathbf{SA\ \% = (S_c / S_r) *100\%} \longrightarrow \quad \text{equation (5.1)}$$

$S_c$ : Strength of control sample at 28 Days

$S_r$ : Strength of 5% replacement at 28 Days

- (5%) Strength Activity Index of 28 Days = (46/48) *100 % = 95.8%

  ASTM C 989 set 90% as minimum of strength activity index, hence the locally produced electric arc furnace slag has higher strength activity index than the minimum required by ASTM C 989.

### 5.2.8 Effect of steel slag on the chemical properties of cement pastes

Table 5.6: Chemical compositions of steel slag blended cement compared with control OPC

| **Sample Code** | **NOPC** | **NS05** | **NS10** | **NS15** | **NS20** |
|---|---|---|---|---|---|
| **$SiO_2$** | 20.83 | 20.98 | 21.06 | 21.22 | 21.62 |
| **$Al_2O_3$** | 04.65 | 04.77 | 04.81 | 04.97 | 05.07 |
| **$Fe_2O_3$** | 04.61 | 05.31 | 06.01 | 06.71 | 07.41 |
| **CaO** | 63.17 | 61.49 | 59.81 | 58.13 | 56.45 |
| **MgO** | 03.53 | 03.66 | 03.79 | 03.92 | 04.05 |
| **L.O.I** | 00.76 | 00.84 | 00.93 | 01.01 | 01.09 |
| **$SO_3$** | 02.20 | 02.15 | 02.00 | 0 1.98 | 01.77 |
| **F.L** | 02.32 | 02.21 | 02.09 | 01.97 | 01.85 |

From the results, as shown in Table 5.6, it is seen that the addition of steel slag increases the percentage of $SiO_2$, $Al_2O_3$, $Fe_2O_3$, MgO and the loss of ignition (L.O.I). Besides that, the addition of steel slag reduces the percentage of CaO, $SO_3$ and Free lime (F.L) than the control OPC.

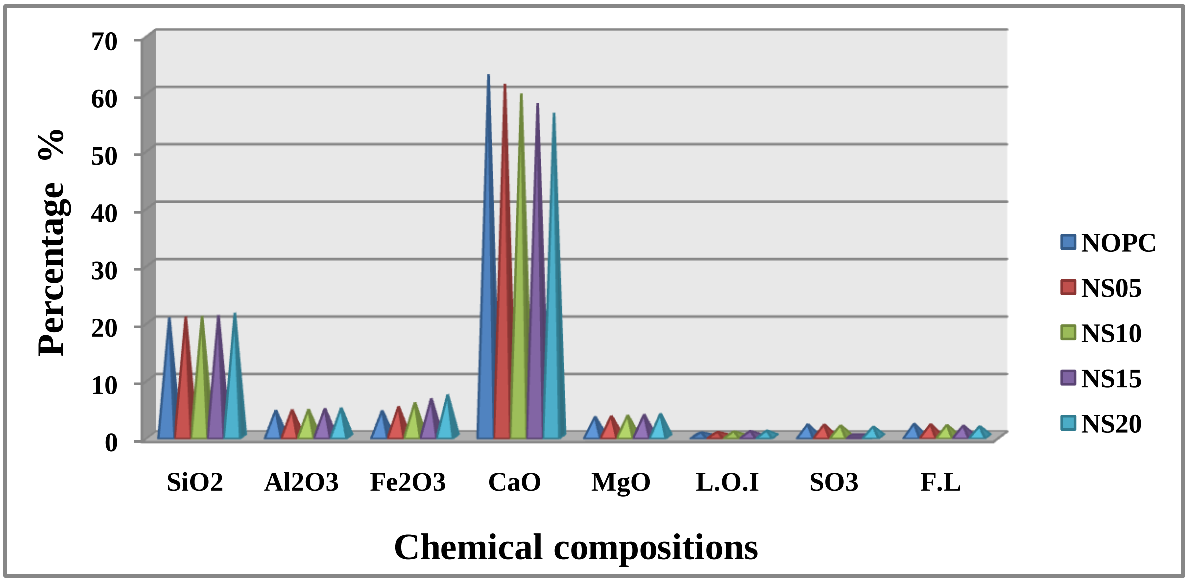

Fig 5.3: Histogram of chemical compositions of steel slag blended cement

## 5.3 Test results and discussions on experiment two

In this part, the test results on performance of concretes made with marble waste blended cement, and test results on performance of concretes made with sand blended with marble waste powder are presented, analyzed and discussed.

### 5.3.1 Workability

Workability is affected by every component of concrete and essentially every condition under which concrete is made. A list of factors include the properties and the amount of cement, grading, shape, angularity and surface texture of fine and coarse aggregates, proportion of aggregates, amount of air entrained, type and amount of pozzolana, type and amount of chemical admixture, temperature of the concrete, mixing time and method, and time since water and cement are in contact. These factors interact so that changing the proportion of one component to produce a specific characteristic requires that other factors be adjusted to maintain workability.

Numerous attempts have been made by many research workers to quantitatively measure workability of concrete. But none of these methods are satisfactory for precisely measuring or expressing this property to bring out its full meaning (Shetty, 2005).

In this experiment slump of all mixes with constant water to cementious material (w/cm) ratio for the same group were measured to get information about workability changes due to the steel slag.

As it is shown below in table 5.7, concrete mixes with cement substituted by steel slag show slump reduction than the control mix.

Table 5.7: Slump of concrete specimens prepared by cement blended with steel slag

| **Sample. Number** | **Mix Code** | **w/c Ratio** | **Slump (mm)** |
|---|---|---|---|
| **1** | CN0 | 0.48 | 40 |
| **2** | CN5 | 0.48 | 30 |

### 5.3.2 Compressive Strength

For observing the performance changes due to the substitution of part of cement by steel slag in concrete production, class of concrete C-50, was prepared and tested for compressive strength at 7 and 28 days.

The test results for all samples are presented on the Appendixes. But for the purpose of discussion the summarized test results are presented in tables 5.8, and 5.9 according to the type of the specimens.

The strength of concrete produced with slag cement is strongly influenced by quality of steel slag used, the manufacturing process (blending versus inter-grinding) and the final particle size distribution of the cement.

The test results, as shown in tables 5.8 and 5.9, show that the seventh and 28th days compressive strengths of specimens with steel slag are less than that of the corresponding control specimens.

These decreases in strength mainly occur due to replacement of Portland cement with steel slag addition causing dilution of $C_3S$ and $C_2S$ which is responsible for strength.

Table 5.8: Seven days compressive strength of concrete specimens prepared by cement blended with steel slag

| **Sample number** | **Code** | **Average Compressive Strength** | |
|---|---|---|---|
| | | **7 Days** | |
| | | **Load kN** | **Strength N/mm$^2$** |
| 1 | CN0 | 808.91 | 35.96 |
| 2 | CN5 | 687.73 | 30.57 |

Table 5.9: Twenty eight days compressive strength of concrete specimens prepared by cement blended with steel slag

| **Sample number** | **Code** | **Average Compressive Strength** | |
|---|---|---|---|
| | | **28 Days** | |
| | | **Load kN** | **Strength N/mm$^2$** |
| 1 | CN0 | 1069.96 | 47.56 |
| 2 | CN5 | 981.09 | 43.61 |

# Chapter six

# Conclusions and Recommendations

## 6.1 Conclusions

Recycling of industrial wastes is one of the solutions given attention worldwide for environmental protection and for economical and sustainable use of resources. In this research, recycling of steel slag for the production of cement and concrete has been studied and the following conclusions are made:

1. According to the physical and chemical analysis of locally produced electric arc furnace slag, it was found that it has acceptable chemical and physical qualities comparing with the standard steel slag.

2. The locally produced steel slag contains a higher proportion of iron in comparison with the results with the standard method ACI 233, due to the iron purification process in Giad steel industry.
3. From the results of compressive strength, it could be seen that the compressive strength increases remarkably with increasing curing time. Also, the compressive strength decreases with an increase of the slag content, as mentioned before this may be due to the chemical composition of slag.
4. Increasing percentage of addition of steel slag to ordinary Portland cement results in general compressive strength reduction than OPC. But other properties of steel slag blended cements such as consistency, setting times, sulphate residue and soundness remain within the acceptable limits of different standards.
5. The strength test results showed that the locally produced steel slag from Giad steel industry is a good pozzolana and its strength activity index was 95.8% which is

greater than the minimum target of 90% set by the American society for testing and material.

6. According to the American society of testing and material standard (ASTM C 989 and ASTM C311), the steel slag product by Giad steel industry in Sudan was classified as a pozzolana of grade 100.
7. In concrete production replacement of 5% cement by steel slag gives comparable compressive strength as of steel slag free concrete specimens; but increasing the replacement range beyond 5% results in strength reduction.
8. The investigation revealed that replacing of cement with steel slag with 5% reduces the slump of concrete mixes.
9. The study indicates that the steel slag can be incorporated in Portland cement production.

## 6.2 Recommendations

The following recommendations could be stated:

1. This research studied the chemical analysis of local steel slag and the compressive strength in order to give conclusive evidence about the pozzolanicity of the materials concerned. However, further studies are recommended to the study the crystalline structure of slag by using X-Ray Diffraction Testing (XRD).
2. For a better utilization of slag, there must be rapid cooling of slag by air or water to gain larger amount amorphous silica which enhances slag quality.
3. The study also recommends to finely crushing of steel slag that will make it better to use as cementitious material in concrete.
4. Evaluation of environmental hazards of huge amount of steel slag in Giad Steel industry and other Steel Industries in Sudan should be studied.

5. In this study, the compressive strength was tested until the age of 28 days only, it is recommended that further studies for longer periods of time in order to get a better understanding of the long-term strength development.
6. It is recommended that this study be implemented at Aslan Cement factory which located near Giad Steel factory.

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**Appendix A:** Seven days compressive strength of concrete specimens prepared by cement blended with steel slag

| Sample number | Code | Cube number | Cube weight kg | Casting date | Age (days) | Max load kN | Strength N/mm$^2$ |
|---|---|---|---|---|---|---|---|
| 1 | CN0 | 1 | 8.025 | 13/3/2018 | 7 | 781.77 | 34.75 |
| | | 2 | 7.975 | 13/3/2018 | 7 | 794.59 | 35.32 |
| | | 3 | 8.135 | 13/3/2018 | 7 | 850.38 | 37.80 |
| Average results | | | | | | 808.91 | 35.96 |
| 2 | CN5 | 1 | 8.070 | 14/3/2018 | 7 | 714.45 | 31.75 |
| | | 2 | 7.773 | 14/3/2018 | 7 | 652.87 | 29.02 |
| | | 3 | 8.029 | 14/3/2018 | 7 | 696.05 | 30.94 |
| Average results | | | | | | 687.73 | 30.57 |

**Appendix B:** Twenty eight days compressive strength of concrete specimens prepared by cement blended with steel slag

| Sample number | Code | Cube number | Cube weight kg | Casting date | Age (days) | Max load kN | Strength N/mm$^2$ |
|---|---|---|---|---|---|---|---|
| 1 | CN0 | 1 | 8.140 | 13/3/2018 | 28 | 1071.42 | 47.62 |
| | | 2 | 7.991 | 13/3/2018 | 28 | 1095.30 | 48.67 |
| | | 3 | 8.022 | 13/3/2018 | 28 | 1043.70 | 46.39 |
| Average results | | | | | | 1069.96 | 47.56 |
| 2 | CN5 | 1 | 7.995 | 14/3/2018 | 28 | 962.61 | 42.78 |
| | | 2 | 8.020 | 14/3/2018 | 28 | 925.71 | 41.14 |
| | | 3 | 8.130 | 14/3/2018 | 28 | 1055.30 | 46.90 |
| Average results | | | | | | 981.09 | 43.61 |

## Photo attachments:

Photo (2): Sand for cement mortars

Photo (3): Mixer for cement mortars

Photo (4): Crushed stone for concrete

Photo 5: Slump measurement

Photo (6): Concrete specimens after casting

Photo (7): Compressive strength testing machine