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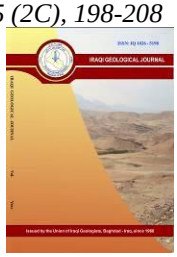

Assessment of Altawseea Ordinary Portland Cement Northern Iraq: Mineralogy, Microstructure, and Hydration

Younus M. Al-Jiboory[1] and Sawsan H. Al-Hazaa[2,*]

[1] Northern Cement Company, Ministry of Industry, Mosul, Iraq
[2] Department of Applied Geology, College of Science, Tikrit University,Tikrit, Iraq
* **Correspondence**: sawsanalhazaa@tu.edu.iq

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Abstract

The current study is an assessment of the Al-Tawsseea Cement Plant, located in the northwest of Iraq, Nineveh. This plant produces Ordinary Portland Cement grade 42.5R according to the I.Q.S No(5)/2019. Ordinary Portland Cement was chosen to determine the mineralogy using X-Ray Diffraction and Scanning Electron Microscope before and after hydration. Physical tests were carried out such as the Fineness Test (Blain) (331.4) M2/Kg, Soundness Test (Autoclave) 0.1% Compressive Strength Test (2 Days 22.35 N/mm2 and after 28 Days 47.81 N/mm2) and Setting Time Test (Initial 150 Min and Final 3:10 Hours) and all the physical tests are within the requirements of limits according to I.Q.S No(5)/2019. XRD analyses revealed that the studied cement is composed of alite, belite, ferrite, and aluminate which indicate good burning and cooling processes, while after hydration the paste is composed of CSH, CH, and Ettringite. The Scaning Electron Microscope showed that the studied Ordinary Portland Cement microstructure, characterized by an intensive intergrowth of hypidiomorphic crystals and belite (spherical to oval shape) is less but bigger than alite. After hydration, the microstructure became dense and calcium-silicate hydrate (C-S-H) formed a lathe-like shape fill the pores of the cement paste and gave the paste hardness and good compressive strength. The pore diameters in studied Ordinary Portland Cement are ranging from 9.9 to more than 15.9 µm. They look like circular or irregular shape and are randomly distributed which decrease the density, and compressive strength of the paste. According to the results of the physical test and mineralogy microstructure, we can say that it is possible to raise the grade of Ordinary Portland Cement of the Al-Tawseea Cement Plant (42.5R) by increasing the proportion of calcium in the raw materials mixture within the specification. This will lead to an increase in the value of LSF and the C3S which is responsible for increasing hardness or compressive strength.

Keywords: Ordinary Portland Cement; Cement phases; Hydration

1. Introduction

The Cement industry is considered one of the important strategic industries and the basis of civilizational and urban development. It is also one of the environmental pollutants, which is responsible for 8% of CO_2 emissions in the air (Tylor,1997). Cement is an artificial rock consisting of hydrate silicate calcium, aluminum, and iron. It is produced in different ways having many types used in special

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circumstances and places (Valencia and Baena, 2015). Portland Cement is used in the construction of houses, giant buildings¸ docks, dams, airports, bridges, roads, block factories, and others. AL-Tawsseea Cement Factory is located in the northwest of Iraq, Nineveh Governorate, Hamidat district, 25 km from the city of Mosul, between the longitude (42º 58' 17") and (42º 97' 13") east and latitudes (36º 24' 55") northern and (36º 41' 52") southern. The quarry of the Plant is located 2 km from the plant (Fig.1).

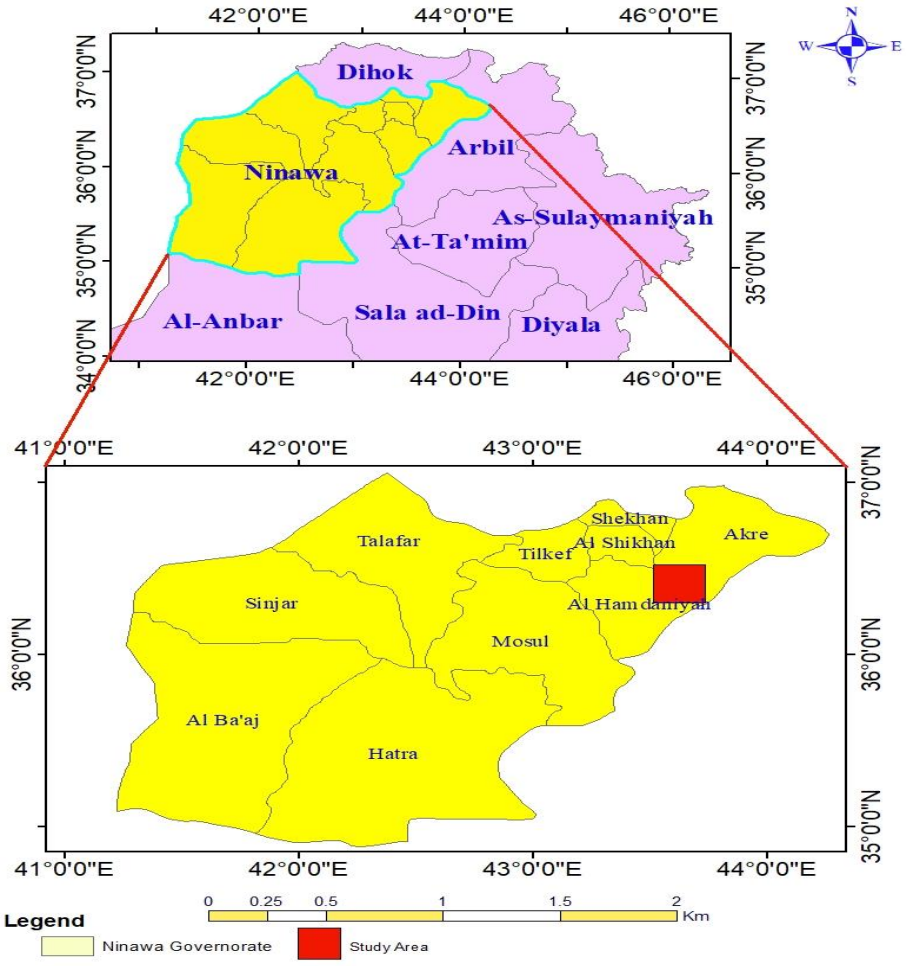

Fig.1. Location map of the study area

Al-Tawsseea Cement Plant produces Ordinary Portland Cement (O.P.C), which is manufactured by the dry method, and the cement produced in it is of a grade (42.5R) according to the European Standard. AL-Tawsseea Cement Plant was established in 1983 by (French F.C.B.) and (Japanese L.H.I). The plant operates by the dry method with pre-heating and pre-calcination and includes one production line with a design capacity of (3200) tons per day, and now the available production capacity of the plant reaches 2900 tons per day of O.P.C conforming to the EN 196 and EN 197. The plant has a quality mark certificate from the Control and holds an international quality mark certificate from the British BM- Trada Company. Portland Cement is made from raw materials from limestone or marl ($CaCO_3$ > 75%) and argillaceous rocks. It is made by mixing precisely calculated proportions of these raw materials and burning them to about 1450 Cº to produce a clinker which after being ground to a powder is mixed with 2-5% ground gypsum (Herfort and Macphee, 2019) in studied cement is added 4.5%. Al-Maadhidee (2000) studied the mineralogical, petrophysical, and chemical constraints of the physical properties of the Iraqi Portland cement and found that the evaluation of the Iraqi PC product must depend on the fineness, homogeneity and chemical, mineralogical composition

of the raw mix condition of mix ignition and conditions of cooling. Al Ali(2004) made an assessment of cement produced at Kufa cement plants and raw materials used in its manufacturing and found that the Kufa cement meets the requirements of the Iraqi Standard for Ordinary Portland Cement No.5(1984). The main aim of this study is to assess and determine the different phases of compositions or constituents and microstructures of the Al-Tawseea Ordinary Portland Cement Plant before and after adding water (cement paste). X-Ray Diffraction (XRD) and a Scanning Electron Microscope (SEM) have been used as the main equipment in the case of cement alone and after adding water to identify and interpreting changes in the mineral phases and microstructure

2. Materials and Methods

A sample of OPC was randomly selected from AL-Tawsseea Cement Plant in October 2021 weighing 50 kg. To improve the cement quality and consistency, X-Ray Diffraction was used for mineralogical analysis, wet chemical, and X-Ray Fluorescence analysis for chemical compositions in the laboratory of Al-Tawsseea Cement Plant for the following oxides: SiO_2, CaO, MgO, Al_2O_3, Fe_2O_3, SO_3, Na_2O3, K_2O. Free lime, and insoluble Residue (I.R). LOI was analyzed according to IQS No. 5 2019. Scanning electron microscopy, (SEM) was used to clarify the microstructure and the cementitious material in studied Portland cement before and after hydration.

3. Results and Discussion

Table 1 illustrates the chemical composition of the studied OPC. It is composed of four major oxides (CaO, SiO_2, Al_2O_3, $Fe_2O_3 \geq 92\%$) and some minor oxides. The main source of lime (CaO) is limestone ($CaCO_3$) while silica oxide (SiO_2) comes from clay or claystone, Iron oxide (Fe_2O_3) refers to impurities in clays. The main source of alumina oxide (Al_2O_3) is the clay, magnesia (MgO) comes from dolomite and clay, alkalis (Na_2O and K_2O) are there in clay, and sulfuric (SO_3) comes from gypsum which is added to the clinker.

Table 1. Chemical composition of AL-Tawsseea Cement (OPC)

Oxide (%)	Common Name	Abbreviation	Duda (1985)	Chemical Composition of sample (O.P.C)%
CaO	Lime	C	58-66	63.50
SiO_2	Silica	S	18-26	21.20
Al_2O_3	Alumina	A	4-12	5.78
Fe_2O_3	Iron-Oxide	F	1-6	2.25
MgO	Magnesia	M	4 max	2.80
*R_2O	Alkaline	N, K	1 max	0.19
SO_3	Sulfuric Anhydride	Š	3 max	2.12
L.O.I	Loss On Ignition	L.O.I	4 max	1.9
Total				99.74
CaO_F	Free lime	FC	----	1.60
I.R	Insoluble Residue	I.R	1.5 max	0.30

*Na_2O+K_2O

3.1. Calculations Boug's Equations and Minerals Phase in Studied O.P.C

There are four main mineral phases that formed the Portland cement: these are (C3S, C2S, C3A, and C4AF) and each phase has its own characteristics and a certain formation temperature (Taylor ,1990). Depending on the Bogue equation, the percentage of the above main phases of Portland cement can be calculated and the coefficients (L.S.F, AM, HM, SM) were calculated as well as the proportions of the cement produced were determined (Newmen and Choo(2003), Brandt(2009) (Table 2).

Table 2. Mineral phase and cement factors of Al-Tawsseea Cement (OPC)

Mineral phase and cement factors	Newman and Choo (2003)	Brandt (2009)	Mineral composition of Al-Tawseea Cement Plant
C3S	45-65	38-60	49.13
C2S	10-30	15-38	23.71
C3A	5-12	7-15	11.51
C4AF	5-12	6-18	6.84
L.S.F	66 – 102	----	91.52
SM	2.8	----	2.64
AM	2.94	----	2.56
HM	.----	----	2.17

3.1.1. Alite (C3S)

It is also called Tri Calcium Silicate (3CaO.SiO2) with minor ionic substitutions (Vikström, 2021). It is consisting of the reaction of three molecules of calcium oxide with one of the molecules of silica oxide inside the rotary kiln at a temperature of (1350-1500) Ċ (Harrisson,2019). The C3S is responsible for the initial set of concrete and reacts quickly with water. The reaction liberates the quantity of heat and is responsible for the early strength of the cement paste (Bergold et al., 2014). This reaction leads to the occurrence of some capillary cracks in concrete (Ylmén, 2013). A mathematical equation to calculate the value of C3S (Equation 1) shows that the C3S in studied cement is 49.13% and it is within an acceptable range to Newmen and Choo (2003) and Brandt (2009) (Table 2):

$$\%C3S = 4.071(\%C) - 7.6(\%S) - 6.718(\%A) - 1.43(\%F) - 2.852(\%Š) \dots\dots (1)$$

3.1.2. Belite (C2S)

The C2S phase is formed as a result of the reaction of the two molecules of calcium oxide with only one molecule of silica oxide inside the rotary kiln at a temperature of (900-1200)Ċ and it is responsible for the late hardness of concrete. The speed of its reaction with water (hydration) is slow, so the heat emitted from the reaction is low (Borštnar et al., 2020). Where the phase is called C2S or it is called Di Calcium Silicate, its chemical dye is 2CaO.SiO2, and the phase calculated from the following mathematical (equation 2) and is 23.71% (Table 2) in the studied cement which is within an acceptable range by Newmen and Choo (2003) and Brandt (2009) (Table 2):

$$C2S = 2.867(\%S) - 0.7544(\%C3S) \dots\dots\dots (2)$$

Through equation (2) that the value of C2S is inversely proportional to the value of the mineral phase C3S and directly to the value of silica oxide.

3.1.3. Aluminous phase C3A

The C3A phase is the product of the interaction of three molecules of calcium oxide with one package of aluminum oxide at a temperature ranging from (1200 – 1280)Ċ inside the rotary kiln. Its reaction with water or hydration process is very fast, so the temperature emitted from this reaction is high, and this phase is characterized by high early hardness, and the late hardness is weak (Yang et al., 2019). This phase is called Tri Calcium Aluminate and is found in clinker as a secondary phase (Yang et al., 2019). Its chemical composition is ($3CaO.Al_2O_3$), this phase can be calculated using the mathematical (equation 3) :

$$\%C3A = 2.650(\%A) - 1.692(\%F) \dots\dots\dots\dots (3)$$

From equation 3 we notice that the value of the C3A phase is directly proportional to aluminum oxide (Al2O3) and inversely proportional to iron oxide (Fe2O3) and showed that the C3A in studied cement is 11.51% which is within an acceptable range by Newmen and Choo (2003) and Brandt (2009) (Table 2):

3.1.4. Ferric Phase (C4AF)

The iron phase (C4AF) consists of the reaction of four molecules of calcium oxide with one molecule of iron oxide package at a temperature of (1250)Ċ inside the rotary kiln. It is medium, and the early and late hardness are very low compared to the previous mineral (Herfort and Macphee, 2019), and it is called Tetra Calcium Aluminate Ferrite or Celite. The value of C4AF can be calculated using the following mathematical (equation 4) and it is 6.84% within an acceptable range by Newmen and Choo (2003) and Brandt (2009) (Table 2):

$$\%C4AF = 3.043(\%F) \dots\dots\dots\dots (4)$$

This phase of C4AF is directly proportional to the increase in the percentage of iron oxide as in equation 4 and noted in (Table 2).

3.2. Cement Factors

They are the linking factors between the main oxides that form the ordinary cement or clinker, and they are determined by a certain percentage for each type of cement. These factors are (L.S.F, AM, SM, and HM). Such factors are more important in clinker than in cement because they determine the operating conditions of the kiln and fuel consumption and are related to the formation of the mineral phase and thus determine the specifications of clinker and then the cement produced (Tylor 1997, Harrisson,2019). These factors can be calculated from the following equations and all are within the limits of Newman and Choo (2003) (Table 2).

$$\%L.S.F = \frac{C - (0.7\check{S})}{2.8S + 1.2A + 0.65F} \dots\dots\dots\dots (5)$$

$$AM = \frac{A}{F} \dots\dots\dots\dots (6)$$

$$SM = \frac{S}{R} \dots\dots\dots\dots\dots (7)$$

$$R = A + F$$

$$HM = \frac{C}{S + R} \dots\dots\dots\dots\dots\dots (8)$$

From the chemical analysis results, mineral phase calculation, and cement factors shown in the above (Tables 1 and 2), the studied cement is conformed to the standards of the chemical specification (Newman and Choo,2003).

3.3. Physical Test

Physical examinations are carried out on the selected Ordinary Portland cement (O.P.C) as a sample from Al-Tawsseea Cement Plant. The results of the physical examinations were in conformity with the (EN 196), as shown in Table 3. The physical specifications are results specified by the chemical specifications for cement, so the mixture of raw materials must be selected and careful follow-up of all details of the production process in cement production because any grain the chemical specification will show its effects on the physical specifications of cement. Physical tests include Fineness Test (Blain) (331.4) M2/Kg, Soundness Test (Autoclave) 0.1%, Compressive Strength Test (2 Days 22.35 N/mm2 and after 28 Days 47.81 N/mm^2), and Setting Time Test (Initial 150 Min and Final 3:10 Hours) and all the physical tests are within the requirements of limits according to IQS No. 5 (2019) (Table 3).

3.4. X- Ray Diffraction Analysis (XRD)

The XRD diffractogram of dehydrated cement and paste (hydrated) for 28 days is shown in Fig. 2. It can be seen that the ordinary Portland cement sample which is taken from the Al-Tawsseea cement plant consists of the major alite phase (C3S - 3CaO.SiO2), the second phase is belite (C2S - 2CaO.SiO2), and little amount of aluminate (C3A - 3CaO.Al2O3), and ferrite (C4AF - 4CaO.Al2O3.Fe2O3) but these phases represent the major phases (after hydration) in the cement paste are relatively high-intensity peaks for C-S-H, C-H, portlandite, and little ettringite as seen in Fig. 3. Alite (C3S) is present at the 2 θ = 29.5º, 32.2º, 32.6º, 34.4º, 41.3º, and 51.7º; 56.6º and belite (C2S)is found at 2 θ 23.0º and 38.8º . Isteri et al. (2022) point out that some reflections of other mineral phases overlap with reflections of alite phase. Mukhopadhyay et al. (2019) conclude that "alite has 7 polymorphs as a function of temperature stabilization: 3 triclinic (T1, T2, and T3), 3 monoclinic (M1, M2, M3) and one rhombohedral". M1 and M3 are the most common alite polymorphs in the clinker. Alite is found in 2 θ at 32.0º to 32.6º and 51.0º to 51.7º (Fig. 2), indicating the M3 polymorph of alite in the studied OPC of the Al-Tawseea Plant. The rapid reaction of OPC is with water to produce a final). The characteristic peaks of C-S-H at 2 θ 32.3º,41.2º, and 63º portlandite reflections is clear at 29º,23.2º, and 62º, and $Ca(OH)_2$ are recorded at 26.7°, 38,4° (Silva et al., 2020). The most rapid and frequent reaction due to the hydrated process of OPC paste is the C-S-H phase and C-H phase and these phases are formed due to C3S and C2S phases' reactions with water as found in Fig. 3. Alite and belite hydration reactions also produce little calcium hydroxide ($Ca(OH)_2$ or (C-H) and portlandite and in the studied OPC the free lime is only 1.6% which is less than 2 according to Mtarfi et al. (2017). The C-S-H is responsible for the strength of the paste and concrete. However, the C-H crystals are less important than C-S-H in terms of increasing compressive strength and the only benefit of C-H is to keep the pH high and postpone the corrosion (Shahbazpanahi et al., 2021). Calcium sulfate reacts with aluminate (C3A) and ferrite to form ettringite, which contributes to early strength development and improves the hydration degree (Cui et al., 2018). The absorption of sulfate ions in alite phase leads to decreasing the dissolution rate of this phase which is responsible for the initial setting time and this will prolong the setting time by approximately 1 day (Minard et al., 2007). CO2 will diffuse and react to different mineral phases specially portlandite when the paste or concrete is exposed to atmospheric conditions. This reaction will delay the OPC hydration process. The formation of ettringite was completed by forming calcium carbonate (CaCO3). The high overlap of data in the XRD pattern of the studied cement paste (Fig.3) may indicate the presence of amorphous materials in the sample.

Table 3. Physical tests results of the studied OPC

Physical Test		Limits (according to I.Q.S NO. 5/2019)	Physical test results of studied OPC
Water ml			110
Setting Time	Initial	≥ 45 Min	150
	Final	≤ 10 Hours	3:10
Fineness (Blain)M^2/Kg		≥ 250 for O.P.C	331.4
Compressive strength	2 Days N/mm^2	≥10 for classes 32.5 and 42.5N ≥20 for classes 42.5R and 52.5N ≥30 for class 52.5R	22.35
	28 Days N/mm^2	≥32.5 for class 32.5 ≥42.5 for class 42.5 ≥52.5 for class 52.5	47.81
Soundness	Autoclave	≤ 0.8%	0.1%

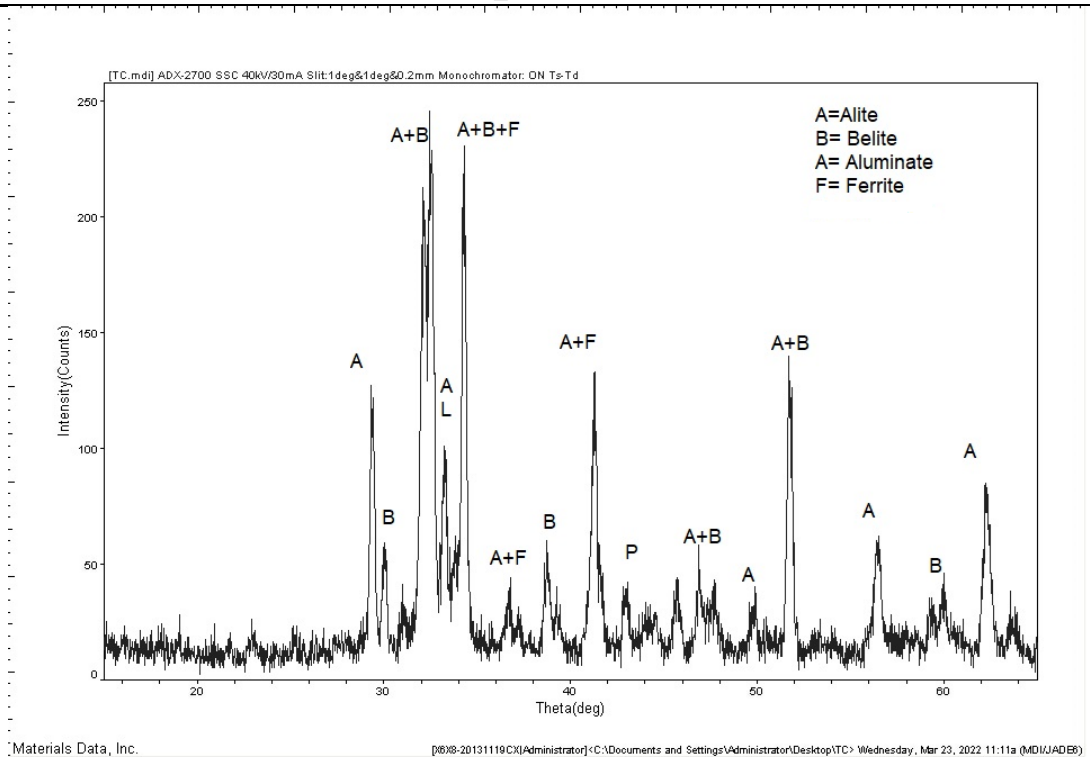

Fig. 2. XRD pattern of OPC from Al-Tawseea cement plant

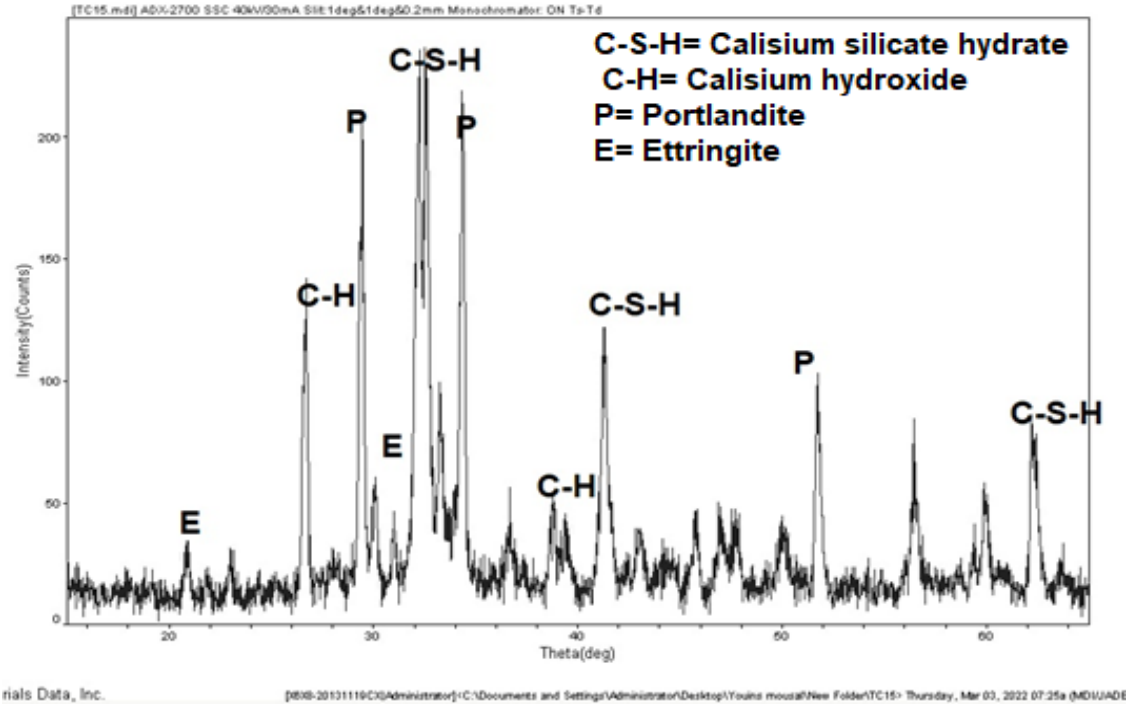

Fig.3. XRD pattern of OPC paste of Al-Tawseea cement plant (after hydration).

5. Scanning Electron Microscope (SEM)

The microstructure and morphology of Al-Tawseea Ordinary Portland Cement (OPC) before and after hydration are shown in Fig. 4. The studied OPC microstructure is characterized by an intensive intergrowth of hypidiomorphic crystals which means that some of the phases are unhedral others subhedral and some are euhedral. Allite (hexagonal or rectangular plate-like shape (Fig.4A). Belite crystals appear to be found as rounded to oval shapes (Fig.4A), and the amount of the belite crystal is lesser but bigger than the size of alite crystals and surrounded by the edge of pores. SEM micrographs (Fig.4B and C) showed that after hydration C-S-H phase was formed and filled the pores, so the microstructure became denser and there is still belite phase found in the sample (Fig.4C). This could be attributed to the hydration of C2S is a slow process, and does not pick up for many days, on the other hand, 5 – 25% of C3A reacts in the first few minutes of hydration (Lua et al., 2018). After hydration, C-S-H which is found as needle-like and reticular formed inside the pore and as flocs-like formed in the matrix are the major hydration products (Fig.3B and C). C-S-H formation during the hydration of cement is yet to be acquired, and several nucleation and growth (Shahsavari and Hwang,2018). In hydrated OPC (Fig. 3C), a white short flocs needle-like structure is visible, which indicates the presence of ettringite (Fig.4C). The particles in the samples have a wide size distribution ranging from tens to hundreds of microns as shown in the SEM images in Fig. 4. SEM results also resemble the XRD results.

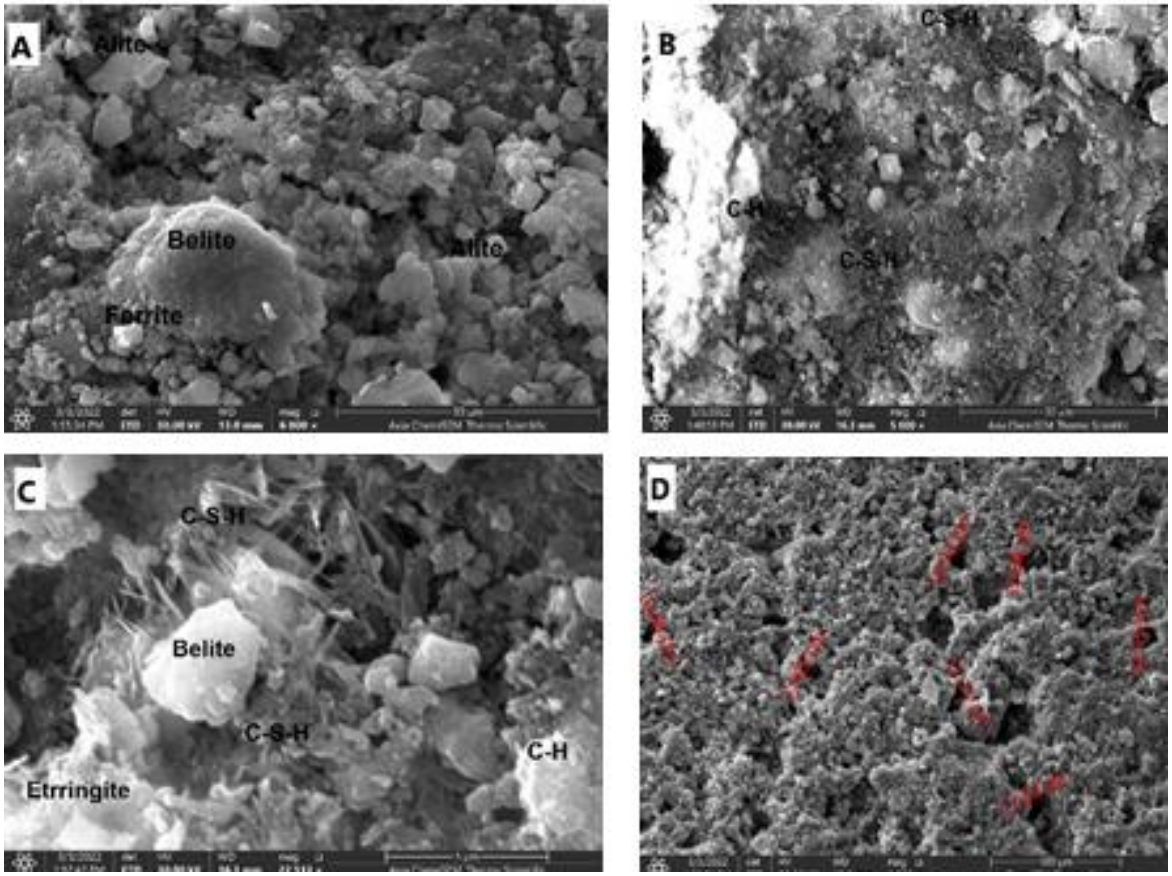

Fig. 4. SEM images of (A) Cement powder; (B) Cement Paste; (C) Cement after hydration ; (D) Pores in cement paste

The pores or capillary pores of OPC paste or concrete are one of the components that are seen under SEM. The pores and cracks are very important for water uptake in cement, and for the transport of water and gases through unsaturated and saturated cement paste (Telescaa et al., 2021). Fig. 4D shows that the pore diameters in the studied OPC is ranging between 9.9 to more than 15.9 µm. They look like circular or irregular shape and are randomly distributed which decrease the density, compressive strength of the paste, and durability of cement and concrete composites (Shahbazpanahi et al., 2021). The cracks distribute in the studied hydrated OPC in a short and un-straight manner. Liu and others (2014) classified pores into four major types: "gel pores (diameter < 10 nm), transitional pores (diameter 10–100 nm), capillary pores (diameter, 100–1000 nm), and macropores (diameter > 1000 nm)". In this study, the pore sizes of 9 µm, 10 –20 µm, and >20 µm mean the removal of water does not cause any shrinkage strains, and water held by capillary tension in small pores causes little shrinkage strains on drying (Cui and Cahyadi,2001). Pores with different diameters and connecting pores have a bad influence on concrete since the size of the pores is assumed to be detrimental to the durability for cement and concrete (Sakaia,2019). It should be noted that cement-based materials are considered as porous media which consist of different types of pores including space between the C–S–H gels as shown in Fig. 4C capillary pores, and void pores (Fig.4D) (Mehta and Monteiro,2005). The products C–S–H gels progressively fill the micro pores of the concrete, which results in a higher density and compressive strength of the concrete (Lua et al., 2018) as seen in the cement paste the of Al-Tawseew Plant and this will increase the compressive strength. According to the physical tests and mineralogy, and microstructure, we can say that its possible to raise the grade of OPC of the Al-Tawseea Cement Plant (42.5) by increasing the proportion of calcium in the raw materials mixture within the specification

(Table 2) .This will lead to increase the value of LSF and the C3S. C3S is responsible for increasing hardness or compressive strength in the concrete in two days and 28 days and this attributes to increasing the C-S-H bond after hydration. The value of LSF and C3S is directly proportional to the increase of CaO, and this will lead to a rise in the rank or grade of OPC of the Al-Tawseea Cement Plant.

6.Conclusions

The OPC in Al-Tawseea Cement Plant has four main phases (alite,belite, ferrite, and aluminate) which indicate good burning and good cooling. The presence of these phases in XRD analysis and SEM before hydration and the results of physical tests indicate that the studied OPC is of good quality and all its physical and chemical tests are within the requirements of the specification of cement. SEM analysis after hydration shows that the mineral phases were changed and C-S-H was formed filling the pores and cracks of the paste and giving the paste a good compressive strength. The alite phase is responsible for the concrete hardness, so if we could increase the C3A or alite phase by increasing the CaO in the raw materials, we could raise the grade of the OPC of the Al-Tawseea Cement Plant.

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