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Influence of GFRP Confinement of Reinforced Concrete Columns Exposed to Sodium Chloride on Microstructure and Hydration Products

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Abstract— **This paper explores the effect of sodium chloride (NaCl) on microstructure and hydration products of cylindrical column concrete and compares the results after covering it with Glass Fiber Reinforced Polymer (GFRP). 20 short RC columns were prepared with a single concrete mixture. The specimens have cured in different concentration of NaCl (0, 5%, 10%, 15%), some specimens cured directly in solution and other specimens exposed to wet and dry cycle. These specimens were tested, a first group after 28 days of curing, second group after 56 days, while the third group was repaired by GFRP after 28 day then returned to the cured environment and tested after56 day. In order to determine the main crystalline phase composition of concrete specimens and for phase identification of a crystalline material, scanning electron microscopy (SEM) morphology of microstructure concrete and X-ray diffraction (XRD) have been used. The results revealed denser microstructure with the concentration of NaCl crystalline, existence of binding chloride as free halite, chemical pound as a Friedel's salt or Physical binding held to the surface of hydration products. In addition, the corrosive compounds were observed, and the hydration product phase decreased due to concentration of curing environment. Furthermore, the results from the samples that were covered with GFRP showed that the value of the hydration products didn't change when compared with the samples that were tested after 28 days, moreover, the voids were found in them when compared with the samples exposed to the environment damage without covering it with GFRP.**

Index Terms: **chloride erosion, RC column, SEM test, XRD test, hydration products.**

I. INTRODUCTION

Chloride attack is an important factor that impacts on the durability of reinforced concrete (RC) structures, where the chloride-induced rebar corrosion is the most widespread cause of degradation in reinforced concrete [1]. If the free chloride ions present close to the reinforcement steel, they can destroy the passive oxide layer on the steel. This phenomenon leads to corrosion in the presence of moisture and oxygen followed by corrosion-induced cracks parallel to reinforcement and then by spalling and loss of concrete section [2]. This corrosion occurs in two phases - the initiation of corrosion from the beginning of penetration of chloride ions into cover concrete and the propagation of corrosion that happens when the passive layer is broken that causes expansion in steel diameter and loss of bond between the steel and concrete then the cover concrete starts spalling [3],[4],[5], However some of the chloride ion are captured by the hydration products when the chloride ion from environmental solutions penetrates into the concrete. It is called chloride binding that can exist either in the pore solution, chemical bound to hydration products such as C3A to form Friedel's salt or C4AF to form a Friedel's salt analogue or Physical binding held to the surface of hydration products as the adsorption of chloride ion to the C–S–H surfaces. The effect of chloride binding must be taken into account when studying the chloride ion transport in concrete for formation of Friedel's salt, which results in a less porous structure and slows down the transport of chloride ion, removal of chloride from the diffusion flux, thus retarding the penetration of chloride to the level of the steel, reduction of the free chloride concentration in the vicinity of the reinforcing steel which will reduce the chance of corrosion[6].While the chloride reaches to the surface of the intersection zone between the reinforcement steel and concrete, it reduces the PH value and the occurrence of corrosion. Corrosion products will form on the intersection surface and fill the voids and pores of the concrete near to the rebar. This causes the concrete to peel or crack due to the fact that the corrosion products occupy a volume larger than the size of the main rebar,

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as they cause stress on the concrete resulting in cracks. The formation of corrosion products are mainly formed by oxo-hydroxide phases on the reinforcement state such as lepidocrocite (γ-FeOOH), goethite (α-FeOOH), magnetite (Fe3O4), maghemite hydrate (γ -Fe2O3 ×H2O), hematite (Fe2O3); where the lepidocrocite is originally formed and transforms into goethite in time[7],[8].

The rate of transport chloride ions in concrete depends on its pore structure, whereas concrete is a porous material as a result of the cement hydration and the pore structure dependent on water-cement ratio, its significantly affect the formation of concrete pore structure, where the increase in W/C resulted in an increase in chloride transport, due to the formation of increased pore structure. In a study carried out by Victor Correia et al [9] the results showed increased absorption by capillarity with increased water/cement ratio, which in turn decreases by increasing the curing period, in another study according to Md. Abdullah & Md. Shafiqul Islam [10] the result showed there was a linear relation between water-cement ratio and chloride penetration, where the porosity of concrete increased with increasing water-cement ratio and at the same time, more chloride penetration occurred into specimen.

Location of the structure could mainly govern in the chloride transport behavior, where the chloride ion movement into concrete could be fostered by humidity, temperature, and chloride concentration. A higher concentration of chloride ions in seawater will increase the chloride ingress. The marine concrete structures near to seawater, especially splash and tidal zone exposed to the accumulation of chloride salts on the concrete surface, where wetting with seawater supplied penetration of chloride ions dissolved in seawater, and by drying chloride salt crystallizes due to the wet/dry cycles [11]. K. Hong & R.D. Hooton [12] found that longer drying times increase the rate of chloride ingress, that's if the wetting period is short, the entry of salt water by absorption will carry the salts into the interior the concrete and be further concentrated during drying. Moreover, High temperature and high humidity will help chloride ions to transport in concrete. Qiang et al [13] presented that temperatures alter the chloride penetration, where the penetration depth decreased with decreased temperature, and chloride permeability enhances with the growing of temperatures [14].

The concrete exposed to aggressive environments such as chloride attack gets degraded resulting in decreased structural performance of the structures sometimes in the early stages of its lifetime necessitating rehabilitation, maintenance, or replacement of parts. Therefore, the construction industry researches for new materials that offer high resistance to the effects of weather and chemical exposure such as Fiber Reinforced Polymer (FRP) composites to strengthen rehabilitate and cause corrosion resistance. The industry needs largely weather-resistant and excellent chemical resistant retrofit RC structures because of their huge advantages such as ease of application, minimum disturbance to the occupants and savings in construction cost and time. FRP composites are lightweight and relatively cheap to manufacture in addition to their advanced mechanical properties. RP composite materials are durable and have reasonable fatigue life. They have high strength-to-weight ratios and are easily adapted almost to any shape and size of structure [15].

FRP materials may be used to cause a decrease of corrosion rates in the steel reinforcement of RC and to prevent the penetration of aggressive ions by delaying formation of crystals that crack structures and cause corrosion of reinforcement [16].

Using GFRP sheet decreases the rate of uptake of the solution and the chloride ion penetration that allows the bars to remain in a passive state for longer periods. However due to imperfect application the GFRP jacket may induce a physical barrier causing incomplete concrete saturation that could lead to oxygen availability and promotion of intense corrosion activity at the rebars[17].

This work focuses on studying the microstructure and hydration product of reinforced concrete that is exposed to different concentration of chlorides ions and to observe the effects of glass fiber reinforced polymer (GFRP) wrapping on chloride penetration in reinforced concrete.
A microstructure study of the reaction mechanisms of both chloride and sodium ions together with the paste was accomplished with scanning electron microscopy (SEM), porosity and pore-size distribution and X-ray diffraction (XRD) .

II. EXPERIMENTAL WORK

A. Materials

Ben Zart cement (CEM I 42.5 N) was used according to European standard EN 197-01:2011, Tunisian standard NT 47-01:2005 and Libyan Standard Specifications 340-2009 F. Aggregate having superior properties as shown in the following Table 1has been used. Reinforced steel ES = 200000 MPa according to NF A 35 – 016 (high adhesion) with diameter 10 mm, and hoop Steel 35/24 soft rebar with diameter 3mm have been used. Fresh clean water available at the Civil Engineering Department Laboratory, National School of Engineers of Tunis, Tunis, has been used. Fiberglass E fabric that can be applied to repair boats and marine facilities has been used in the experiment undertaken. “Figure. 1,” shows the glass fiber sheet having a standard mat (450 g/m2). Sikadur_30 glue was used due to its favorable characteristics, Thixotropic2-component epoxy adhesive without solvent.

Table 1. Aggregate Properties.

Properties	**sand**	**Gravel**		**Stander**
Specific Gravity	2.6127	2.68		BS 812 : Part 2 : 1995
Water Absorption (%)	1.1	1.17		
Material finer than 75µ (%)	4	1.18		ASTM.c : 142 : 78 a

Figure 1. Glass fiber sheet.

B. Specimens preparation

The control mix Batching was done by the British method (B.S.1881 part 1970). Concrete mix ratio of C:S:G 1:2.3:2.6 by weight of concrete, 0.65 water-cement ratio were used. Mixing was done by electric mixer and the materials were thoroughly mixed in the dry state, after which water was added gradually while thoroughly mixing the concrete. Mixing of the concrete specimen continued by turning the mixture of cement, water and aggregates until the concrete was uniform in color and consistency.

The test cylinder were cast inside steel mould of size 200X100(mm).The inside of the mould was smear with oil so as to enhance easy removal of the set concrete, the fresh concrete mix with 2356.68Kg/m3 wet density, and three steel reinforcing bars with a diameter of 10 mm was used in addition to reinforcing of diameter 3 hoops and 10mm cover concrete was fully compacted by tamping rods, to remove trapped air for each batch, 20 cylinders were cast to be divided among the treatment methods. Reinforcement details are shown in the following "Figure.2,".

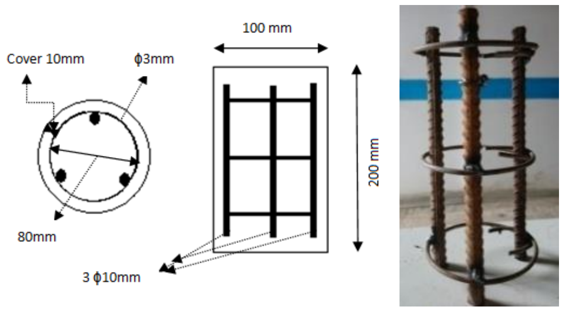

Figure 2.Reinforcement details.

C. Curing test specimens

Samples were treated and exposed to the chloride salts environment for two periods, the first group for 28days and the second for 56 days, in addition to group that repaired by GFRP after 28 day of exposure then retrain to it until tested after 56 day.

The samples were divided into two types of exposure condition, in addition to the samples that were treated in pure water. Where in pure water, 2 samples are cured of reinforced concrete columns, which are considered the reference samples, exposure directly to NaCl solution in a solution of sodium chloride with its different concentrations (5, 10, 15)%, where 3 samples are cured at each concentration, wet and dry cycles in a solution of sodium chloride with different concentrations (5, 10, 15) %, where 3 samples are treated in each concentration, in addition to subjecting these samples to wet and dry cycle, one day in the solution and two days in oven drying at a temperature of 60°C, which is gradually raised and lowered. Curing condition details in solution with different concentration of NaCl are shown in the following "Figure. 3,".

Figure 3. Curing condtion a) in solution (0,5,10,15% NaCl; b) in oven of 60˚C.

D. Repair by GFRP

After 28 days have passed since the samples were exposed to different damage conditions, one sample from each group is taken out and dried superficially from the solution. It is repaired by three layers of glass fibers and polymer GFRP as shown in "Figure. 3,". The first step prior to GFRP application was cleaning the concrete surface. Then a primer of resin was applied to the surface of the column to ensure the adhesion of the fiberglass fabric to the column. The glass fiber fabric was then placed on the resin around the column, and the resin was applied through the glass fiber fabric, where three layers of the fabric were placed on the column, and the resin was placed between each layer and the other. A roller was used to ensure that the layers were adhered to each other, forming a single fabric around the column, in addition to making sure that there weren't air bubbles between the fabric layers. and kept until it dries completely for two days and then returned to the damage environment in which it was present, until tested after another 28 day.

Figure 4. Covered column with GFRP.

III. EXPERIMENTAL TESTING

A. *SEM test.*

Scanning electron microscopy (SEM) analyses of concrete specimens were carried out at Tunisian national oil company (ETAP), Tunis .Samples of pieces of concrete are prepared by taking 1 cm^3 from the concrete cover of a reinforced concrete column that were completely dry, pictures were taken at the concrete surface that contact with the rebar. Shown in “Figure. 5,”.

Figure 5. SEM samples.

B. *X- Ray test.*

The X-ray diffraction (XRD) analyses of concrete specimens were carried out at Department of Geology, College of science, University of Tunis El Manar, Tunis. The XRD analysis was done on the powdered concrete pastes that were prepared by taking them from the completely dry concrete cover of a reinforced concrete column, were crushing and grinding to a very fine powder using the described pulverizer “Figure. 6,” shown that,. The XRD analysis was performed for diffraction angle 2h ranged between 5˚ and 70˚.

Figure 6. XRD samples.

IV. RESULTS

A. *SEM Test results*

“Figure. 7,” (a)-(c) illustrates the hydration products after cured in pure water (0%NaCl) for 28 day, while the hydration products for concrete that cured for 56 day in the condition shown in (d)-(f).

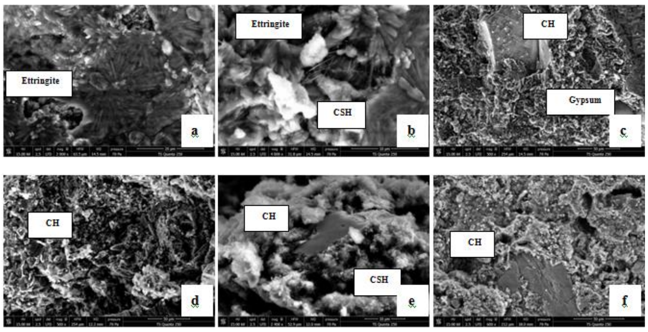

Figure 7. SEM for sample cured in 0% NaCl.

“Figure. 8,” illustrates the sodium chloride crystayled in hydration products for concrete that cured in 5% concentration of NaCl for different age 28, 56 day, While the samples that subjected to wetting and drying cycles with the same concentration 5%, in addition to the presence of salt crystals on the hydration compounds and their increase with age, the presence of iron hydroxide was observed pictures (f)(g)(j)(l) shown that, Vera et al., 2009 [7] study also supports the formation of corrosive compounds. Where the samples that were repaired with GFRP at the age of 28 days and then returned to the treatment medium to the age of 56 days as shown in (c)(j)(k)(l), the presence of voids and incomplete hydrogenation process were observed, the reason may be that covering it with GFRP prevents the arrival of the solution and the lack of complete saturation of the sample, which slows down the hydration process, this may cause air to reach the rebar and thus be a cause of corrosion, this result supports the findings of da Fonseca et al. (2015) study[17].

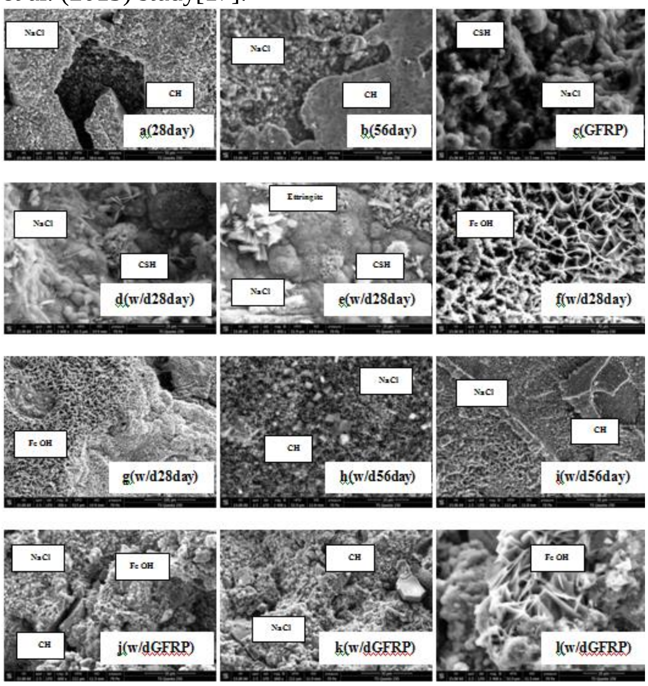

Figure 8. SEM for sample cured in 5% NaCl.

“Figure. 9,” shows the sample that were exposed to a concentration of 10 in which the formation of iron hydroxide was found as shown in (d),(e), In addition to the salt crystals of NaCl present on the hydrogenation compounds.

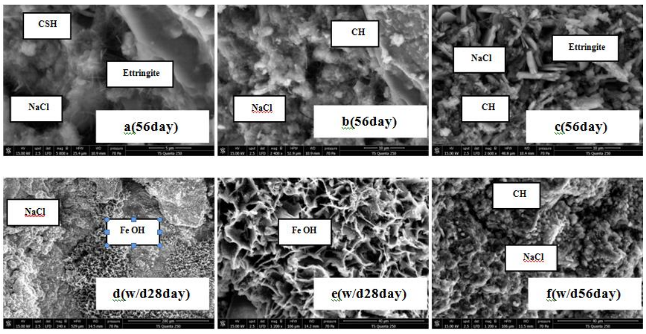

Figure 9. SEM for sample cured in 10% NaCl.

While the samples exposed to the concentration of 15 decreased the voids and the presence of salt crystals clearly if compared with other concentrations, in addition to the absence of Iron hydroxides, and the reason may be due to the lack of moisture and air access to the surface of the rebar due to the action of the chloride binding, which in turn closed the pores as shown in “Figure. 10,”.

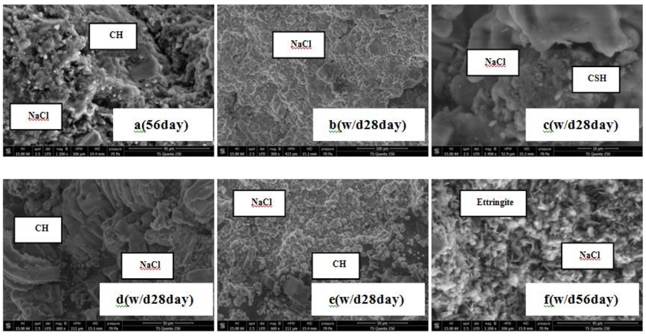

Figure 10. SEM for sample cured in 15% NaCl.

The results of Goñi et al (2013) study support this, as they also show the formation of Friedel's salts [18].

B. XRD

“Figure. 11,” shows the hydration products for the ages of 28 and 56 days for the samples who cured in pure water (0% NaCl).

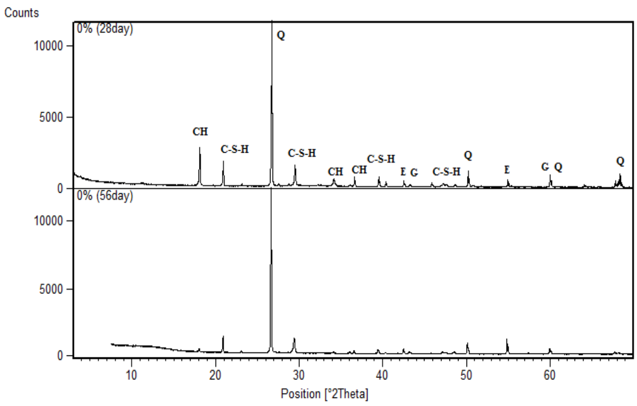

Figure 11. XRD results of samples were cure in 0% NaCl.

Where “Figure. 12,” to “Figure. 17,” shown the samples that cured in different concentrations of sodium chloride (5%,10%,15%) directly or subjected to wet and drying cycles, Where Halite compounds appeared in these samples In addition to Friedel's Salt.

At the concentration 5% of NaCl at age 56, the hydration compounds were reduced except for Ettringite and CSH increased compared with age 28 day, The reason for the rise may be due to the formation of the compound salt above the hydrogenation compounds, In addition to a slight appearance of corrosive compounds, While the same values remained as result of 28 day as the samples coated with GFRP, whether the samples were subjected to wet and drying cycles or were present directly in the solution as shown in “Figure. 12,”, “Figure. 13,”.

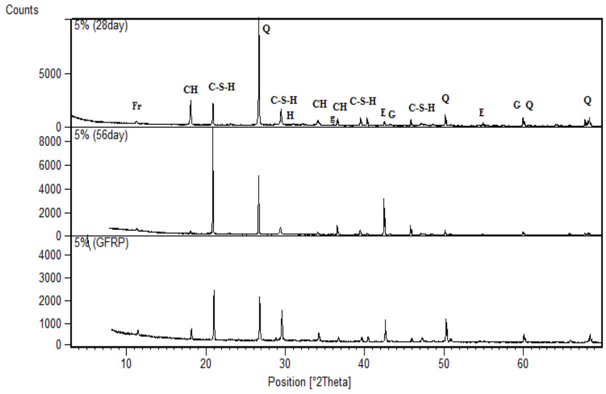

Figure 12. XRD results of samples were cure in5%NaCl.

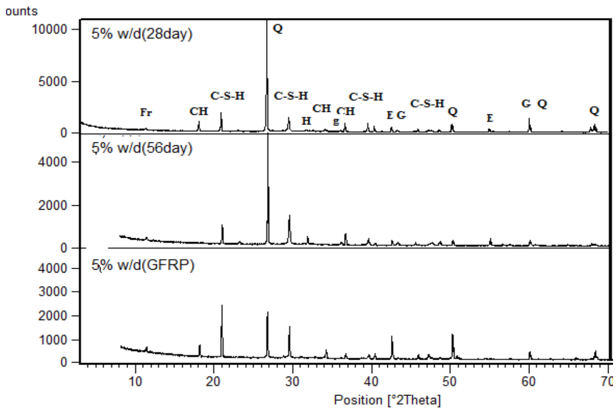

Figure 13. XRD results of samples were cure in5%NaCl(w/d).

In case of 10% concentration of NaCl the hydration products decreased at the age of 56,the reason for the decline in Portlandit may be due to the formation of Friedel's salt instead of it, as a result of the interaction of the basic components of cement with the sodium chloride salt, which results from this interaction Friedel's salt, and remained in the same state as the age of 28 as after GFRP covered, In addition to the appearance of corrosive compounds, iron hydroxides as gethite, maghemite, this means that chloride salts reach the surface of the rebar and begin to corrode that shown in “Figure. 14,”, “Figure. 15,”.

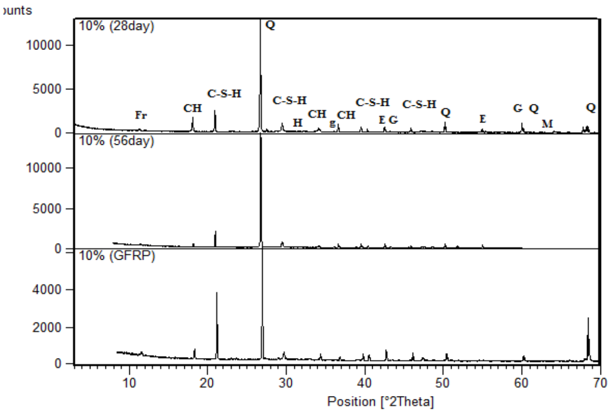

Figure 14. XRD results of samples were curein10%NaCl.

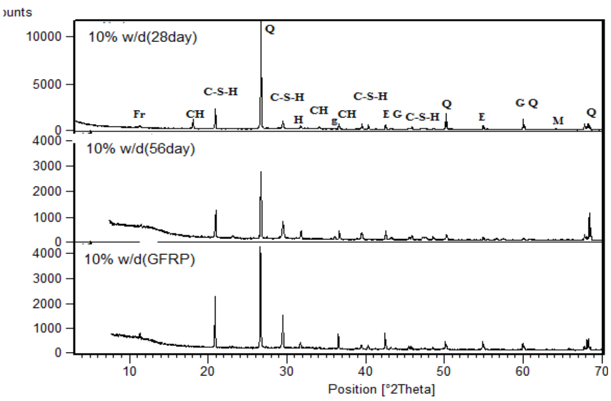

Figure 15. XRD results of samples were curein10%NaCl(w/d).

Where "Figure. 16,", "Figure. 17," shown the samples that cured in 15% concentration of NaCl, a clear rise in CSH at the age of 56.

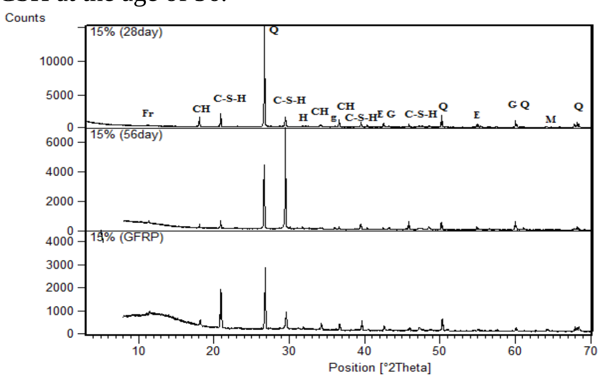

Figure 16. XRD results of samples were cure in15%NaCl.

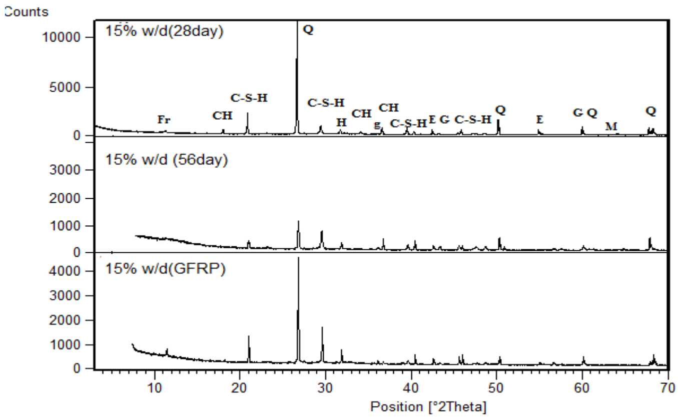

Figure 17. XRD results of samples were cure in15%NaCl(w/d).

Findings from previous studies support the finding, a study for Sun et al., 2020 [19] also showed the formation of Friedel's salts by XRD, while Vera et al., 2009 study showed corrosive compounds for XRD test [7].

V. Conclusions

After observing the experimental results, the following concluded can be drawn:

- Sodium chloride salt penetrates through concrete forming a chloride binding, where the results showed its presence as free salt in the form of halite, physically on binding to hydration products, in addition chemically pound to the formation of Friedel's salt.
- In the concentration of 10%, it was observed that iron hydroxides formed due to the chloride reaching the surface of the intersection between the reinforcement steel and concrete that mean the beginning of the corrosion. In the samples that cured in 5% concentration of NaCl the chloride salts appeared in the form of halite and Friedel salts and as binding to the hydration products. However the corrosion products found in sample that exposed to wet and dry cycle with 5% NaCl, and in a concentration of 15%, sodium chloride filled the voids and concrete pores, as compared with other concentrations, this closure prevented the access of air and moisture to the reinforcement steel and led to No erosion.
- There was nearly no change in the hydrogenation compounds between the results of the samples that were covered with GFRP after 28 days, and then returned to the damage environment again to tested after another 28 days, and the samples that were tested after 28 days of being in the damage environment, the reason may be due to the isolation of the samples from the exposed environment that led to un saturation of the samples from the solution.
- The presence of voids was observed in microstructure of concrete cover, for the samples that were covered with GFRP after 28 days, and then returned to the damage environment again to tested after another 28 days, It was compared with samples that were exposed to damage for 56 consecutive days.

- The covering of samples with GFRP, may be lead to the lack of saturation of the sample and the presence of voids that allow air penetration to occur for erosion in the rebar, where appearance of iron hydroxide compound in the sample that was repaired by GFRP.

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