

Effects of Different Types of Sulphates and Concentrations on Steel

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ABSTRACT

Steel which is indispensable in infrastructures, transportation and manufacturing has a major drawback which is its susceptibility to deterioration (corrosion) that causes economic damage. Some structural failures cannot be unconnected to steel deterioration. Different factors cause deterioration of steel and steel structures but less attention has been given to the sulphate attack based on surrounding environment as one of the factors. In this work, the effects of different types of sulphates and different concentrations of sulphate solutions on the physical properties of steel over a period of time were considered. The sulphates include Calcium sulphate (CaSO_4), Ammonium sulphate $[(\text{NH}_4)_2\text{SO}_4]$, Sodium sulphate (Na_2SO_4) and Magnesium sulphate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$) with concentrations of 1%, 3% and 5%. Weight loss analysis and physical observation were adopted to ascertain the deterioration of steel samples. Results show a progressive increase in weight loss for all the samples in the different solutions as the duration of immersion increases. With the exception of ammonium sulphate, the weight loss reduces with increasing sulphate concentration. Ammonium sulphate solution has a more significant effect on the steel samples than the other sulphate solutions. The deterioration also reflected in the colour and physical appearance of the steel samples. Hence sulphate causes deterioration in steel, therefore, appropriate precautions and prevention should be applied in constructions involving steel in a sulphate-rich environment.

KEYWORDS: steel, deterioration, sulphate attack, calcium sulphate, ammonium sulphate, Sodium sulphate, magnesium sulphate

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1. INTRODUCTION

Steel which is one of the world's most important engineering and construction material is an alloy of iron and other elements, primarily carbon that is widely used in various applications because of its high tensile strength and low cost. The base metal for steel is iron, the carbon content of steel is between 0.002% - 2.1% by weight, although these values vary depending on the alloying element. Steels are widely used in the construction of roads, railways, buildings and other common application. Large modern structures such as stadiums, telecommunication mast, skyscrapers, bridges and so on are supported by a steel skeleton, also most concrete structures in use today employ steel rebar as reinforcements. Steel is also used in variety of other construction materials such as bolt nuts and screws. Some other common application of steel includes steel pipe for pipelines offshore construction and so on.

Steel which is indispensable in infrastructures, transportation and manufacturing has a major drawback which is its susceptibility to corrosion that causes economic damage. Deterioration of steel (corrosion) has been a major problem in the construction processes and industries or for steel pipes and piles. Across all industrial sectors, the inferred costs of metal corrosion have been estimated to range between 2% and 3% of gross domestic product (GDP) in developed countries (Enning and Garrelfs, 2014)

How to cite this paper: Okere, C. E | Bertram, D. I | Okongwu, E. C | Ohaegbu O | Tom, G | Tadas-Okonkwo, A "Effects of Different Types of Sulphates and Concentrations on Steel" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-4 | Issue-4, June 2020, pp.1361-1365, URL: www.ijtsrd.com/papers/ijtsrd31481.pdf



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Different factors cause deterioration of steel and steel structures but less attention has been given to the sulphate attack as one of the factors. Hence unmindful exposure of steel to sulphate attacks exists. The knowledge of the types of sulphate together with the actual concentrations of the sulphate that result to negative effects becomes necessary.

Sulphate is a polyatomic anion with the empirical formula SO_4^{2-} (Lewis, 1916). Sulphates are salts of sulphuric acid. There are different types of sulphate salts in aqueous solution that attack the steel structures and reinforcements; some of these sulphates includes calcium sulphate, magnesium sulphate, potassium sulphate, sodium sulphate, ammonium sulphates and so on. These salts of sulphates alter the physical properties and behavior of the steel when they come in contact with the steel itself. This interaction is termed "Sulphate Attack". It is also the effect of sulphates on steel and the consequent deterioration of the steel over a period of time, affecting its strength and appearance.

Some structural failures cannot be unconnected to steel deterioration. In steel structures this corrosion alters the behavior and properties of the steel structures and failure or collapse becomes imminent. This deterioration also affects bolted connection, weakens the bolts and hence causes failure at the joint which also leads to collapse of the steel

structures. Reinforced concrete structures are not left out in this menace. Sulphates attack the concrete which spalls and breaks off, exposing the steel rebar to the same attack. Methods of determining the rate of corrosion of steel embedded in concrete is quite different from that of steel in soil medium or steel in stand-alone as could be seen from the works of some researchers although the weight loss analysis was adopted by all the researchers (Trivedi et al., 2016, Gezawa et al., 2015, Corral-Higuera et al., 2011).

The sources of the sulphate attack may differ, for example in a reinforced concrete, the attack can be in two main forms, either internal or external sulphate sources, but for steel structures, the sulphate attack is mostly of external sources. Internal sources of sulphate to the steel occurs in a situation where the sulphate ions are incorporated in the mixing and the casting of the concrete either as hydraulic cement, aggregate or sulphate-contained admixtures or the use of sulphate dissolved water in the mixing, casting and curing of the reinforced concrete. External sulphate sources are mostly due to the interaction and penetration of the sulphates in solution to the steel. The external sources of the sulphate attack are more common and are usually as a result of high sulphate soil (sulphate-rich soil) and groundwater or can be as result of atmospheric or industrial pollution. These external sources can introduce the sulphate to the steel in the following ways: the soil may contain sulphate salts in excess which may be natural nor incorporated by the use of sulphate-rich fertilizers, Also, sulphates dissolve in groundwater which is transported to foundation of structures by capillary action mostly on the foundation of structures like buildings, earth retaining walls, pile foundations, piers, overhead bridges, underground structures and storage tanks and so on. Also, other external sources of sulphate to the steel are through sulphate dissolved fluids such as sea waters, industrial wastes, water treatment plant wastes and so on. Sea water pose a greater source of external sulphate attack since sea water contains lots of dissolved salts in them, including salts of sulphates. However, there are still other sources of sulphate attacks like use of sulphate-contained paints and finishes.

2. Materials And Methods

2.1. Materials

The following materials were used in this work:

- Salt of sulphates: These sulphate salts are a white powdery substances which were used in the preparation of the sulphate solutions. They include
 - A. Calcium Sulphate (CaSO_4): The anhydrous form of calcium sulphate was purchased with molar mass of 136.14g/mol, and a percentage purity of 96%. It is whitish and powdery in nature.
 - B. Magnesium Sulphate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$): The monohydrate form of the magnesium sulphate was purchased, with a molar mass of 138.28g/mol and a percentage purity of 90%. The magnesium sulphate is crystalline (granular) in nature and whitish in colour.
 - C. Sodium Sulphate (Na_2SO_4): The anhydrous form of sodium sulphate was used in the experiment, with a molar mass of 142.04g/mol and a percentage purity of 99%. Sodium sulphate (anhydrous) is whitish in colour and powdery in nature
 - D. Ammonium Sulphate [$(\text{NH}_4)_2\text{SO}_4$]: The anhydrous form of ammonium sulphate was used, with a molar

mass of 132.14g/mol and a percentage purity of 99% (minimum assay). This sulphate salt is in form of a whitish crystals (granules)

- Distilled water: Distilled water is the water that has had many impurities removed though distillation. This distilled water is efficient for preparations of sulphate solutions to ensure a pure aqueous solution free of impurities. Twenty (20) litres of this distilled water was used in preparing the sulphate solutions of different sulphate salts and different concentrations of the sulphates.
- Steel samples: Structural steel section and 8mm thick mild steel reinforcement bar were used.

2.2. Methods

The following methods were adopted:

2.2.1. Preparation of the sulphate solutions

The sulphate solutions of calcium sulphate, magnesium sulphate, sodium sulphate and ammonium sulphates to a concentration of 1%, 3% and 5% for each of the sulphate salt for a volume of 600ml were prepared using percentage by weight method. The prepared different solutions were poured into transparent airtight containers.

2.2.1. Preparation of the steel sample

The steel samples were cut to their appropriate sizes so they can fit into the airtight containers housing the prepared sulphate solutions comfortably. The structural steel plate was cut into a rectangle of size 40mm x 20mm. A 3mm diameter hole was drilled on the sample for passing the rope used for suspending the sample in the sulphate solution. The 8mm thick mild steel was cut to a size of 40mm x 8mm. The samples were cleaned (using pickling method), rinsed with alcohol and dried to make them free from impurities that may affect the results. A total of 60 samples of the mild steel of size 40mm x 8mm and a total of 60 samples of steel plate of size 40mm x 20mm were prepared.

2.2.3. Weight loss analysis

This was used in determining the rate of corrosion. The samples were weighed on the analytical 4-digit electronic weighing balance to obtain their initial weight. For each sulphate solution, the samples were immersed for a period of 7days after which they were removed from the solution, washed with alcohol, brushed to scrub away the corrosion products, dried and reweighed. This procedure and activities on the seventh day was repeated on the 14th day, 21st day, 28th day and the 35th day for other samples.

2.2.4. Physical observation of samples

During the preparation of solutions and weight loss analysis, physical observations of the solution and the samples were made.

3. Results and Discussions

3.1. Results

The results of the experimental investigations are presented on **Table1 to 12**. The results of the physical observation of solution are as stated. The salt of calcium sulphate (CaSO_4) is partially soluble in water, as some of the solute particles can be seen from the transparent air-tight container. Magnesium sulphate, Ammonium sulphate and Sodium sulphate are very soluble in water because a very clear solution was obtained after the preparation. The colour of the samples changed to

brownish red and the appearance looked rusty after immersion. The solutions that were crystal clear before immersion turned to brownish colour and blackish brown.

3.2. Discussions

The weight loss analysis was the method adopted in estimating the rate of deterioration (corrosion) of the steel samples. This method is simple with no sophisticated instrument required. A direct measurement is obtained with no theoretical assumption or approximations. A close look at the tables shows the difference between the initial weight before immersion in solution and the final weight on removal from solution. The difference gives the weight loss of the sample and it signifies that deterioration has occurred. The tables show a progressive increase in weight loss for all the samples in the different solutions as the duration of immersion increases. Surprisingly, as the concentration percentage of the sulphate solution increased, the weight loss of 95% of the samples reduced. This occurred in all the sulphate solutions except that of ammonium. Ammonium sulphate solution has a more significant effect on the steel sample than the other sulphate solutions. The rate of attack of the sulphate solutions on both the 8mm thick mild steel reinforcement bars and structural steel plate are similar because the weight loss values are within close range.

4. Conclusion

- Steel samples were subject to different concentrations (1%, 3% & 5%) of sulphates namely: Calcium sulphate (CaSO_4), Ammonium sulphate $[(\text{NH}_4)_2\text{SO}_4]$, Sodium sulphate (NaSO_4) and Magnesium sulphate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$), to determine their effects on steel over a period of time.
- Weight loss analysis and physical observation were adopted to ascertain the deterioration of steel samples.
- There is a progressive increase in weight loss for all the samples in the different solutions as the duration of immersion increases.

- As the concentration percentage of the sulphate solution increased, the weight loss of 95% of the samples reduced. This occurred in all the sulphate solutions except that of ammonium.
- The deterioration also reflected in the colour and physical appearance of the steel samples.
- Ammonium sulphate solution has a more significant effect on the steel samples than the other sulphate solutions.
- The rate of attack of the sulphate solutions on both the 8mm thick mild steel reinforcement bars and structural steel plate are similar because the weight loss values are within close range.

Acknowledgements

The author hereby acknowledges the following persons for their contributions to this research: Messrs Okongwu, E.C, Ohaegbu O., Tom, G. and Tadas-Okonkwo, A.

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Table1. Initial weight, final weight and the weight loss for Calcium sulphate solution 1% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 16.0190 | 16.0082 | 0.0208 | 10.6862 | 10.6654 | 0.007 |
| Day 14 | 15.7196 | 15.6895 | 0.0071 | 12.3237 | 12.3166 | 0.0254 |
| Day 21 | 15.4085 | 15.3666 | 0.0306 | 11.2952 | 11.2646 | 0.0383 |
| Day 28 | 15.1433 | 15.0891 | 0.0435 | 11.5809 | 11.5374 | 0.0509 |
| Day 35 | 15.4298 | 15.3685 | 0.0788 | 11.0485 | 10.9697 | 0.0567 |

Table2. Initial weight, final weight and the weight loss for Calcium sulphate solution 3% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 16.1515 | 16.1445 | 0.0095 | 11.1598 | 11.1503 | 0.0108 |
| Day 14 | 15.5501 | 15.5247 | 0.0214 | 11.3038 | 11.2824 | 0.0175 |
| Day 21 | 15.8302 | 15.7919 | 0.0379 | 12.8388 | 12.8009 | 0.0281 |
| Day 28 | 14.8100 | 14.7591 | 0.0362 | 11.2572 | 11.2210 | 0.0574 |
| Day 35 | 14.9363 | 14.8796 | 0.0692 | 11.4344 | 11.3652 | 0.0307 |

Table3. Initial weight, final weight and the weight loss for Calcium sulphate solution 5% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 14.9628 | 14.9520 | 0.0108 | 11.0006 | 10.8900 | 0.0116 |
| Day 14 | 15.2050 | 15.1875 | 0.0175 | 11.3830 | 11.3614 | 0.0246 |
| Day 21 | 15.6838 | 15.6557 | 0.0281 | 11.2895 | 11.2632 | 0.0263 |
| Day 28 | 15.6598 | 15.6024 | 0.0574 | 11.7809 | 11.7255 | 0.0554 |
| Day 35 | 15.8887 | 18.8580 | 0.0307 | 11.6714 | 11.6270 | 0.0444 |

Table4. Initial weight, final weight and the weight loss for Magnesium sulphate solution 1% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 15.7083 | 15.7017 | 0.0071 | 11.8716 | 11.5051 | 0.3665 |
| Day 14 | 15.6875 | 15.6587 | 0.0288 | 11.0225 | 10.9973 | 0.0252 |
| Day 21 | 15.6006 | 15.5535 | 0.0471 | 11.5189 | 11.5009 | 0.0180 |
| Day 28 | 15.1726 | 15.1122 | 0.0604 | 10.9377 | 10.8835 | 0.0542 |
| Day 35 | 14.9979 | 14.9241 | 0.0738 | 11.1830 | 11.1106 | 0.0724 |

Table5. Initial weight, final weight and the weight loss for Magnesium sulphate solution 3% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 14.9905 | 14.9819 | 0.0086 | 10.9926 | 10.9767 | 0.0159 |
| Day 14 | 15.6954 | 15.6733 | 0.0221 | 11.2588 | 11.1608 | 0.0980 |
| Day 21 | 15.9720 | 15.9376 | 0.0344 | 11.1842 | 11.1037 | 0.0805 |
| Day 28 | 15.6761 | 15.6169 | 0.0592 | 12.3371 | 12.2934 | 0.0437 |
| Day 35 | 15.9124 | 15.8536 | 0.0588 | 10.6844 | 10.6278 | 0.0566 |

Table6. Initial weight, final weight and the weight loss for Magnesium sulphate solution 5% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 15.8230 | 15.8124 | 0.0102 | 10.2786 | 10.2674 | 0.0112 |
| Day 14 | 14.8391 | 14.5199 | 0.0192 | 10.8584 | 10.8382 | 0.0202 |
| Day 21 | 15.2800 | 15.2505 | 0.0295 | 11.0228 | 10.9951 | 0.0277 |
| Day 28 | 15.0071 | 14.9559 | 0.0512 | 11.2462 | 11.1990 | 0.0472 |
| Day 35 | 15.7942 | 15.7394 | 0.0548 | 11.4892 | 11.4361 | 0.0531 |

Table7. Initial weight, final weight and the weight loss for Sodium sulphate solution 1% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 14.7148 | 14.6943 | 0.0205 | 11.0715 | 11.0665 | 0.0050 |
| Day 14 | 15.2985 | 15.1235 | 0.1750 | 11.9904 | 11.9669 | 0.0235 |
| Day 21 | 14.9565 | 14.9009 | 0.0556 | 11.2245 | 11.1920 | 0.0325 |
| Day 28 | 15.5001 | 15.2653 | 0.2348 | 11.4103 | 11.3620 | 0.0483 |
| Day 35 | 15.1286 | 15.0044 | 0.1242 | 11.5337 | 11.4750 | 0.0587 |

Table8. Initial weight, final weight and the weight loss for Sodium sulphate solution 3% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 14.9652 | 14.9641 | 0.0011 | 11.4856 | 11.4839 | 0.0017 |
| Day 14 | 14.8668 | 14.8551 | 0.0117 | 11.6703 | 11.6593 | 0.0110 |
| Day 21 | 15.7093 | 15.6856 | 0.0237 | 11.4240 | 11.3815 | 0.0425 |
| Day 28 | 15.0650 | 15.0221 | 0.0429 | 11.1833 | 11.1592 | 0.0241 |
| Day 35 | 15.7856 | 15.7430 | 0.0426 | 10.1650 | 10.1357 | 0.0293 |

Table9. Initial weight, final weight and the weight loss for Sodium sulphate solution 5% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 15.3788 | 15.3758 | 0.0030 | 11.6595 | 11.6484 | 0.0111 |
| Day 14 | 14.9740 | 14.9645 | 0.0095 | 11.6890 | 11.6826 | 0.0064 |
| Day 21 | 15.5526 | 15.5348 | 0.0178 | 10.7082 | 10.6925 | 0.0157 |
| Day 28 | 15.7303 | 15.6933 | 0.0370 | 10.4505 | 10.4308 | 0.0197 |
| Day 35 | 15.7696 | 15.7247 | 0.0449 | 10.3621 | 10.3384 | 0.0237 |

Table10. Initial weight, final weight and the weight loss for Ammonium sulphate solution 1% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 14.7853 | 14.7555 | 0.0298 | 10.7806 | 10.6627 | 0.1179 |
| Day 14 | 14.9867 | 14.9363 | 0.0504 | 10.7195 | 10.5882 | 0.1313 |
| Day 21 | 15.3173 | 15.2158 | 0.1015 | 11.1869 | 11.1656 | 0.0213 |
| Day 28 | 15.7160 | 15.5975 | 0.1185 | 11.1102 | 11.0729 | 0.0373 |
| Day 35 | 15.8890 | 15.7726 | 0.1164 | 11.2953 | 11.0968 | 0.1985 |

Table11. Initial weight, final weight and the weight loss for Ammonium sulphate solution 3% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 15.0403 | 15.0171 | 0.0232 | 11.4052 | 11.3869 | 0.0183 |
| Day 14 | 15.5020 | 15.4548 | 0.0472 | 11.0740 | 11.0491 | 0.0249 |
| Day 21 | 15.1384 | 15.0939 | 0.0445 | 11.5683 | 11.4145 | 0.1538 |
| Day 28 | 15.2892 | 15.1495 | 0.1397 | 11.5991 | 11.3881 | 0.2110 |
| Day 35 | 15.9264 | 15.8335 | 0.0929 | 11.5919 | 11.2942 | 0.2977 |

Table12. Initial weight, final weight and the weight loss for Ammonium sulphate solution 5% concentration

| DURATION | 8MM THICK MILD STEEL | | | STRUCTURAL STEEL SECTION | | |
|----------|----------------------|------------------|-----------------|--------------------------|------------------|-----------------|
| | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] | INITIAL WEIGHT [g] | FINAL WEIGHT [g] | WEIGHT LOSS [g] |
| Day 7 | 15.4927 | 15.4644 | 0.0283 | 10.5665 | 10.5475 | 0.0190 |
| Day 14 | 15.1248 | 15.0796 | 0.0452 | 11.7548 | 11.7205 | 0.0343 |
| Day 21 | 15.2612 | 15.2099 | 0.0513 | 12.7173 | 12.6456 | 0.0717 |
| Day 28 | 15.6924 | 15.5553 | 0.1371 | 11.4377 | 11.2727 | 0.1650 |
| Day 35 | 14.8268 | 14.6683 | 0.1585 | 11.4482 | 11.2220 | 0.2260 |



Figure1. Deteriorated/ corroded structural steel and steel sections in sulphate solutions characterized by colored solution