

**Republic of Iraq** Ministry of Higher Education and Scientific Research University of Mosul College of Petroleum and Mining Engineering

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#### **"STUDY OF THE EFFECT OF TWO TYPES OF COATING ON THE CORROSION BEHAVIOR OF METALS IMMERSED IN SULFURIC ACID (H<sub>2</sub>SO<sub>4</sub>)** AND QAYARA OIL AS A CORROSIVE MEDIUM"

A Project Submitted As Partial Fulfillment For The Requirements of The Degree of Bachelor of Science In Mining Engineering

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﴿وَقُلِ اعْمَلُوا فَسَيَرَى اللَّهُ عَمَلَكُمْ وَرَسُولُهُ وَالْمُؤْمِنُونَ وَسَتُرَدُّونَ إِلَىٰ عَالِمِ الْغَيْبِ وَالشَّهَادَةِ فَيُنَبِّئُكُمْ بِمَا كُنْتُمْ تَعْمَلُونَ ﴾

صدق الله العظيم سورة التوبة (105)

#### الاهداء

إلى من بلغ الرسالة وأدى الأمانة. ونصح الأمة إلى نبي الرحمة ونور العالمين "سيدنا محد (صل الله عليه وسلم)" إلى من كللهم الله بالهيبة والوقار إلى من علمونا العطاء بدون انتظار.. إلى من نحم لأسمائهم بكل افتخار ... نرجو من الله ان يمد في اعمار هم لنرى ثمار ها قد حان قطافها بعد طول انتظار وستبقى كلماتهم نجوم نهتدي بها اليوم وفي الغد وإلى الأبد

وفي الختام الحمد لله تعالى الذي وفقنا في تقديم هذا البحث، وها هي القطرات الأخيرة في مشوار هذا البحث وقد بذلنا كل الجهد والبذل لكي يخرج هذا البحث في هذا الشكل . مشوار هذا البحث وقد بذلنا كل الجهد والبذل لكي يخرج هذا البحث في هذا الشكل ..

#### **Abstract**

Metals reliability is becoming more important in our society especially when safety is compromised ,for example oil and natural gas are transmitted across continents through high pressure pipelines that must operate for decades without failure so that neither groundwater nor air is polluted therefore corrosion management became critical in petroleum industry. Thus in this research we will study several corrosion resistance after improving its properties to resist corrosion by using the treated samples by coating epoxy.

At the end of this study, we found a great improvement in corrosion resistance for sulfur and crude oil for the treated specimens compared to the untreated specimens .

And the weight loss of each metal increases slightly with increasing H2SO4 concentration.

Subject	Page	
Abstract	Ι	
Table of content	II	
Lest of figures	III	
Chapter one: INTRODUCTION	1	
1.1 Background	1	
1.1.1 Effects resulting	2	
1.1.2 How to prevent it	2	
1.2 Sulfuric acid	3	
1.2.1 Preparation of sulfuric acid	3	
1.2.2. Sulfuric acid uses	3	
<b>1.2.3.</b> Physical properties of sulfuric acid	4	
<b>1.2.4.</b> Chemical properties of sulfuric acid	4	
1.3 material selection	4	
1.3.1 Aluminum	5	
1.3.2. CK-45	5	
1.3.3. Copper	5	
1.4 .Resin Epoxy:	5	
1.5 Reasons for study corrosion	6	
1.6 Project objective	8	
<b>1.2.1 LITERATURE REVIEW</b>	9	
1.2.2 Summary	11	
Chapter Two: Experimental Work		
2.1. Introduction	13	
2.2. The Experimental Plan Work Can Be Summarized	13	
Below		
2.3 Preparation of Specimens	14	
2.4 The Preparation of Sulfuric Acid for Corrosion Test	16	
2.5 starting the crude oil Corrosion Test	19	
CHAPTER 3: Results and Discussion		
3.1 Introduction	23	
3.2. The description and analyze	23	
CHAPTER4: CONCLUSIONS & RECOMMENDATIONS		
4.1 Conclusion	28	
4.2 Future works	28	
REFERENCES	29	

#### Table of content

#### **Table of Figures**

NO	Figure	Page
1-1	corrosion of piping	2
2-1	coating of selected metals by (Epoxy)	13
2-2	shape of Specimen	14
2-3	CNC Machine	15
2-4	Properties of The Sulfuric Acid	16
2-5	Shows uncoated Specimens into Sulfuric Acid	17
2-6	Shows the Specimens into 5% Sulfuric Acid	18
2-7	Shows the Specimens into 10% Sulfuric Acid	19
2-8	Shows the Specimens into The Qayara oil	20
2-9	Shows the Corrosion of The Specimens	21
3-1	shows the weight loss of aluminum over time	23
3-2	shows the weight loss of copper over time	24
3-3	shows the weight loss of copper over time	24
3-4	Shows the Weight Losses of Aluminum Alloy	25
	Specimens with The Time	
3-5	Shows the Weight Losses of Brass Alloy Specimens	26
	with The Time	

## CHAPTER 1 INTRODUCTION

#### **INTRODUCTION**

#### **1.1 Background**

**Corrosion** Defined as destructive, irreversible damage and degradation, the destruction of tissues and materials due to a chemical or electrochemical reaction, almost all environments can undergo corrosion to some ex tent, the corroded state being the most stable state of matter.<sup>[1]</sup>

Corrosion generally occurs as a result of chemical reactions (mainly oxidation). This occurs when a chemical gas or liquid attacks an exposed surface (usually metal). Warm temperatures, acids, and salts accelerate this process. Corrosion products (such as rust and patina) usually form and protect the surface. Removing these deposits exposes the surface again, and erosion may occur again. Some materials naturally resist this, and we can protect other materials with paint<sup>[2]</sup>

Corrosion is an electrochemical reaction that appears in several forms, such as chemical corrosion and atmospheric corrosion. When acidic substances (such as water) come into contact with metals (such as iron or steel), the corrosion process begins.

Iron (Fe) particles are exposed to oxygen and moisture (steam or immersion). When steel is exposed to water, the iron particles lose acidic ions in the water. The iron particles then oxidize, resulting in the formation of  $Fe^{++}$ . This causes two electrons to escape and flow to another area. Of steel known as the cathode.

Oxygen causes electrons to rise and form hydroxyl ions (OH). Hydroxyl ions react with FE<sup>++</sup> to form hydrated iron oxide (FeOH), commonly known as rust. Now the iron particles are damaged, and a corrosion pit is formed. The corrosion product is called (rust).

Corrosion is possible in any case, depending on the environment in which the metal is. However, since atmospheric corrosion is so widespread, effective precautionary measures are recommended when it comes to preventing it.<sup>[3]</sup>



Figure (1-1) corrosion of piping

#### **1.1.1 Effects resulting**

The annual collateral cost of metal corrosion is estimated at more than \$2 trillion. Poorly planned projects can lead to unusable buildings due to corrosion. This is a waste of natural resources, not to mention safety concerns, loss of life, other additional costs and of course, reputational damage, experts believe. From 25 to 30% of corrosion can be avoided by applying appropriate protection steps.<sup>[4]</sup>

#### 1.1.2 How to prevent it

There are several effective ways to prevent corrosion, including:

- Use of paint: The outer layer surrounding the metal surface helps protect the metal from corrosion. The effectiveness of the paint in protecting the metal depends on the environment to which it is exposed. The aim of the paint process is to keep the metal dry and avoid exposure to moisture.
- Use of non-corrosive metals, such as stainless steel or aluminum.
- Keep the metal surface clean and dry.
- Use of drying agents.
- Laying a layer of stone, for example limestone, with underground pipes.<sup>[5]</sup>

#### **1.2 Sulfuric acid**

A strong, inorganic, chromatic heavy oily acid. It is composed of sulfur, hydrogen and oxygen. It ionizes quickly. The concentrated acid is called vitriol, and is used as a drying agent and oxidizing agent. In sulfuric acid there are two replaceable hydrogen atoms (dibasic), so it is both normal and acidic salts. It is a stable acid of important chemicals, used to manufacture dyes, drugs, explosives, fertilizers, batteries and petroleum refining <sup>[7]</sup>.

#### **1.2.1 Preparation of sulfuric acid**

The process of preparing sulfuric acid depends on sulfur and sulphates, which are used as raw materials. It also needs high purity as a result of the intense chemical activity of the acid. Sulfuric acid can be prepared either in the laboratory or on an industrial scale.

Sulfuric acid is prepared industrially in two ways, namely the lead chamber process and the contact process.<sup>[8]</sup>

#### 1.2.2. Sulfuric acid uses

- 1) In the manufacture of fertilizers
- 2) The production of steel and iron
- 3) It is used in chemical manufacturing industries
- 4) In the petroleum refining
- 5) It is used to produce phosphoric acid

6) It is used as a cleaning agent in industries to remove rust from steel and iron

7) Used as a catalyst for the conversion of ox cyclohexanone into caprolactam used in the nylon industry

8) It is used in lead-acid batteries as an electrolyte

9) It is used in making ammonium sulfate

10) It is used in storage batteries <sup>[10]</sup>

#### 1.2.3. Physical properties of sulfuric acid

Sulfuric acid is a thick, colorless, oily liquid.

It has a specific gravity of 1.84 at 298 K.

The boiling point of the acid is 611 K. The higher boiling point and thickness of this chemical are attributed to hydrogen bonding.

This powerful chemical reacts with water and releases a very large amount of heat. Thus, you should never add water to H2SO4. Instead, the acid should be added to the water slowly with appropriate stirring.

#### 1.2.4. Chemical properties of sulfuric acid

Sulfuric acid is a strong dibasic acid. Also, it is protein and ionizes in two phases in the aqueous solution.

This chemical is highly corrosive, reactive, and soluble in water. It has a very high oxidizing power and thus acts as a strong oxidizing agent.

It has very low volatility. For this reason, it plays a role in the preparation of more volatile acids than its comparative salts.

Concentrated sulfuric acid is a very strong drying agent. Thus, this chemical is used to dry many wet gases that do not react with the acid.

In addition, it expels water from natural mixtures such as starches.

Since it is a good oxidizing agent, it can oxidize both metals and nonmetals. Moreover, it itself reduces sulfur dioxide. <sup>[11]</sup>

#### **1.3 material selection**

It is very important carefully selecting the material of construction of pipes use for molten sulfur handling because it has an influence on all the manufacturing operations, including machining. The machinability of the pipe depends directly on the material used to manufacture the pipe, and for each given material, specific precautions must be taken in order to ensure goodquality machining <sup>[4].</sup>

Some of these materials:

#### 1.3.1 Aluminum

Aluminum is very widely used in the industry. Aluminum pipes are inexpensive, easy to form and assemble. They are also light and corrosion-resistant, making them a natural choice in the aeronautics, transport and construction sectors. So it may use as material of construction of sulfur handling pipes.

#### 1.3.2. CK-45

is a medium strength steel with good machinability and excellent tensile properites, ck-45 round steel is generally supplied in the black hot rolled or occasionally in the normalised condition, with a typical tensile strength range 570-700 Mpa and Brinell hardness range 170-210 in either condition.

#### 1.3.3. Copper

it is malleable, ductile, and an extremely good conductor of both heat and electricity. It is softer than zinc and can be polished to a bright finish.

#### 1.3.4. Bronze

due to its corrosion resistance and unique coloring , bronze is commonly used in the manufacture of coins, hardware mounts, and it is melting point of 950 degree Celsius.

#### 1.3.5. Brass

compared to bronze, Malleability and formability brass is more malleable, brass has a melting point of high melting point approximately 900 Celsius.

#### **1.4 .Resin Epoxy**:

A chemical substance that is considered one of the types of thermosetting plastics, with two-component, A primer (resin) and a hardener (hardener), which are highly adhesive and resistant to friction and chemicals, whether they are acids, bases or solvents, as an insulating layer is formed when it dries, used as a coating or mortar or adhesive, the most productive types of epoxy resin are those resulting from the reaction between the two chemicals epichlorohydrin and bisphenola.

#### 1.5 . Reasons for study corrosion

We study corrosion because of the damage causes, which include the following:

1- Dimensional change and loss of mechanical properties: Corrosion leads to a loss of weight due to the dissolution of the metal and consequently to its dimension change. Therefore, corrosion Allowance is often given when it is present and at design. These areas are thicker in the medium in which the wear rates are higher than in those in which the wear rates are low. Dimensions of the metal part due to corrosion have an impact on mechanical properties, as they are less tolerant to external loads, i.e. increasing their plastic deformation and Elastic Deformation. The use of metal in corrosive media causes low values for many mechanical properties, especially fatigue strength and cracks that lead to fast fracture.

2- Appearance: The appearance of the metal is highly affected when it is corroded, as the metal always looks bad. Therefore, corrosion-resistant metals such as aluminum or stainless steel should be used in place of carbon steel, as building materials such as windows and materials, especially in the facades of external buildings, and the good appearance of these materials is due to their resistance to air corrosion. As for metals with weak resistance to corrosion, they are coated with different types of coatings to improve their appearance by reducing their wear.

3- Economic damages due to preventive measures: The economic damages resulting from corrosion are numerous and important, as this failure often causes productive facilities to stop work unprogrammed, and the corresponding additional unforeseen economic costs. Likewise, the occurrence of corrosion leads to a high cost of periodic maintenance, as in many cases it is necessary to replace the damaged metal part with another new part. There are many examples that indicate that choosing a relatively high-cost material but with good economic resistance to

corrosion is preferable to using a particular material that is cheaper but is subject to rapid damage due to corrosion. What requires that it be changed periodically, and in both cases, it is observed that corrosion causes economic damage due to increased costs. Also, preventive measures to reduce corrosion are included in the costs of operation and maintenance. Corrosion sometimes leads to an unexpected failure of metal parts in production complexes, and here lies mainly the seriousness of the problem of corrosion. As failure occurred suddenly, it may lead to significant damage greater than that caused by the expected erosion. In this regard, we must carefully determine the rates of wear in the metal parts during the course of the production process, through continuous measurements, periodic checks of rates of wear and continuous examination of metal parts to take preventive measures before the degree of corrosion reaches the extent that causes production to stop working or affect the course of the production process.

4- Production pollution: The corrosion products change the chemical nature of the medium, i.e. its pollution and it is often not desirable as the commercial requirements are obtaining a pure product with specific specifications and free from pollution.

5- Safety loss: Corrosion sometimes or often leads to disasters if preventive measures are not taken to prevent or reduce it, for example dealing with hazardous materials such as toxic gases such as hydrogen sulfide gas (H2S) and concentrated acids such as sulfuric and nitric acid, flammable materials, radioactive materials and chemicals at high Temperatures and when High pressure requires the use of certain mineral Substances that do not corrode significantly under such conditions. <sup>[6]</sup>

#### **1.6 Project objective:**

This study aims to evaluate the effect of two types of coatings on the corrosion behavior of metals immersed in sulfuric acid (H2SO4) and Qayara oil as a corrosive medium. Different metals will be studied, such as, aluminum, copper, bronze, and others,

This study will also include an evaluation of the effect of the coatings used on the corrosion behavior of metals.

Corrosion tests will be conducted on metals immersed in acid and Qayara oil in the following conditions: without coating and with coating

#### **1.2.1 LITERATURE REVIEW**

Patil and Sharma (2011) have undertaken a study on the corrosion kinetics of iron in acidic and basic media using weight loss method Potassium hydroxide, sulfuric acid, and nitric acid solutions were used in carrying out the research. The study was done at different intervals of time, within a temperature range of 25.0 °C to 30.0 °C. They observed that there was a variation in the rate of corrosion with different time intervals and different acid-base. Concentration sand that iron corroded most in nitric acid, followed by sulfuric acid, and lastly potassium hydroxide which was the least corrosive of the three solutions. By further studies, they noticed a first order kinetics for iron in all three solutions<sup>[14]</sup>

#### Zainab Azeez Betti

Corrosion-fatigue occurs by the combined actions of cyclic loading and corrosive environment. The effect of shot peening on cumulative corrosion -fatigue life of 1100-H12 Al alloy was investigated. Before fatigue testing, specimens were submerged in 3.5 %NaCl solution for 71 days. Constant fatigue tests were performed with and without corrosive environment.

Cumulative corrosion-fatigue tests were also carried out in order to determine the fatigue life before and after shot peening. The constant fatigue life wassignificantly reduced due to corrosive environment and the endurance fatigue

#### M. E. Matarneh

Experiments to evaluate the corrosion of 304L specimens were conducted using an aggressive solution as corrosion promoter. A glass aquarium filled with a solution consisted of 0.1 M HCl and distilled water was used as the corrosive environment. The aquarium temperature was around  $23\pm2^{\circ}$ C. The test duration for the specimens in this aquarium was 384 hours of exposure , For shot peening, the specimens showed best corrosion resistance at peening pressure of 60 psi up to 240 hours of immersion. However, the results suggest better corrosion resistance for 80 psi for longer immersion durations past 240 hours. This may have been caused by the presence of localized failures that were eliminated during the first part of immersion duration. These failures mat have been reduced for periods longer than 240 hours<sup>[16]</sup>

Afzal and Mustafa (2007) investigated The corrosion of aluminum

in aqueous chloride and nitrate media, and its inhibition by nitrite. The

commercial grade aluminum was observed through the potential dynamic method to be affected in both media at pH 4 and pH 10. Results showed

increase in the corrosion rate of aluminum with increase in concentrations of chloride and nitrate ions. It was also observed that for both media ,

corrosion behavior appeared to be significant at higher pH value due to

the instant dissolution of metal ions as complex. In order words ,

aluminum was comparatively more corrodible in basic condition than in

acidic condition. Finally, it was deduced that nitrite serves as an

important corrosion inhibitor in both chloride and nitrate media, and that the inhibition is more prevalent at lower pH and at higher concentrations of nitrite.<sup>[17][</sup>

Muna K. Abbass, in 2015 studied the corrosion resistance of metal matrix composite of an aluminum alloy (Al 6061) reinforced by SiC particles  $\cdot wt\%$  and 20wt%. It was found that adding of SiC particles to the aluminum alloy matrix increases the corrosion rate. It was shown that the corrosion resistance decreases with increasing of SiC particles as compared of base alloy<sup>[18]</sup>

#### 1.2.2 Summary

Widespread cases of corrosion have opened up ground for scientists to investigate how some metals and alloys behave in a number of corrosive environments. Using the weight loss technique and others, scientists have published large amounts of data on corrosion kinetics and corrosion inhibition, thus establishing a guiding principle for the corrosion behavior of materials in various applications. The literature review gives an overview of previous works on corrosion of metals. Several factors have been identified to influence corrosion, which include; temperature, pressure, PH, water wetting, sulfur, presence of organic (acetic) acid, and others.

## CHAPTER 2

### **Experimental Work**

#### **2.1. Introduction**

Throughout the present work, the project was conducted on a set of samples (metals) that were processed by epoxy coating and other remained normal without treatment, and we placed them in sulfuric acid and Qayara oil.

#### 2.2. The Experimental Plan Work Can Be Summarized Below

1. Material selection(metals).

2. preparing selected specimen for experimental work.

3. put metals before coating in all of the (qayara oil,sulfuric acid diluted) on his own

- 4. coating of selected metals and processed by (Epoxy).
- 5. after diluting the acid, We put each metal separately in the beker.

6.put another metals treated by coating epoxy in qayara oil .



Fig. (2-1): coating of selected metals by (Epoxy).

A group of minerals has been selected (Copper, Brass, Bronze, Ck-45, Aluminum), Its mechanical properties such as good mechanical strength, excellent machinability and high in corrosion strength.

#### **2.3 Preparation of Specimens**

The specimens are made according to standard specification 50 mm (length) 10 mm dimeter, Figure (2-2).



Fig. (2-2): shape of Specimen

The preparation of specimens is made by (CNC) machine in – northern – technical university – mechanical workshop.

The figure below explains the machine that used in the preparation of specimens.



Fig. (2-3) CNC Machine.

In the next step after the specimens are prepared, the specimens must be purifying from calculation and impurities by water to get a pure metal (in general the substances increasing the weight of the specimens).

The measurement of weight of each specimen is fundamental by accurate balance .

#### **2.4 The Preparation of Sulfuric Acid for Corrosion Test**

In this step the specimens will be divided into three groups for each metal (copper,brass,bronze,ck-45,alumnum)

The first group (untreated group) will be put in the H2SO4, which has properties For H2SO4 that show in Figure (2-5)

AC20692500 2,51	2986BATCH 17453403
SPECIFICATIONS assay (acidimetric).95 - 97 % colour (Hazen)max. 10 chlorides (cl)max. 0,00001 % nitrates and nitrites (as NO3)max. 0,00005 % aluminium (Al)max. 0,00005 % aluminium (Al)max. 0,00001 % arsenic (As)max. 0,00001 % barium (Ba)max. 0,00001 % barium (Ba)max. 0,00001 % bismuth (Bi)max. 0,000001 % bismuth (Bi)max. 0,000005 % boron (B)max. 0,000001 % calcium (ca)max. 0,000001 % calcium (ca)max. 0,00001 % cobalt (Co)max. 0,00001 % cobalt (Co)max. 0,00001 % cobalt (Co)max. 0,00001 % copper (Cu)max. 0,00001 % gallium (Ga)max. 0,00001 % gallium (Ga)max. 0,000001 % gallium (Ga)max. 0,00001 % gallium (Ga)max. 0,00001 % gallium (Ga)max. 0,00001 % gallium (Ga)max. 0,000005 % bernanium (Ce)max. 0,000005 % gallium (Ga)max. 0,000005 % gallium (Ga)max. 0,000005 % gallium (Ga)max. 0,000005 %	<pre>iron (Fe)max. 0,0001 % lead (Pb)max. 0,00001 % lithium (Li)max. 0,00001 % magnesium (Mg)max. 0,00001 % molybdenum (Mo)max. 0,00001 % molybdenum (Mo)max. 0,00002 % nickel (Ni)max. 0,00001 % platinum (Pt)max. 0,00001 % silver (Ag)max. 0,00001 % silver (Ag)max. 0,00001 % silver (Ag)max. 0,00001 % stilver (Ag)max. 0,00002 % stilver (Ag)max. 0,00003 % st</pre>

Fig. (2-4) Properties of The Sulfuric Acid.

For period of twenty eight days, in every Seven days each specimen should be cleaned then compare the original weight with the current weight to determine the loses amount of the weight for each specimen. The uncoated specimens during the formation of corrosion in the sulfuric acid explained in the figure (2-6).



Fig. (2-5) Shows uncoated Specimens into Sulfuric Acid.

The second group (treated group) will be put in the dilute soluton 5% H2SO4 as shown in figure (2-7)



Fig. (2-6) Shows the Specimens into 5% Sulfuric Acid.

The third group treated will be put in the dilute 10% H2SO4 as shown in figure (2-8)



Fig. (2-7) Shows the Specimens into 10% Sulfuric Acid.

#### 2.5 starting the crude oil Corrosion Test

After finishing the preparation of the specimens, we will also prepare an enough amount of crude oil from (Qayyarah oil field). this oil field is rich of sulfur, then the specimens will be divided into two groups for each metal.

First group of specimens(treated, coating by epoxy) using the previous device to enhance the specimens surface properties and then all the specimens will be immersed in the crude will and let the corrode for 28 days.

• Second group of specimens( un treated) will be immersed in the crude oil directly without going through coating by epoxy process and we will let these specimens in the crude oil for the same time as the treated specimens which is 28 days.

• Both of the two groups of specimens will be put to corrode for 28 days in the crude oil, during this period at every seven day all the specimens will be extracted from the crude oil, cleaned with acetone, and then weighted ,each specimen weight will be recorded for later experiment calculations.



Fig. (2-8) Shows the Specimens into The Qayara oil.



Fig. (2-9) Shows the Corrosion of The Specimens.

# CHAPTER **3**

### **Results and Discussion**

#### **3.1 Introduction**

Throughout the present work, measurement were made to establish the acid in treated material on corrosion behavior in oil pipelines.

About 35 days after the samples were affected by sulfuric acid ( $H_2SO_4$ ) and crude oil (Qayyarah). However, we deal with several type of metals, ( aluminum , copper , brass , bronze ,c4k45) becouse each type has special chemical and mechanical properties. One of the first results is the weight of the sample after 7 days, as the difference between the original weight and the weight increased during the time. We divided the samples into two groups ,the first group is treated by coating but the second group is non treated by coating ,each groups immersed in dilute  $H_2SO_4$  (5%) and another time immersed in dilute  $H_2SO_4$  (10%),as well as immersed in crude oil.

#### **3.2.The description and analyze**

the results for the subject samples in crude oil.

we notice from the figure (3-1) that the weight of the aluminum simple decreases over time, but the coated sample is better than the uncoated one when they are fermented in crude oil.



Figure (3-1) shows the weight loss of aluminum over time.

In figure (3-2) show that the weight of the copper simple decreases over time, but the coated sample is better than the uncoated one when they are fermented in crude oil.



Figure (3-2) shows the weight loss of copper over time.

from figure (3-3) observed that the the weight of the ck4-45 decreases over time, but the coated sample is better than the uncoated one when they are fermented in crude oil.



Figure (3-3) shows the weight loss of copper over time.

3.3. The description and analyze the results for the subject samples in  $H_2SO_4$ .

We notice from the figure that the weight of the sample decreases over time, but the coated sample is better than the uncoated one when they are fermented in oil



Figure (3-4)Shows the Weight Losses of Aluminum Alloy Specimens with The Time

from the figure (3-5) show that the weight of the Brass decreases over time, but the coated sample is better than the uncoated one when they are fermented in diluted acid.



Figure (3-5) Shows the Weight Losses of Brass Alloy Specimens with The Time

notice from the figure below (3-6) that the weight of the sample Bronze decreases over time, but the coated sample is better than the uncoated one when they are fermented in diluted acid



Figure (3-6) Shows the Weight Losses of Brass Alloy Specimens with The Time

### CHAPTER4 CONCLUSIONS & RECOMMENDATIONS

#### **£.1** Conclusion

This study experimentally examined the corrosion behavior of six different metals after coating with epoxy and being immersed in H2SO4 acid for four weeks. These metals are CK-45, Brass, Carbon steel, 201-stain, Bronze and Aluminum. Further, two different concentration was taken into our consideration. These concentrations are 5%, and 10%. The main conclusion of this study is listed below:

The weight of all considered metals decreased in different ratios after immersion in H2SO4 acid.

The weight loss of each metal increases slightly with increasing H2SO4 concentration.

The epoxy-treated samples showed corrosion resistance and positive results compared to the untreated samples

#### 4.2 Future works

Investigate the corrosion behavior of the metals under consideration after immersion in a different acid.

Study the cumulative fatigue damage under corrosion and laser peening.

Study the effects of Shot Peening on corrosion in metals.

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