Study on the Corrosion of Carbon Steel in Gasoil-Brine Mixture in presence of acid

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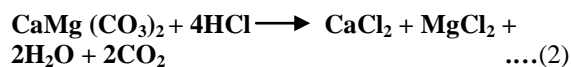Al-Nahrain University
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Abstract

Corrosion tests of carbon steel in two phase flow of two immiscible liquids ($CaCO_3$ solution-gasoil mixture) under different operating conditions of temperature, agitation velocity, and phase fractions, were carried out using weigh loss method. Flat blade disc turbine mixer was used to simulate the two phase flow conditions. In two immiscible phases tests, the ranges of operating parameters were agitation velocities of 0 - 1200 rpm, temperature of 25- 45 ^{o}C, gasoil volume percent of 1-10% vol. The effect of presence of acids such as HCl and H_2SO_4 on the corrosion rate in two immiscible phases mixture was also investigated under different conditions. The corrosion rate in brine-gasoil mixture had unstable trend with concentration of gasoil and rotational velocity.

Key words: corrosion, carbon steel, carbonates calcium, salt, gas oil, agitation mixer**.**

1. Introduction

Corrosion problems may occur in numerous systems within the petroleum industry. Acidization of oil and gas wells is probably the most widely used work over and stimulation practice in oil industry [Allen and Roberts, 1989]. Corrosion which takes place in an oil pipeline is basically no different from the corrosion which occurs in a producing oil well or any water-containing system. The water associated with pipelines always contains ions from dissolved gases (e.g. CO_3^{2-} obtained by dissolved CO_2) and solids (e.g. NaCl), thus functioning as a charge-conductive electrolyte [Brondel, 1994]. Oil well stimulation, usually done with hot solutions of hydrochloric acid, may induce severe corrosion attack on production tubing, downhole tools and casing. oil well stimulation is the general term describing a variety of operations performed on a well to improve its productivity. Opening up new channels in the rock for the oil and gas to flow through is called stimulation.

Three stimulation treatments are commonly used: explosives to break up the rock, injection of acid to partially dissolve the rock, and hydraulic fracturing to split the rock and prop it open with proppants. Stimulation operations can be focused solely on the well bore or on the reservoir; it can be conducted on old wells and new wells alike; and it can be designed for remedial purposes or for enhanced production. Its main two types of operations are matrix acidization and hydraulic fracturing [Williams and Nierode, 1972]. Hydraulic fracturing, which includes acid fracturing, involves the injection of a variety of fluids and other materials into the well at rates that actually cause the cracking or fracturing of the reservoir formation. The variety of materials includes, amongst others: water, acid, special polymer gels, and sand. The fracturing of the reservoir rock and the subsequent filling of the fractured voids with sand ("proppant") or the creation of acid channels allows for an enhanced conduit to the well bore from distances in excess of a hundred feet. Hydraulic fracturing involves the injection of water into the formation, while CO_2 fracturing uses gaseous carbon dioxide. The acid reaction may be represented by the following equation [Williams and Nierode 1972]:

$$2HCl + CaCO_3 \longrightarrow CaCl_2 + H_2O + CO_2 \quad ....(1)$$

$$CaMg\,(CO_3)_2 + 4HCl \longrightarrow CaCl_2 + MgCl_2 + 2H_2O + 2CO_2 \quad\quad ....(2)$$

Acidic solutions of hydrochloride acid and sulphuric acid have wide industrial applications, the most important fields being acid pickling, acid descaling in petroleum and chemical processes, industrial cleaning and oil-well acidizing [Banas et.al, 1992]. The commonly used acids are hydrochloric acid, sulphuric acid, nitric acid, etc. Acid is used to remove damage near the wellbore in all types of wells. In carbonate formations, acid may be used to create linear flow systems by acid fracturing.

The rate of corrosion of carbon steels in dilute acids depends strongly on steel chemical compositions, especially the carbon content.

Accordingly, the oxidation reaction of iron (Fe $=Fe^{2+} + 2e^-$) occurs on the ferrite phase, and the cathodic reduction reaction of hydrogen ($2H^+ + 2e^- = H_2$) occurs on the cementite phase. When carbon steel contacts sulfuric acid, the former is in fact reduced to form H_2 and the iron oxidizes with the formation of ferrous sulfate ($FeSO_4$), as shown in reaction:

$$H_2SO_4 + Fe \longrightarrow FeSO_4 + H_2 \qquad \dots (3)$$

The $FeSO_4$ adheres to the steel surface and forms a protective layer. This layer prevents the metal against further attack by sulfuric acid [Dean and Grab, 1985]. Carbon steel, the most widely used engineering material, accounts for approximately 85%, of the annual steel production worldwide.
It is well known that the rate controlling step in most natural water corrosion process is the cathodic half reaction. The most important cathodic process in aerated waters is oxygen reduction. The rate of this half reaction is generally limited by the speed at which oxygen can reach the surface of the metal. This oxygen is transported from the bulk water to the surface across the boundary layer by diffusion. The oxygen concentration will vary from a minimum at the surface to bulk concentration at the outside edge of the boundary layer. This results in the concentration gradient and promotes the transport of oxygen [Smith et.al, 1989]. Ifthe corrosion process is under cathodic diffusion control then the flow increases the corrosion rate. This effect generally occurs when an oxidizer is present in small amounts, as is the case for dissolved oxygen in acids or water [Fontana and Greene, 1989]. The effects of concentration, velocity and temperature are complex and it will become evident that these factors can frequently outweigh the thermodynamic and kinetic considerations [Shreir et.al, 2000]. It is widely recognized that the corrosion of carbon steel may be accounted for by the anodic reaction as in equation (3) and cathodic reaction in presence of oxygen:

$$\tfrac{1}{2} O_2 + H_2O + 2e^- \rightarrow 2OH^- (6) \qquad \dots (4)$$

The aim of this work is to study the corrosion behavior of carbon steel in oil-brine solution as well as the effect of the presence of acids (HCl) in the solution by using flat blade mixer (Rushton type mixer) to investigate the effect of different operating conditions such as agitation velocity, temperature, concentration of petroleum fraction, and acid on the corrosion rate.

Experimental Work

The agitation system consisted of cylindrical Perspex vessel of wall thickness 4 mm, vessel diameter D_t=30 cm and H=30 cm height, filled with test solution to height equal to its diameter and covered with flat perspex sheet. Five equally spaced vertical baffles made of perspex each of width equal to J=2.5 cm. A prespex 4-blade disc impeller of diameter equal to Da=10 cm, blade length L=2.5 cm, blade width W=2 cm, as shown in Fig.1, was used and located E=10 cm from the tank bottom. Before each test, the vessel was washed with tap water using liquid detergent, rinsed with tap water, and concentrated (HCl) to remove corrosion products and deposits, then washed with tap water, followed by distilled water.

Before each experiment, the carbon steel specimen (working electrode) was polished with 120, 180, 220, 400 and 2000 grade silicon carbide papers, washed with brushing by plastic brush with running tap water, immersed in ethanol for 30 second, dried with clean tissue, and then dried by using electrical oven to temperature of about 105 ^{o}C for 5 minutes [Mahato et.al, 1980, Hasan and Sadek, 2014]. The specimen then was stored in vacuum desiccator over high activity silica gel until use. When caring out the experiment, the specimen was weighted (w_1) by using digital balance. After that the specimen was exposed to corrosion environment for 3.5 h in brine solution- gasoil- acid two phase mixture. At the end of experiment, the specimen was washed by tap water with brushing to remove the corrosion products that formed on the outside surface and then immersed in 10% hydrochloric acid containing chemical inhibitor (thioharnstoff) for 30 second [Fontana, 1986; Slaiman et al., 2010]. Blank tests showed no appreciable weight loss caused by cleaning with inhibited acidic solution. After that, the specimen was washed by tap water, distilled water, dried with clean tissue, rinsed with ethanol and dried by using electrical oven to a temperature about 100 ^{o}C for 5 minute. Then the specimen was kept in the desiccator to cool and weighted (w_2). The corrosion rate in gmd can be determined using:

$$CR = \frac{\Delta W}{A \times t} \qquad \dots (5)$$

Where, CR is the corrosion rate in gmd, ΔW is the loss in weight in gram, A is the specimen area in m^2, and t is the time in day. Each run was carried out twice with third one when the reproducibility was in doubt. Figure 2 shows the experimental apparatus which was composed of stirrer to obtain different rotational velocities, (0 – 1000) rpm,

heater and controller to obtain different solution temperatures, oxygen meter was used to measure the solubility of oxygen in solution in mg/l, pH meter was used to monitor the pH of the solution, and conductivity meter to measure the electrical conductivity of the solution. Carbon steel rod

specimen was prepared to fit the specimen holder with a surface area of 14.3256 cm^2 having a dimensions of 35 mm long (L), 12 mm outside diameter (d_o). The composition of used carbon steel specimens is shown in Table 1. It serves as a cathode.
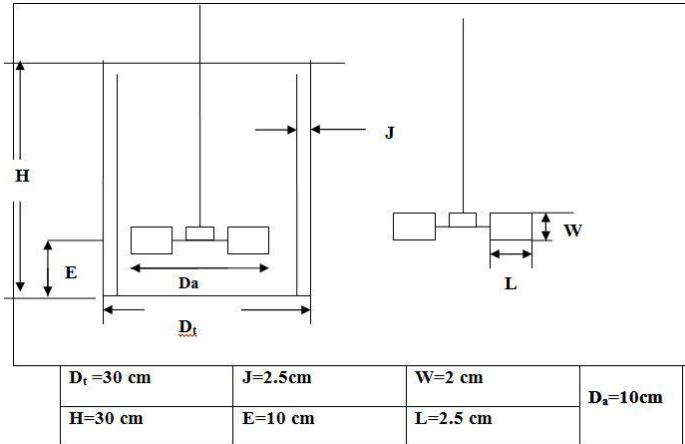

| D_t =30 cm | J=2.5cm | W=2 cm | |
| H=30 cm | E=10 cm | L=2.5 cm | D_a=10cm |

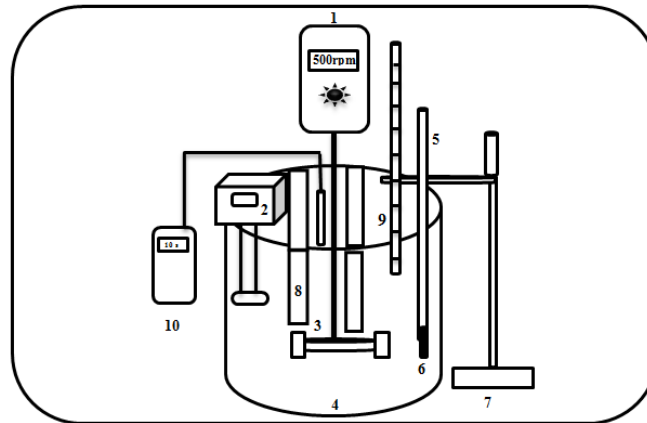**Figure1:** Dimensions of Tank and Disk Turbine (four- Blades Turbine).

Figure 2: Experimental apparatus
1- Stirrer, 2- heater and controller, 3- impeller with 4 blade turbine, 4- agitation tank, 5- holder, 6- work electrode (specimen), 7- stand, 8- baffles, 9- thermometer, 10- conductivity meter.

2. Results and discussions

Figure 2 shows the effect of rotational velocity on corrosion rate of carbon steel (CS) in 2.5×10^{-3} M $CaCO_3$ at T =35 ^oC that expressed in gmd with flow velocity. The figure indicates that the corrosion rate increases with rotational velocity. This trend is in agreement with pervious works [Mahtato et al, 1980, Saliman et. al, 2008, Hasan and Sadek, 2014]. The increase in CR with flow velocity is due to the increased transport of oxygen from bulk of the solution to the metal surface the rate of oxygen reduction reaction is generally

limited by the speed at which oxygen can reach the surface of the metal. Previous studies [Foroulis, 1979; Scheers, 1992; Shreir et. al., 2000, Slaiman and Hasan, 2010] indicated that the greater turbulence due to high velocities results in more uniform O_2 concentration near the surface. Fig.1 reveals that at extremely high velocity the CR tends to decrease. This can be ascribed to the fact that at high velocity the formation of corrosion product is higher which leads to protect and passivate the metal surface [Revie and Uhlig, 2008; Fontana, 1986].

Figure 3 shows the effect of agitation velocity on corrosion rate of carbon steel (CS) in 2.5×10^{-3} M $CaCO_3$ and different concentrations of gasoil at T $=35$ $^{\circ}$C that expressed in gmd with flow velocity. It is clear that in two phase mixture, increasing velocity leads to increase the turbulence and increase the number of dispersed aqueous phase droplets. Increasing velocity lead to an increase in the number of droplets [Coulson et.al, 1999] and thus lead to more intimate contact between the dispersed phase droplets and the metal specimens. Increasing velocity increases the corrosion rate by increasing O_2 transport to the surface from dispersed and continuous phases to the metal leading to an increase in the corrosion rate. When further increasing rotation velocity (mixing velocity) the corrosion rate goes down. This is attributed to the fact that when increasing rotational velocity the shear forces increase leading to break the dispersed phase (gasoil) drops to small droplets which, in turn, increases the contact area between dispersed phase (gasoil) and the metal as well as the probability of striking the metal by gasoil droplets also increases leading to a decrease in the corrosion of carbon steel metal because the gasoil is less corrosiveness than brine owing to its low electrical conductivity as shown in Table 4. Some studies for corrosion under two phase flow, confirmed that the drop impact velocity at the surface affects corrosion rate [Bozzini et. al, 2003; El-Batsh et. al, 2012].

Comparing Figs. 2 and 3 indicates that corrosion rates in two phases (concentration of gas oil (1, 2, 5%vol.)) is lower than that in single phase of $CaCO_3$ solution. This is ascribed to the fact that the electrical conductivity of gasoil is lower than that of water ($CaCO_3$ solution). The solubility of oxygen in gasoil is much higher than that in water, for example, at room temperature the solubility of oxygen in gasoil is 154 cm^3/L [Nelson, 1958],while in water it is about 30 cm^3/L [Perry and Chilton, 1997; Shreir, 2000]. Also, the figure reveals that as the volume percent of gasoil increases, the corrosion rate increases, this increase is attributed to the fact that the presence of dispersed phase (gasoil) in relatively high percents leads to the increase in the O_2 concentration in the solution of two-phases (because of high O_2 content of gasoil) therefore the corrosion rate will be higher.

Figure 4 shows the effect of agitation velocity on corrosion rate of carbon steel (CS) in 2.5×10^{-3} M $CaCO_3$ + 2% HCl and different concentration of gasoil (2, 5, and 10%v/v) at T =35 $^{\circ}$C. It is clear that increasing the flow velocity from 200 to 400 rpm leads to an increase in the corrosion rate. This increase is due to the increase in the oxygen transport to metal surface with increase the flow

velocity. This agrees with previous studied that found increase in the corrosion rate of iron in oxygen saturated acid solutions, with increasing velocity of the solution [Ross et.al, 1966, Hasan and Sadek, 2014]. George and Nesic [2007] stated that the increase in CR with flow velocity in acid solutions is due to the increase of hydrogen diffusion toward metal surface. The corrosion rate in 2.5×10^{-3} M $CaCO_3$ + 2% HCl + different concentration of gasoil is higher than in 2.5×10^{-3} M $CaCO_3$. The increased corrosion rate of iron as pH decreases (i.e. the pH of 2.5×10^{-3} M $CaCO_3$ is 8.84 and it decreases to 0.29 in 2.5×10^{-3} M $CaCO_3$ + 2% gasoil+ 2% HCl mixture) is caused by increased hydrogen evolution reaction that leads to greater accessibility of oxygen to the metal surface on dissolution of the surface oxide favors oxygen depolarization. Fig.4 reveals, also, that as the volume percent of gasoil increases (2, 5, and 10%) with constant volume percent of HCl, the corrosion rate increases. This increase is attributed to the fact that the presence of dispersed phase in relatively high percent leads to the increase in O_2 concentration of acidic two-phase solution because high O_2 content of gasoil therefore the corrosion rate will be higher. The oxygenated acids are characterized by high corrosiveness as has been evidenced by several studies [Shrier, 2000, Hasan and Sadek, 2014]. Previous studies of Alwash et.al [1987] and Turkee [2009] stated that the corrosion rate in aerated acidic solutions increases as the flow increases.

Figure 5 shows the effect of rotational velocity on corrosion rate of carbon steel (CS) in 2.5×10^{-3} M $CaCO_3$ + 2% Hcl +5% gasoil at different temperatures (25, 35, 45 $^{\circ}$C) that is expressed in gmd . It is clear that increasing velocity, leads to an increase in the corrosion rate. An increase in temperature of acid solutions can affect the corrosion behavior of materials in different ways: (i) the rate of chemical reaction increases with temperature. This issue is here very effective because the system was under mass and activation control because of the presence of acid [Khadom et.al, 2009], (ii) The solubility of some of the reaction products may change resulting in different corrosion reaction products,(iii) viscosity is decreased leading to an increase in the diffusion coefficient of O_2 [Bird et.al, 2002; Brodkey and Hershey 1989].

Figure 6 shows the variation of corrosion rate expressed in gmd with velocity at 35 $^{\circ}$C in 2.5×10^{-3} M $CaCO_3$ + 2% H_2SO_4 + 5 and 10%. The figure indicates that the corrosion rate increases with rotational velocity. This increase is due to increase the mass transport of oxygen from the bulk of the

solution to the metal surface. Consequently, the corrosion rate of the carbon steel increases. Also, the figure reveals that as the volume percent of gasoil increases (5 to 10%) with constant volume percent of H_2SO_4, the corrosion rate increase. This increase is because of high O_2 content of gasoil that will diffuse in the solution due to high turbulence accompanying the agitation; therefore, the corrosion rate will be higher.

Figure 7 shows the variation of corrosion rate with temperature at 300 rpm in 2.5×10^{-3} M $CaCO_3$ + 2% H_2SO_4 + 5% gasoil that expressed in gmd. It is clear that corrosion rate increases slightly when the temperature increases from 25 ℃ to 35 ℃ and increases considerably when the temperature increases to 45℃. The increase of CR with temperature is well known phenomenon the acid solution. This behavior can be explained as follows: increasing temperature leads to change two variables that act in a conflicting way. Firstly, increasing temperature ccelerates the reaction rate as dictated by Arrhenius equation. Moreover, diffusion rate of dissolved oxygen increases by increasing the molecular diffusion coefficient [Henry and Scott, 1999; Uhlig, 1979]. Zehbour et.al, [2012] in their study on corrosion of carbon steel pipes and tanks by concentrated sulfuric acid, stated that the corrosion mechanism of carbon steel in sulfuric acid, accelerated by several factors such as concentration, temperature, and velocity flow. Corrosion rate show high increase in its value in brine-acid solution when comparing with solution containing brine only. This increase is due to the increased electrical conductivity of two-phase compared to single phase (brine) as shown in Tables 3 and 4. Table 2 shows the experimentally measured solubility of oxygen (C_b) in ppm (mg/L) by using oxygen meter at various carbonates calcium concentrations. From the table it is clear that at a given temperature, increasing the dissolved salt ($CaCO_3$) concentration leads to a decrease in the O_2 concentration. Also, the temperature has an effect on the solubility of oxygen at constant salt concentration, that is, increasing the temperature leads to a significant decrease in the O_2 concentration.   Table 3 shows the conductivity of different concentration of $CaCO_3$ solution at different temperature. Table 4 shows the conductivity values of $CaCO_3$ - gasoil, acid mixtures at different concentrations and T= 35 ℃, u=200 rpm. Table 5 shows pH values for $CaCO_3$ and $CaCO_3$- gasoil, acid mixtures at different concentrations and T= 35 ℃ and u=200 rpm.

Conclusions

From the results of this work it can concluded that:

1. The corrosion rate in single phase brine $CaCO_3$ solution increases about two times with increasing agitation velocity from 0 to 1200 rpm.

2. The corrosion rate in two phase brine-gasoil mixture increased with the increase in agitation velocity. Further increase in velocity of agitation causing more dispersion of gasoil, so the number of droplets increases which leads to decrease the corrosion rate...

3. In brine $CaCO_3$- gasoil in presence of HCl and H2SO4, the corrosion rate is higher than that in single phase $CaCO_3$ solution and increases with increasing agitation velocity and temperature. The limiting current density (corrosion rate) increases with the increase rotational velocity, salt concentration, and temperature.

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Nomenclature

CS: Carbon steel

CR: corrosion rate

A: the area $= \pi * d_o * L + A = \pi * r^2$ in m^2.

t: time of exposure (hours)

ΔW: the weight loss $= w_1 - w_2$ in grams.

C_b: the solubility of oxygen in (mole/m^3).

rpm: revolution per minute.

u: rotational velocity in rpm.

Table 1: Composition of carbon steel specimens.

Eleme nt	C%	Mn %	P%	S%	Cu %	Fe
Weigh t %	0.45 4	0.68 5	0.01 1	0.02 7	0.29	balanc e

Table 2: measured oxygen solubility in ppm (mg/L) at different $CaCO_3$ concentrations and temperatures and u= 0 rpm.

$CaCO_3$ concentrations in M	C_b ppm		
	25 °C	35°C	45°C
9.99×10^{-4}	7.80	7.32	6.22
2.5×10^{-3}	6.95	6.15	4.40
4.99×10^{-3}	6.85	5.84	4.05
9.99×10^{-3}	6.2	5.23	4.72

Table 2: Measured conductivity values of different concentration $CaCO_3$ solution and different temperature

Solution / T, °C	Conductivity, µS		
	25 °C	35°C	45°C
2.5×10-3	41.5	44.5	47.1
4.99×10-3	42.9	45.9	49.4
9.99×10-3	45.7	48.3	52.1

Table 4: Measured conductivity values of $CaCO_3$ - gasoil, acid mixtures at different concentrations and T= 35 °C, u=200 rpm.

Solutions concentration	Conductivity, µS
2.5×10^{-3} M$CaCO_3$+ 2% gasoil	15.26
2.5×10^{-3} M$CaCO_3$+ 2% gasoil+ 2% HCl	188.5
2.5×10^{-3} M$CaCO_3$+ 5% gasoil+ 2% HCl	169
2.5×10^{-3} M$CaCO_3$+ 5% gasoil+ 2%H_2SO_4	94.8

Table 5: Measured pH values for $CaCO_3$ and $CaCO_3$- gasoil, acid mixtures at different concentrations and T= 35 °C and u=200 rpm.

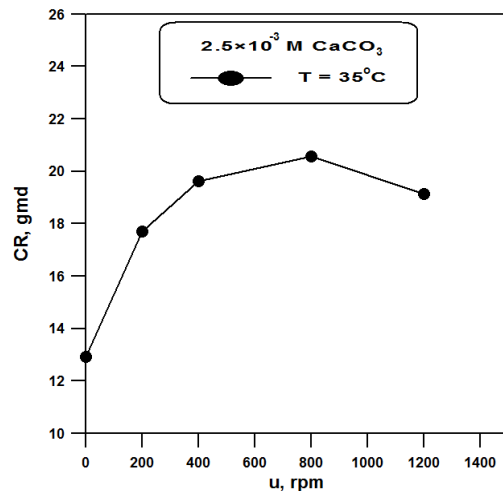| Solutions concentration	pH
gasoil	7.5
2.5×10^{-3} M $CaCO_3$	8.84
2.5×10^{-3} M$CaCO_3$+ 1% gasoil	8.52
2.5×10^{-3} M$CaCO_3$+ 2% gasoil	8.32
2.5×10^{-3} M$CaCO_3$+ 5% gasoil	8.02
2.5×10^{-3} M$CaCO_3$+ 2% gasoil+ 2% HCl	0.29
2.5×10^{-3} M$CaCO_3$+ 5% gasoil+ 2% HCl	0.34
2.5×10^{-3} M$CaCO_3$+ 5% gasoil+ 2% H_2SO_4	0.55
2.5×10^{-3} M$CaCO_3$+ 10% gasoil+ 2% H_2SO_4	0.58

Figure 2; Variation of CR with velocity in single phase $CaCO_3$ at 35 °C.
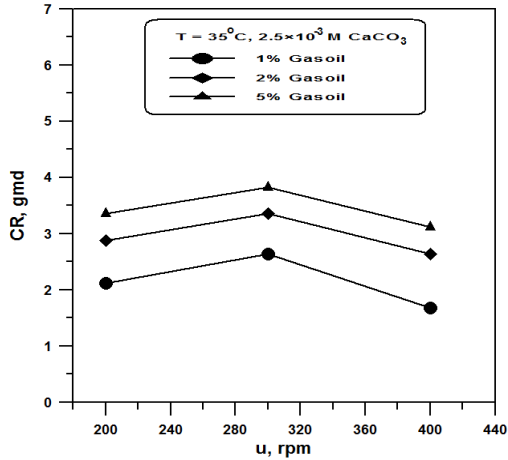
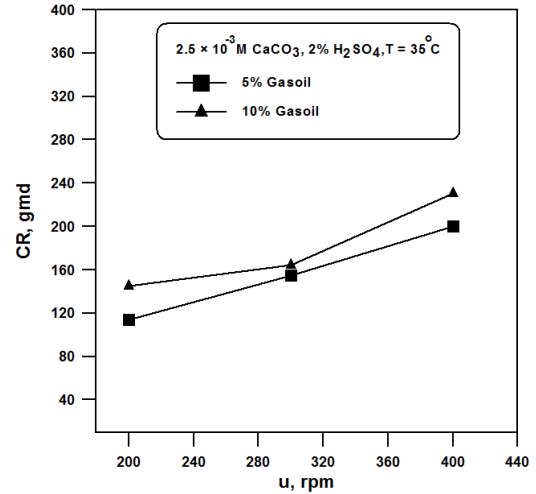**Figure Fig. 3:** Variation of CR with velocity in two phase $CaCO_3$ + gasoil at 35°C.

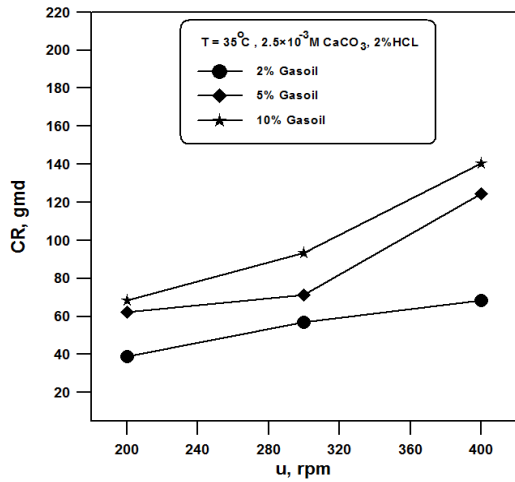**Fig.4:** Variation of CR with velocity in two phase brine- gasoil in presence of HCl.

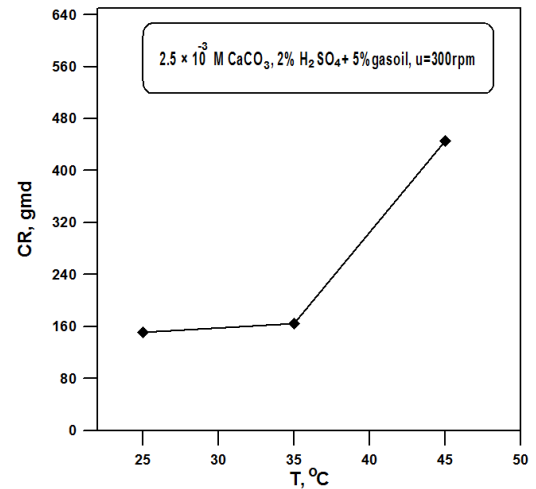**Fig. 5:** Variation of CR with velocity in two phase brine- gasoil in presence of Hcl at different temperature.

Figure 6: Variation of CR with velocity in two phase brine- gasoil in presence of H_2SO_4.

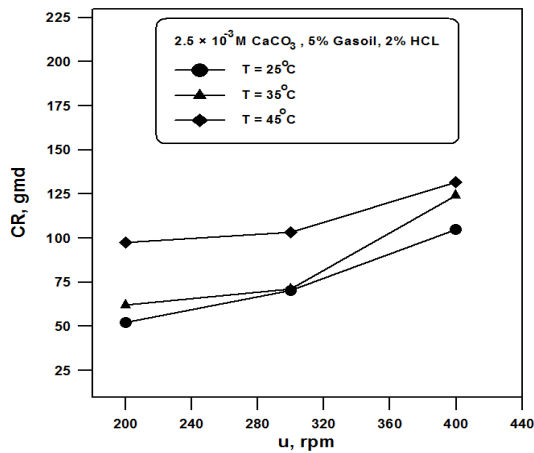**Figure 7:** Variation of CR with temperature in two phase brine- gasoil in presence of H_2SO_4.

دراسة التأكل الفولاذ الكاربوني في خليط ملحي وزيت الغاز بوجود الحامض

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الخلاصة:

تم اجراء تجارب تأكل الفولاذ الكاربوني في طور احادي وثنائي المحلول من السوائل غير الممتزجه (محلول كاربونات الكالسيوم وزيت الغاز) وتحت ظروف تشغيل مختلفة من درجة حرارة، وسرعة جريان سائل، والنسبة الحجمية لزيت الغاز باستخدام طريقة فقدان الوزن . حيث تم استخدام خلاط متكون من قرص وشفرات لخلط الطور الثنائي .
تم اجراء التجارب تحت سرع خلط نتراوح بين0-1200 دوره/ دقيقة، ودرجة حرارة بين 25- 45م o، ونسبة حجم زيت الغاز(1-10%). زيادة سرعة الدوران في طور أحادي المحلول الملحي (كاربونات الكالسيوم (CaCO3 تؤدي الى زياده معدل التأكل. كذلك تم دراسة تأثير وجود الحوامض مثل حامض الهيدروكلوريك وحامض الكبريتيك على معدل التأكل في مرحلتين امتزاج الخليط ايضا تحت ظروف مختلفة .

معدل التأكل يوضح زياده عاليه في قيمته في المحلول الملحي يحتوي على الحامض عند مقارنه مع محلول يحتوي على محلول ملحي فقط. وهذه الزياده نتيجه الزياده العالية في التوصيل الكهربائي للطور الثنائي مقارنة مع الطور الاحادي (ملح). كان معدل التأكل في خليط محلول الملحي وزيت الغاز يتجه الى كونه غير مستقر مع تركيز زيت الغاز وسرعة الخلط. كذلك قيمه معدل التأكل في محلول الملحي يحتوي على الحامض تزداد بشكل طفيف عندما تزداد درجة الحرارة من 25 الى 35 م o وزياده كبيرة عند زياده درجة الحرارة الى 45م o.