

Inhibitory Effects of Calcium Sulphate Concentrations on Cookware Aluminium Corrosion in Highly Acidic and Alkaline Media at Elevated Temperatures

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ABSTRACT

Aluminium corrosion can deleteriously reduce the structural strength, integrity, safety, and serviceability of the metal. In addition, it is an inevitable issue of health concern in food environments because the substances resulting from the corrosion can have toxicological, mutagenic, and carcinogenic effects in the human body, potentially leading to many chronic ailments. This paper presents an inquiry into the inhibitory effects of calcium sulphate as a cheap, worldwide-available, and environmentally friendly food substance on corrosion of cookware aluminium in highly acidic and alkaline media. Coupons were produced from the aluminium 6061 alloy as a common cookware aluminium type and immersed for 1-48 hours in sequentially treated acidic media of 2.5, 2.7, and 3 pH and alkaline media of 10.5, 10.7, and 11 pH with anhydrite calcium sulphate concentrations of 0, 20, 25, 30, and 35% of the media at 30, 50, and 70°C temperature conditions. Corrosion penetration rates (CPRs), inhibition efficiencies, and micro-topographic changes of the coupons were evaluated relative to those of the coupons immersed in the untreated media. Results show a CPR range of 0.121-1.579 in the acidic and 0.101-1.105 mm/yr in the alkaline media. The CPR increased with increasing exposure temperature and media acidity and alkalinity levels but decreased with the immersion durations and the treatment concentrations to more or less constant values. The highest corrosion inhibition efficiency of 74.34% occurred in the acidic media after a 3-hour immersion in the 2.7-pH medium at 30°C, with 35% treatment of the medium, while the highest value in the alkaline media was 72.95% after a 3-hour immersion in the 10.5-pH medium at 30°C, with 30% treatment of the medium. SEM micro-topographic analyses indicate that calcium sulphate facilitates corrosion inhibition of the cookware coupons in the media by forming coatings on them. Thermodynamic and kinetic analysis at the temperature conditions of the media shows that the inhibitory process of the sulphate is essentially by physisorption and is well fitted to the Langmuir adsorption isotherm model, as indicated by the Gibbs free energy, enthalpy, and entropy of the sulphate adsorption on the cookware coupons with average values of -23.345 kJ, -30.13 kJ, and 0.195 kJ/K, respectively. The study indicates that anhydrite calcium concentrations of greater than 35% can satisfactorily inhibit corrosion of cookware aluminium under the media conditions.

Key words: Cookware aluminium, Corrosion issues, Calcium sulphate, Inorganic substance, Environmentally friendly, Biocompatible, Widely available, Inhibition investigation.

INTRODUCTION

The aluminium metal has excellent benefits in food handling due to its low cost, light weight, high temperature tolerance, wide availability, ability to heat and cool quickly, high corrosion resistance, perennial shiny appearance in unpolluted natural environments, and excellent formability into kitchenware shapes [1-3]. However, in comparison with some other food-grade metals like stainless steel, aluminium metal has lower strength and hardness integrity and is leachable with a lower level of health safety for use in handling food [1, 3, 4]. There has been many engineering, scientific, and medical concerns about the corrosion of aluminium metal since its debut for food handling due to the toxicological, mutagenic, and carcinogenic effects of its corrosion ions and other substances in the human body system as free and highly unstable radicals compared to the strongly and stably bound, benign, and less absorbable aluminium ions and other substances that are naturally found in

food or water, which present no or minimal health risks in humans [1, 3, 4, 5]. Leached ions or substances from corroded aluminium can inadvertently find their way into the human body system from time to time through food, water, or inhalation and endanger health if their total content exceeds the health-safe guideline value, or if they cause an unacceptable change in the composition of the food or deterioration of its organoleptic properties [1-3, 4, 5]. According to the European Food Safety Authority (EFSA) and the Food and Drug Administration (FDA) standards, the regulatory limits of critical metals in food-contact aluminium are lead < 0.2 mg/kg, cadmium < 0.5 mg/kg, arsenic < 0.1 mg/kg, and maximum total migration of < 10 mg/dm² [6, 7].

Cookware aluminium refers to a specific category of aluminium alloys, such as Al 1100, 1050, 1060, 1100, 3003, 3105, 5005, 5052, 6061, 6063, and 8011, which are specially designed and tested for direct contact with foods and beverages by limiting all elements in them below threshold values for food safety compared to the other aluminium alloys [3, 4, 5, 8-15]. Cookware aluminium alloys are used to produce pots, frying pans, skillets, saucepans, stockpots, baking pans, aluminium foil and wrappings, baking trays, moulds, and food storage containers [7, 8]. Apart from food handling, cookware aluminium alloys have applications in the following [6,7]:

- Electrical systems such as conductors and high-voltage overhead power lines as a lighter and cost-effective copper alternative.
- Drug, cosmetic, and industrial chemicals packaging
- Transportation system components, such as aircraft fuselage and wings; automobile engine parts and chassis; and train and ship components, for reduced weight and fuel efficiency improvements.
- Building components, such as window and door frames, curtain walls, roofing, siding, and architectural insulation
- Manufacturing various machinery, tools, and equipment parts, including ladders, solar panel frames, and robotic components.
- Chemical processing equipment, such as tanks and piping.
- Reflective coating for mirrors in telescopes and other optical instruments

The 6061-aluminum alloy is the most popular and cost-efficient cookware aluminium alloy. It is used for many parts, from our typical bike to electrical equipment to beverage cans [11, 12]. It is extremely strong, heat-treatable, weldable, formable, machinable, and rust-resistant compared to other cookware aluminium alloys [11, 12]. Cookware aluminium or its products are frequently processed, applied, or stored in hot, salty, acidic, and alkaline environments where they undergo various corrosion forms, such as erosion, crevice, pitting, filiform, cavitation, exfoliation, intergranular, galvanic, and microbiologically influenced corrosion. In near-neutral pH environments, aluminium cookware tends to resist corrosion well due to an oxide layer that forms on its surface and protects it from further corrosion [1, 3-5]. Cookware aluminium or products made from it are often used in contact with acidic environments of very low pH such as hydrochloric and other acidic solutions in many industrial processing and storage as well as foods such as lemons, oranges, and grapefruit (average pH 3.0-4.3), strawberries (pH 3.0-3.9), cranberry juice (pH 2.6), plums (pH 2.8-3.4); grapes (pH 2.8-3.0), blueberries (pH 3.1-3.3), raspberries (pH 3.2-4), apples (pH 3.3-4.0), prunes (pH 3.6-3.9), peaches (pH 3.3-4.0), apricots (pH 3.3-4.8), pineapple (pH 3.2-4.0), and prunes (pH 3.6-3.9), tomatoes (average pH 3.5-4.9), coffee (pH 4.0-4.3), lemonade (pH 2.6), energy drinks (pH 3.1), lemon juice (pH 2.3), orange juice (pH 3.9), sports drinks and flavoured water (pH 3.3), pineapple juice (pH 3.4), flavoured tea (pH 3.5), apple juice, (pH 3.6), yoghurt (average pH 4.0- 4.4), and alcohols such as whiskey and rum, red and white wine, and beer (pH 3.1-4.5) [15-18]. On the hand, there are several environments with pH above 7 but the ones that are highly alkaline with pH of at least 9 include several natural conditions in some alkaline soils, soda lakes, sodium hydroxides, calcium hydroxides, potassium hydroxides, foods, cement and lime manufacturing waste, pulp and paper sludge, coal and biomass ash, soapy waters, bleach, oven cleaners, caustic soda, liquid drainers, trisodium phosphate, sodium carbonate. soda lakes and hypersaline inland seas, etc. with pH values in the range 9-12. etc. Examples of highly

alkaline foods are leafy greens such as spinach (pH 9.0-9.5), kale (pH 8.0-9.0), and baking soda or sodium bicarbonate pH (8.0-9.0). Acidic ions, such as chloride, can break down aluminium metal, causing pitting corrosion and forming small holes on its surface. Alkaline environments become more corrosive to aluminium metal as their pH increases to 14. In such environments, the protective oxide film on the aluminium becomes more porous as pH increases, leading to localized dissolution of the aluminium [15-19]. Furthermore, elevated temperatures and certain foods containing aggressive compounds like allicin or diallyl-disulfide can accelerate the corrosion process of cookware aluminium or its products [20-23].

Cookware aluminium equipment and containers, such as household utensils and foils for wrapping food, are often protected against corrosion or leaching by surface coatings but can be de-coated by corrosion or wear and tear with exposure to adverse corrosion effects [1, 3-5, 11]. Apart from the use of suitable coatings, the corrosion of cookware aluminium can principally be prevented or mitigated by the use of suitable corrosion inhibitors [1, 24, 25]. The use of corrosion inhibitors is the most versatile and commonly employed technique in aqueous environments because it is economical and reliable if properly applied in the environment at the optimal concentration for the correct exposure duration. Corrosion inhibitors can also be employed to supplement the coating protection to have confidence in the protection [24, 25]. Many organic corrosion inhibitors have been studied and used in very low concentrations of about 0.1-10% of the environmental media to inhibit corrosion of aluminium alloys with very high efficiencies. However, no inhibitor has been found to have all the qualities of an ideal corrosion inhibitor in terms of all requirements such as environmental compatibility, safety to personnel, cost, and availability globally with the capability of inhibiting corrosion by 100% under very minimal or negligible concentration in various possible corrosive media for infinite exposure time [24, 25]. Therefore, there has been a continued search for better corrosion inhibitors among the numerous inorganic and organic chemical substances, plant, and animal extracts for aluminium cookware and other metal types in various aqueous media, but the ideal inhibitor is still far from being found for most metals [24, 25]. Calcium sulphate is an inorganic mineral that is biocompatible with the human body, can be loaded with antibiotics, and is absorbed over time. It is white-solid and odourless. It has a melting point of 1,400°C, a density of 2.96 g/cm³, and a solubility of 2.63 g/L at 25°C [26-33]. It has various uses in the following areas [26-33]:

- In the medical field, bone grafting, medical casting of bones, and splinting.
- Firming agents that help keep foods like canned tomatoes, potatoes, and carrot firm.
- Dough adjustment, dough firming and stabilizing, and providing food for the yeast.
- Due to its high calcium content, nutritional supplements can be added to foods and beverages to fortify them with calcium, especially in products like breakfast cereals and plant-based milk alternatives.
- Coagulant in the production of tofu to help it set and be cut-resistant.
- Provision of nutrients for yeast in yeast foods to aid in yeast leavening.
- The key component in building materials in the form known as ‘gypsum’ in drywall and cement, filler in paints, paper, and other products, and hemihydrate form to create moulds and cast objects.
- Fertilizer to improve soil structure, reduce alkalinity, and provide essential calcium and sulphur for plant growth.

Calcium sulphate occurs in three main forms: anhydrite (CaSO₄), gypsum or dihydrate (CaSO₄·2H₂O), and hemihydrate or plaster of Paris (CaSO₄). Water content distinguishes the three forms. Anhydrite contains no water but is the most soluble form in water, whereas the other two forms contain different levels of water [26-28]. Calcium sulphate is produced as a byproduct of industrial processes such as, flue-gas desulfurization, phosphoric acid production, and certain chemical and wastewater treatment processes. It also comes from natural sources, such as gypsum and anhydrite deposits, which are mined from the earth. It is widely available globally and has an annual production rate of over 260 million tons, with natural gypsum production at approximately

127 million tons per year. It is sometimes found as an extender component in corrosion inhibition primer pigment systems, such as commercial praseodymium-rich primer, which has been qualified for military use [29-34]. Although calcium sulphate is a food substance that is generally environmentally friendly, including to the human body, and widely available from different production sources with its sulphate ion is reportedly capable of suppressing corrosion, it is not commonly known and used as a corrosion inhibitor. It is also not clear from the literature with sufficient experimental facts whether calcium sulphate can inhibit corrosion of commonly used structural materials like aluminium alloys and carbon steel in certain environments [29-33]. The aim of this paper is to investigate the inhibitory potentials of calcium sulphate concentrations of 0-35% in highly acidic media of 2.5 to 3-pH and alkaline media of 10.5 to 11-pH at 30, 50, and 70°C temperatures on the corrosion of cookware aluminium in the media for immersion durations of 1-48 hours using the gravimetric weight loss technique and surface micro-topographical analyses and provide clear experimental facts.

MATERIALS AND METHODS

Materials and equipment used

The following materials and equipment were used for the research work:

Aluminium 6061 sheet of dimensions 2 mm by 20 mm and length 12,000 mm sourced from a Tower Aluminium depot in Lagos, Nigeria, for producing coupons for the tests.

The Japanese-made Shimadzu PDA-7000 metal analyser was used at the R&D unit of DICON, Kaduna, to ascertain the status quo of the sourced Al 6061-alloy rod by nominal composition analysis as a cookware aluminium.

- iii. Reagent-grade hydrochloric acid, was obtained from the chemistry laboratory, NDA, Kaduna, for preparing the corrosive acidic media for the study.
- iv. Reagent-grade sodium hydroxide was obtained from the chemistry laboratory, NDA, Kaduna, for preparing the corrosive alkaline media for the study.
- v. Distilled water was obtained from the chemistry laboratory, NDA, Kaduna, to prepare the corrosive alkaline media to the required pH levels for the study.
- vi. Anhydrite calcium sulphate (CaSO_4) was obtained from the chemistry laboratory, NDA, Kaduna, as the test corrosion inhibitor for evaluation.
- vii. Reagent-grade nitric acid was obtained from the chemistry laboratory, NDA, Kaduna, for cleaning the coupons.
- viii. Vices at the production workshop of the Department of Mechanical Engineering, NDA, Kaduna, Nigeria, for holding materials or coupons during cutting or preparing them.
- ix. A metric steel rule, metal scribe, and hacksaw were used in the production workshop of the Department of Mechanical Engineering, NDA, Kaduna, for measuring, marking out, and cutting out coupons to the required lengths from the Al 6061-alloy sheet.
- x. A Mettler analytical weighing balance with an accuracy capability of up to 0.001 g was used at the chemistry laboratory, NDA, Kaduna, to determine weights of properly cleaned coupons before and after corrosion.
- xi. A grinding machine at the production workshop of the Department of Mechanical Engineering, NDA, Kaduna, was used for removing burrs and surface marks on coupons during initial surface preparation.

- xii. A polishing machine at the production workshop of the Department of Mechanical Engineering, NDA, Kaduna, was used for producing coupons to consistent surface finish.
- xiii. A thermostatically controlled water bath at the chemistry laboratory, NDA, Kaduna, was used to maintain the temperature of the prepared corrosive media constant at various required elevated levels for the study.
- xiv. A desiccator at the chemistry laboratory, NDA, Kaduna, was used for removing moisture from the atmosphere surrounding coupons
- xv. A digital pH meter at the chemistry laboratory, NDA, Kaduna, was used to ascertain the required pH values of the prepared corrosive media.
- xvi. Teflon flasks at the chemistry laboratory, NDA, Kaduna, were used to contain various calcium-sulphate treated prepared corrosive media for immerse-exposing coupons.
- xvii. An optical microscope at the R&D unit of DICON, Kaduna, was used to study the surface micro-topographic patterns of coupons before and after corrosion.
- xviii. Conical flasks at the chemistry laboratory, NDA, Kaduna, for mixing and storing liquid reagents; and measuring cylinders for accurately measuring the required volumes of liquid reagents for preparing required corrosive media for immerse-exposing coupons.
- xix. A Vernier calliper at the production workshop of the Department of Mechanical Engineering, NDA, Kaduna, for accurately determining the dimensions of coupons during their production to the required specifications.
- xx. Pipettes for accurately drawing volumes of reagents to be mixed to required volume and pH levels in preparing the acidic and alkaline media for the study.
- xxi. Sandpapers of 250-, 300-, 400-, and 600-grit mesh sizes were purchased from the Kaduna Central Market for successively polishing coupons to consistent smooth finish.
- xxii. Power supply arrangement for thermostatically and continuously controlling the water bath temperatures to desired temperatures of 30, 50, and 70°C throughout the immersion duration of coupons in the prepared media.

METHODOLOGY

Sample preparation.

The procured aluminium 6061-alloy sheet was first confirmed to meet the nominal composition requirement by the European Union standard for cookware aluminium by analysis using the Japanese-made Shimadzu PDA-7000 spectrometer. The confirmed sheet was handsewn into 450 similar coupons with dimensions 20 mm length, 20 mm breadth, and 2 mm thickness. The sewn-out coupons were first scrubbed with a fine-wire brush to remove machining burrs and contaminants on their surfaces. The coupons were successively polished similarly using 250, 300, 400, and 600 grit papers to remove any roughness and contaminants on their surfaces, rinsed under running water, and dried with a lint-free towel to remove moisture on the surface to an average final dimension of 16 mm length, 16 mm width, and 1.6 mm thickness. Plate 1 shows a sample of the as-sawn-out coupons from the aluminium 6061 alloy sheet.



Plate 1: Top view of a sample of the sawn-out coupons from the Al 6061-alloy sheet

The cleaned coupons were immersed in 70% nitric acid in a glass beaker for 2-3 min, removed, rinsed in distilled water, dried with a clean handkerchief, placed in a desiccator for 1 hour to remove moisture on them, and weighed using the digital Mettler analytical balance to obtain their initial individual weights in milligrams (W_1). The cleaning process was conducted in accordance with the ASTM G1-25, June 2025, standard practice for preparing, cleaning, and evaluating corrosion test specimens [35]. A desiccator was used to store the prepared coupons in a moisture-free condition before the corrosion tests to prevent premature or unwanted reactions with the ambient atmospheric moisture. The desiccator was a sealed specialist container containing silica gel desiccant, which readily absorbs water vapor. Acidic media for the corrosion test were prepared by first pouring 500 mL of the reagent-grade hydrochloric acid into a clean Teflon beaker. Using a pipette, drops of distilled water were gradually added to the acid in the beaker while stirring the content to mix it up and checking its pH with a digital pH meter until a required solution pH value of 2.5 was obtained. The same procedure was repeated to separately prepare the 2.7- and 3-pH media. Reagent-grade sodium hydroxide of 99.9% purity was similarly used with distilled water to prepare alkaline media with pH values of 10.5, 10.7, and 11. The prepared acidic and alkaline media were separately stored in large Teflon containers before use.

Corrosion tests

The prepared acidic solution of pH 2.5 was drawn with a clean pipette and poured into five different 250-mL Teflon containers to equal volumes of 200 mL and separately treated with 0, 0.2, 0.25, 0.3, and 0.35 parts per unit mass of the media using the Mettler analytical weighing balance. This was repeated for each of the other prepared acidic solutions of pH 2.7, 3, and the alkaline solutions of pH 10.5, 10.7, and 11. Three coupons were lifted from the desiccator and immersed in the calcium-sulphate-treated acidic and alkaline media in Teflon containers. Coupons that were immersed in media not treated with calcium served as the control coupons for each set of immersion conditions, while coupons in the media treated with various calcium sulphate concentrations of 20%, 25%, 30%, and 35 % were used to evaluate the effect of the sulphate concentrations in the media conditions on the corrosion of the coupons therein relative to the respective control coupons. The produced media containing various concentrations of calcium in the containers with the coupons immersed in them were sequentially maintained in batches at 30°C, 50°C, and 70°C for immersion times of 1 hour, 2 hours, 3 hours, 24 hours, and 48 hours using a thermostatically controlled 1-kW water bath, which housed the containers under its temperature control. Electricity supply for continuous thermostatic control of the bath temperature at desired values, up to 48 hours, was achieved using supply from the Nigerian national grids, backed up with a 5-kVA uninterruptible power system (UPS) and a 2-kW generator. One coupon was removed from each medium set of conditions at the ends of 1-hour, 2-hour, 3-hour, 24 hour, and 48-hour immersion times, washed with tap water, and scrubbed with a bristle brush under running tap water to remove the corroded particles on it, and finally washed with distilled water and dried in the desiccator for 1 hour in accordance with the ASTM G1-25, 2025, standard practice for preparing, cleaning, and evaluating corrosion test specimens [30]. The final weight of the coupon in mg was determined in each case using the digital Mettler analytical balance. The gravimetric weight loss of each coupon during its immersion duration in the acidic or alkaline media was used to calculate its corrosion penetration rate. The weight loss in milligrams (W) was determined by finding the difference between the initial weight (W_1) and final weight (W_2) in a respective medium was evaluated using Equation 1[34], and Microsoft Excel.

$$W = W_1 - W_2 \dots \dots \dots (1)$$

The obtained W value of each coupon in its relevant medium set of conditions was used to evaluate its corrosion penetration rate (CPR) in mm/yr using Equation 2 [34], and Microsoft Excel.

$$CPR = \frac{87.6W}{\rho At} \dots \dots \dots (2)$$

Where, ρ is the density of the coupon in units of grams per cubic centimetre and equals 2.70 g/cm³, A is the average surface exposure area of all coupons in square centimetres and equals 6.15 cm², and t is the immersion time of the coupon in hours in the respective media conditions. The percentage corrosion inhibition efficiency (IE) of the coupon by the calcium sulphate concentration in each medium condition was evaluated using equation 3 [34], and the Microsoft Excel.

$$IE = \left(\frac{CPR_a - CPR_p}{CPR_a} \right) 100 \% \dots \dots \dots (3)$$

Where, CPR_a and CPR_p are the corrosion rates of the coupon in the absence and presence of a specific calcium sulphate concentration as an inhibitor, respectively.

Microtopographic examination

In addition to the ASTM G1-25 standard practice [30], the ASTM E 3-11, 2025 standard, for the optical microscopy analysis of coupons [36] was used for micro-topographic observations of selected uncorroded coupons and corroded coupons under specified corrosion inhibition levels using an optical microscope with an inbuilt camera. Coupons were progressively ground with emery papers of fine grades 220, 320, 400, and 600 grit sizes using water as a coolant. The samples were polished using 1-micron-sized alumina powder suspended in distilled water, followed by etching in Keller reagent (5% HNO₃ + 3% HCl + 2% HF + 190 ml distilled water) for about 10-30 seconds at room temperature [36].

Inhibitory performance analysis using standard adsorption isotherm model

The corrosion inhibition performance of the calcium sulphate concentration on the cookware aluminium coupons was further investigated by evaluating Gibbs free energy of adsorption at temperatures of 303 K (30°C), 323 K (50°C), and 343 K (70°C) using the obtained experimental corrosion data and the Langmuir adsorption isotherm model, a standard linear adsorption isotherm model, given by equation 3 [37,38].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \dots \dots \dots (3)$$

Where k_{ads} is the adsorption equilibrium constant, C is the inhibitor (calcium sulphate) concentration, and θ is the surface coverage of the cookware coupon taken as the percentage inhibition efficiency in the respective media divided by 100 for each of the three temperatures. Using the adsorption isotherm model, the C values in the acidic media were plotted against $\left(\frac{C}{\theta}\right)$. This was also repeated for the alkaline media. From the plots, it was examined to see whether there was indeed a linear relationship with unit or nearly unit gradient between the two variables in each case in accordance with the ideal Langmuir behaviour or not. From any observation of Langmuir behaviour, K_{ads} was determined from the intercept $\left(\frac{1}{K_{ads}}\right)$ of each of the three plots at different temperatures, otherwise, another model from other isotherm such as Freundlich, Frumkin, Temkin or more advanced models was to be used to obtain the more accurate value for each K_{ads} . With the obtained K_{ads} for each temperature, the standard Gibbs free energy of adsorption (ΔG^*_{ads}) was calculated using equation (4) for the acidic and then alkaline media [37, 38]

$$\Delta G^*_{ads} = -RT \ln(55.5 K_{ads}) \dots\dots\dots (4)$$

Where, R is the universal gas constant (8.314 J/mol·K), T is the absolute experimental temperature in Kelvin (303 K, 323 K, and 343 K), K_{ads} is in units of moles. By this, ΔG^*_{ads} values were obtained and tabulated. Generally, ΔG^*_{ads} values of around -20 kJ/mol or less negative suggest physisorption (physical adsorption), and values around -40 kJ/mol or more negative typically indicate chemisorption (chemical adsorption) involving a stronger bond between the inhibitor molecule and metal surface [37,38].

The Gibbs-Helmholtz equation (5), 5a, and 5b were further used to determine the standard enthalpy (ΔH^*_{ads}), and entropy (ΔS^*_{ads}) of adsorption of the calcium sulphate on the cookware coupons by plotting $\Delta G^*_{ads}/T$ versus $1/T$ using a standard graph paper and obtaining values from the intercept (Int) and slope (S) respectively. Where, ΔG^*_{ads} in this case was the respective average value for the three temperatures for the acidic and alkaline media [37,38]. Using the obtained Int, and

$$\Delta H^*_{ads} = \Delta G^*_{ads} + T\Delta S \dots\dots\dots (5)$$

Where, $Int = \frac{\Delta S^*_{ads}}{R} \dots\dots (5a)$, and $S = \frac{\Delta H^*_{ads}}{R} \dots\dots\dots (5b)$

RESULTS AND DISCUSSIONS

Results

Table 1 shows the nominal composition of the aluminium 6061 alloy, which ascertained it as a cookware aluminium material. The results of the corrosion rates of the coupons after full-immersion exposure durations of 1-48 hours in acidic media of 2.5, 2.7, and 3 pH and alkaline media of 10.5, 10.7, and 11 pH under three different temperature conditions of 30°C, 50°C, and 70°C are presented in Figs. 1a-18a, while the corresponding percentage corrosion inhibition levels by various calcium sulphate concentrations levels of 0, 20, 25, 30, and 35% in the respective media are depicted in Figs. 1b-18b. The SEM-analysed micro-topographies of the as-prepared and uncorroded aluminium coupon, the prepared and corroded coupon in the most corrosive acidic medium, the prepared and corroded coupon in the most corrosive alkaline medium, the most corrosion-inhibited coupon in the acidic media, and the most corrosion-inhibited coupon in the alkaline media by the calcium sulphate concentrations are depicted in Plates 2a-2e, respectively. Results of thermodynamic and kinetic analysis of the inhibitory performances by the calcium sulphate concentrations on the cookware coupons in the study media using Langmuir adsorption isotherm model are presented in Fig. 19a, Fig 19b and Table 2.

Table 1: Nominal composition of the Al 6061 alloy procured for the study

Element	Cr	Si	Mg	Ti	Mn	Fe	Al	Cu	Others
[%] Wt.	0.251	0.748	0.915	0.096	0.047	0.615	96.92	0.296	The balance

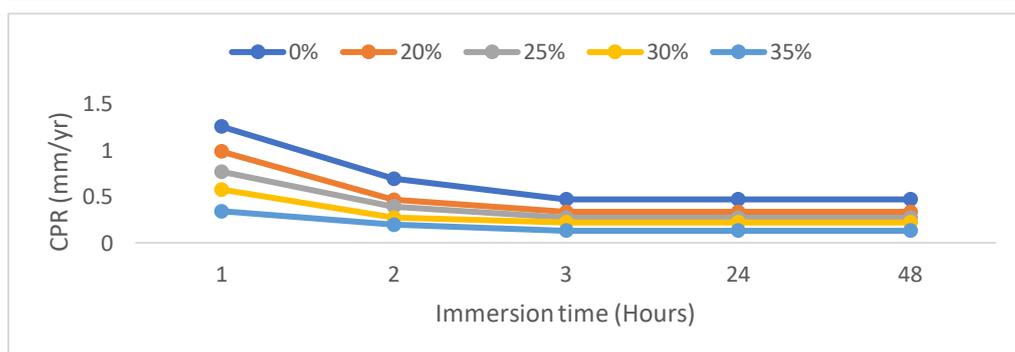


Fig. 1a: CPR of the Al 6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 2.5.

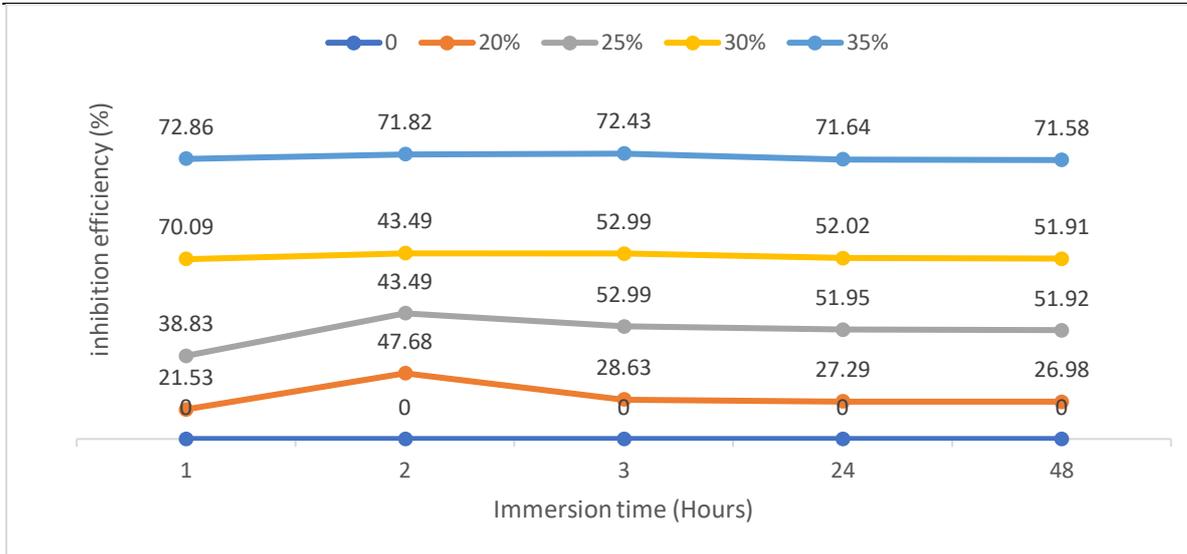


Fig.1b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 2.5-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

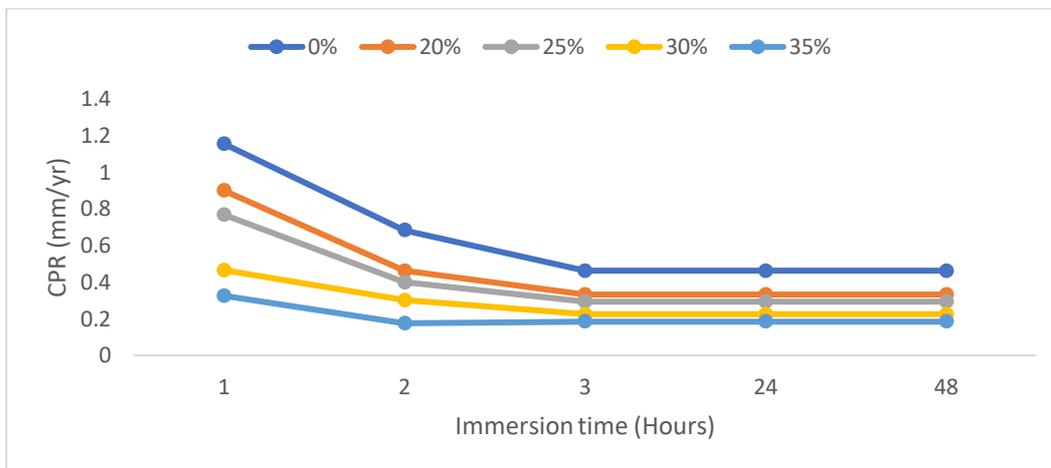


Fig.2a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 2.7.

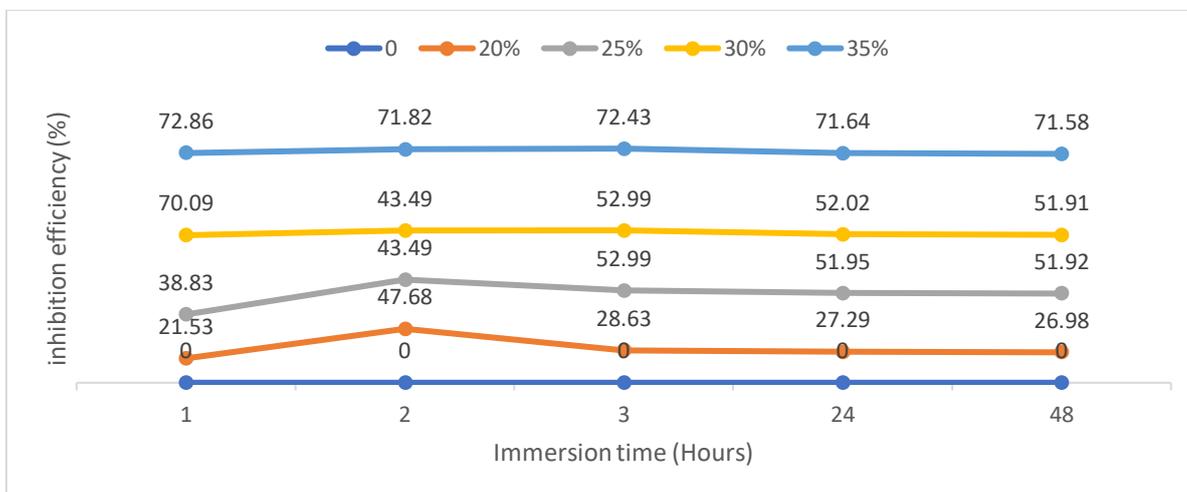


Fig.2b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 2.7-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

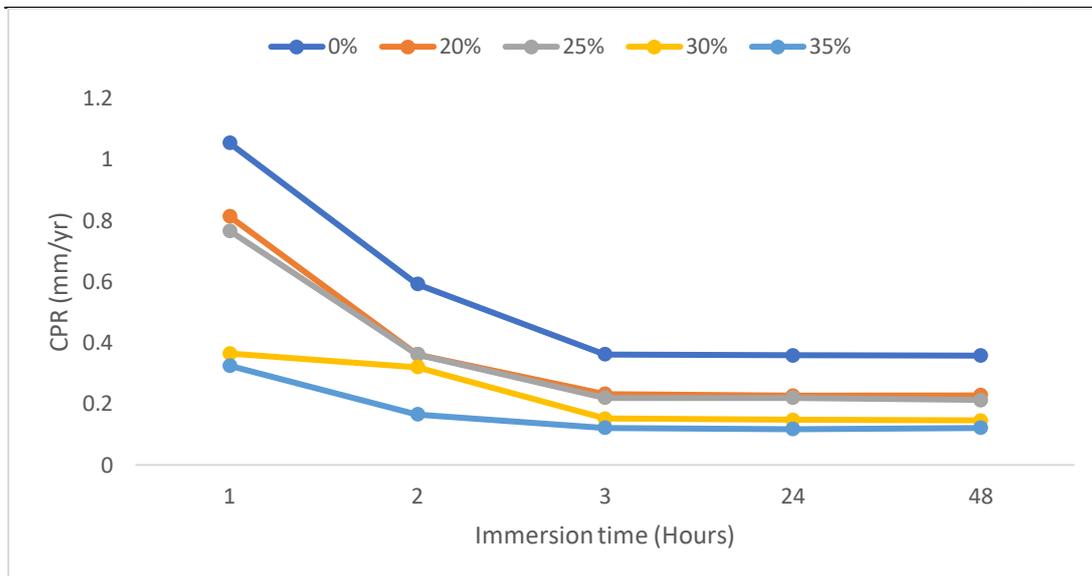


Fig.3a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 3.

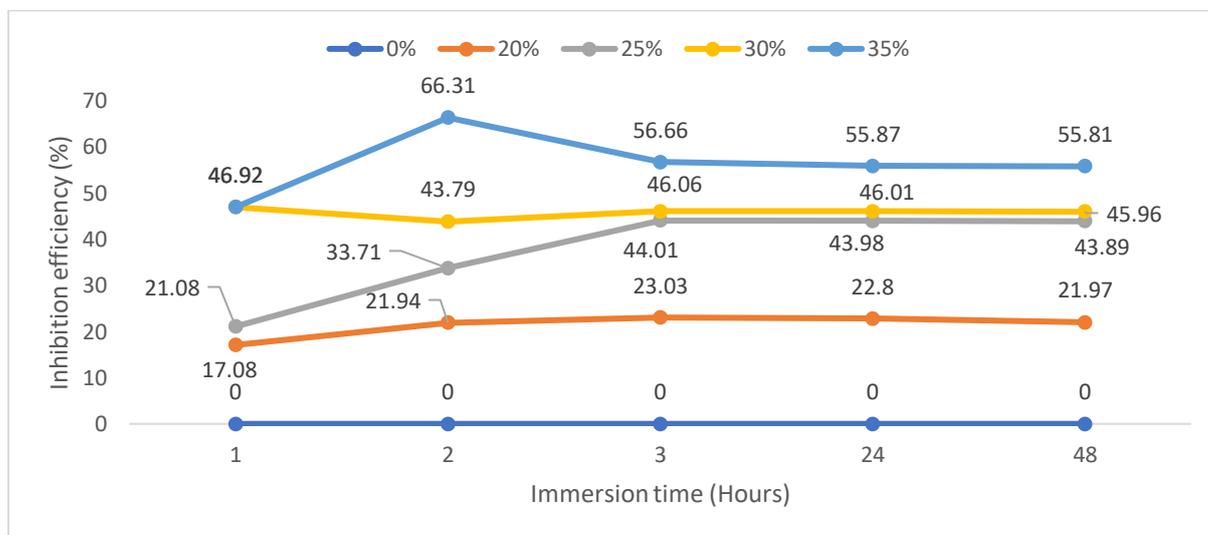


Fig.3b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 3-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

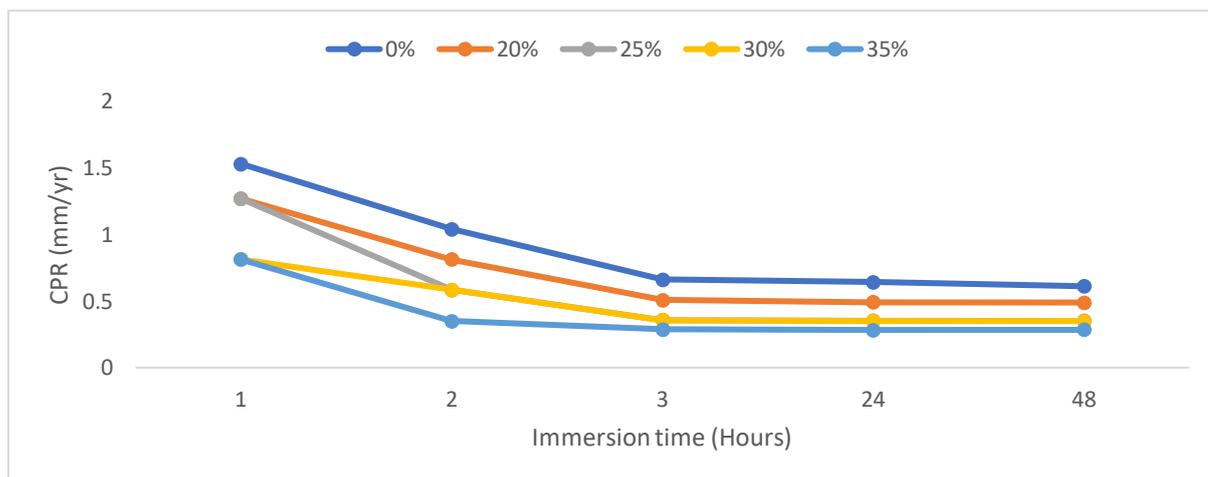


Fig.4a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 2.5

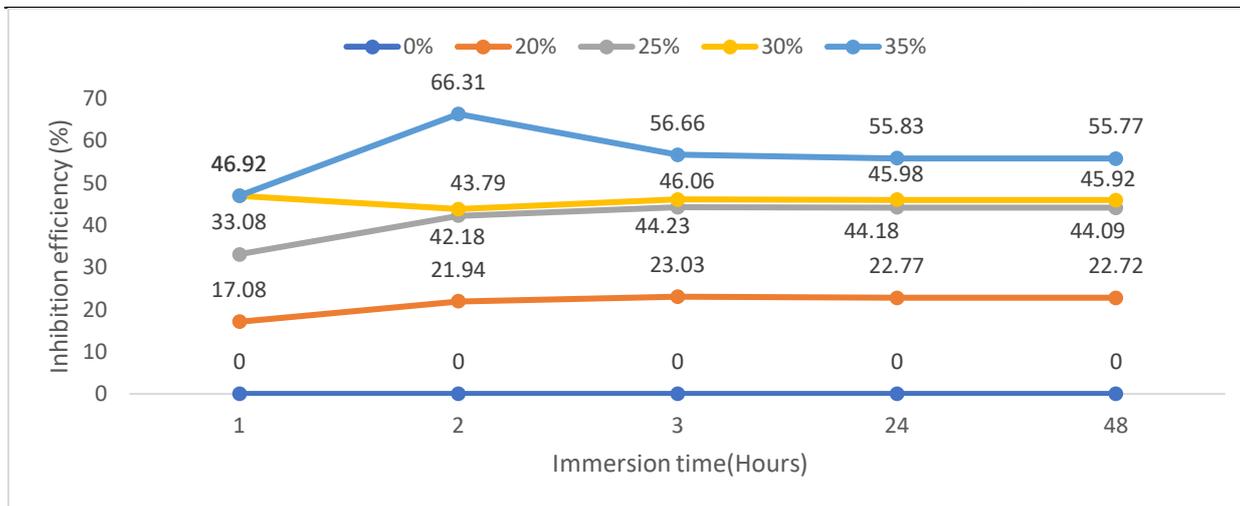


Fig.4b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 2.5-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

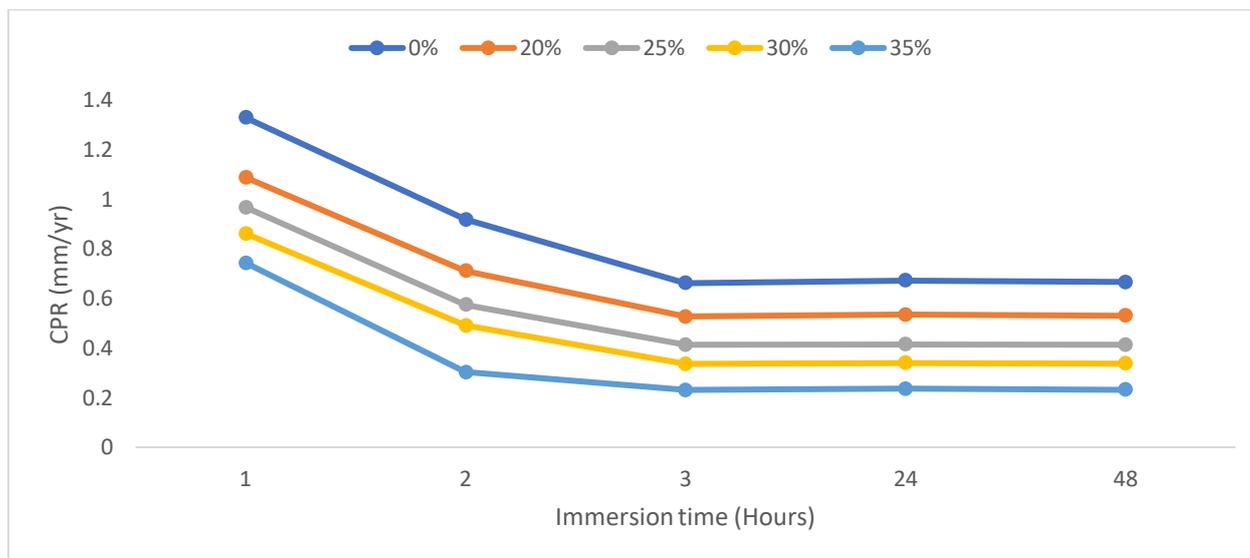


Fig.5a: CPR of the Al-6061 coupons for exposure durations of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 2.7

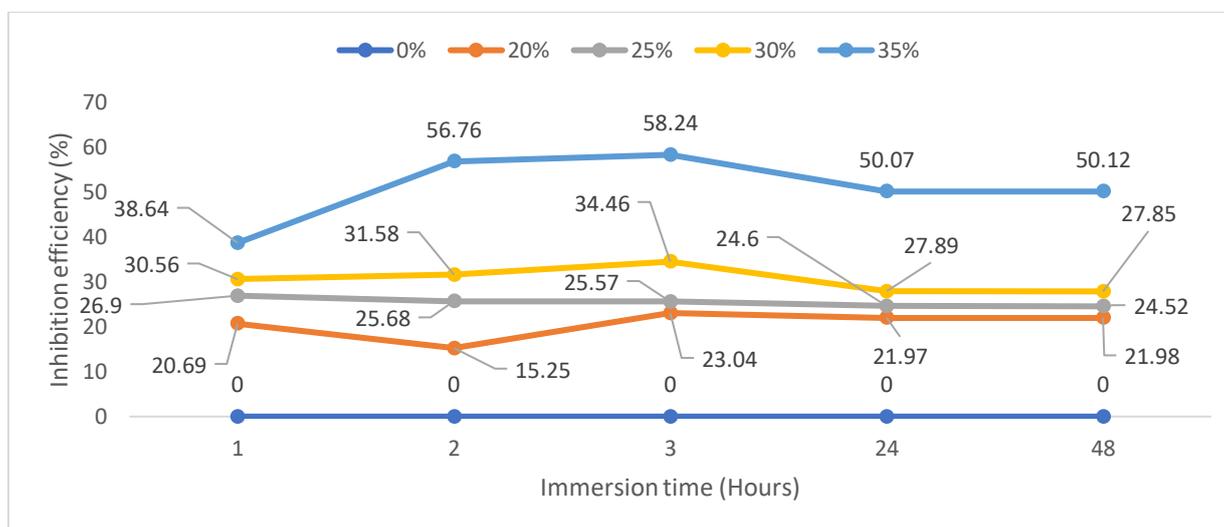


Fig.5b: Corrosion inhibition efficiency (%) the of Al-6061 coupons in various acidic media of 2.7-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate of the media

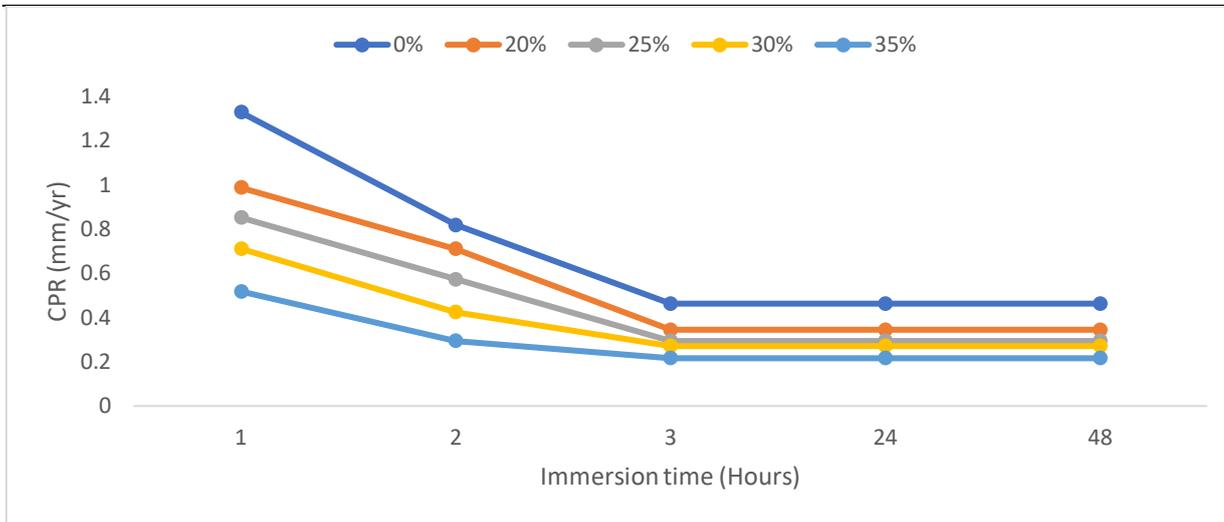


Fig.6a: CPR of the Al-6061 coupons for exposure durations of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 3

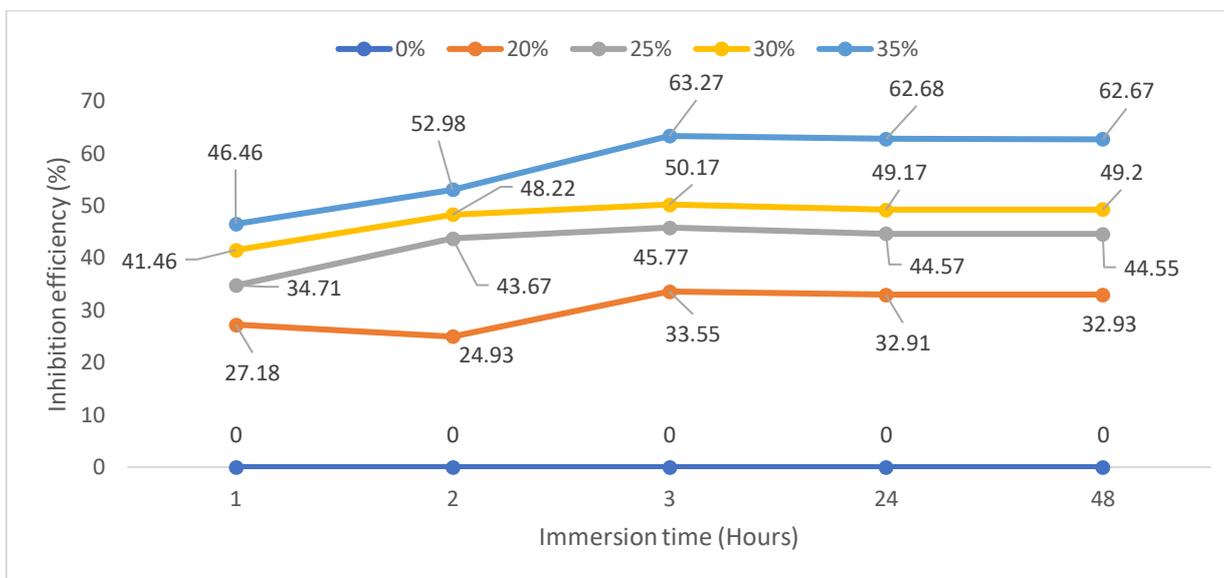


Fig.6b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 3-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate of the media

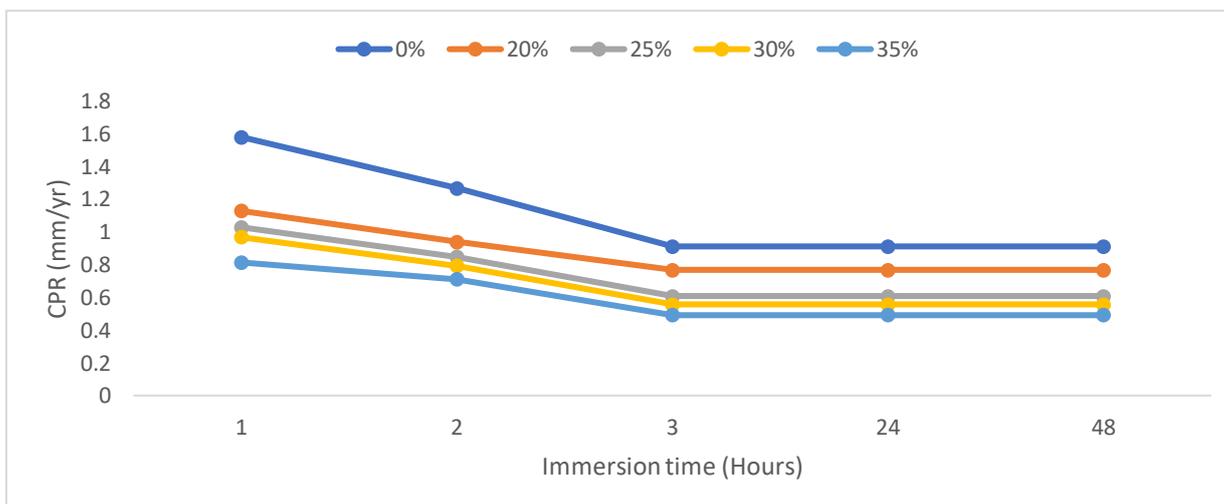


Fig.7a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 2.5

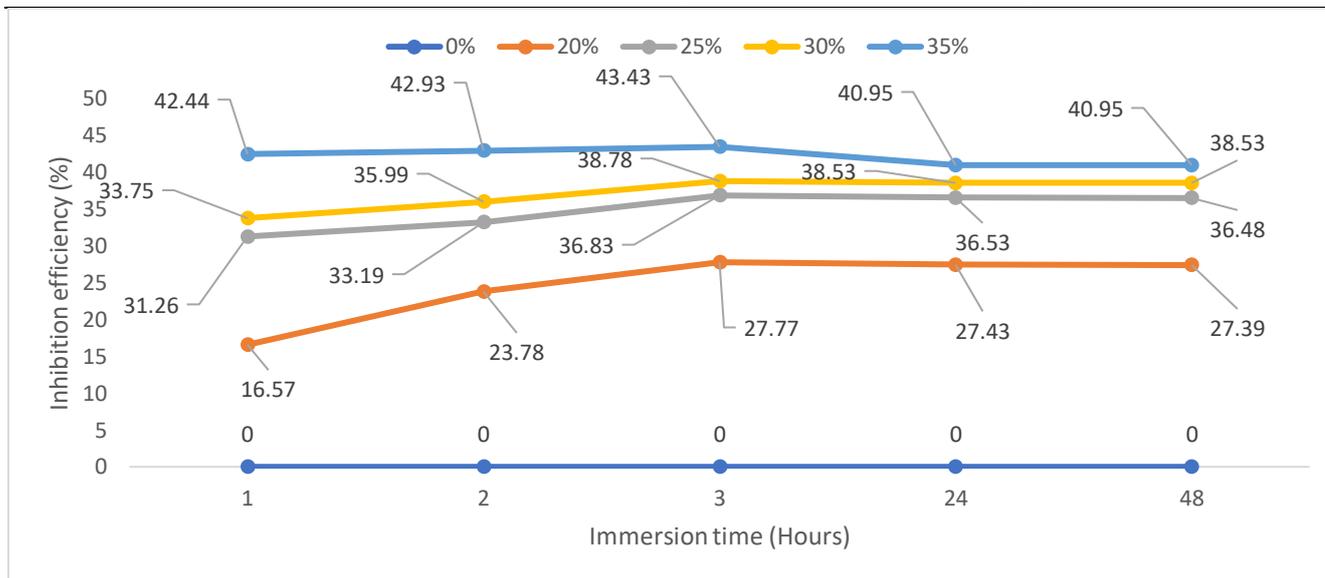


Fig.7b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 2.5-pH and temperature of 70°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

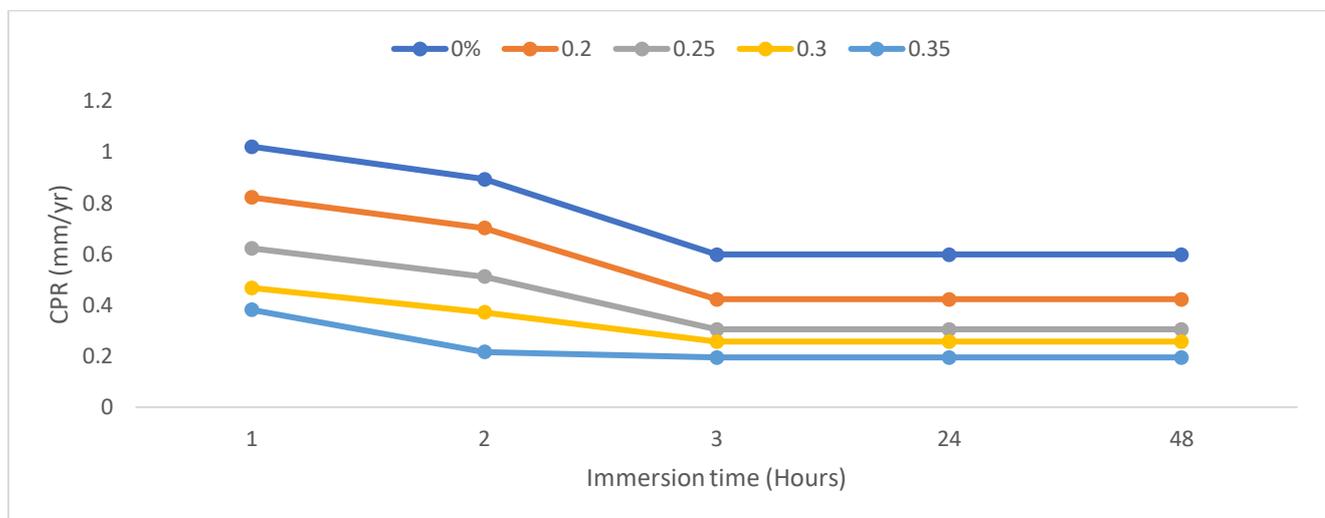


Fig.8a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 2.7

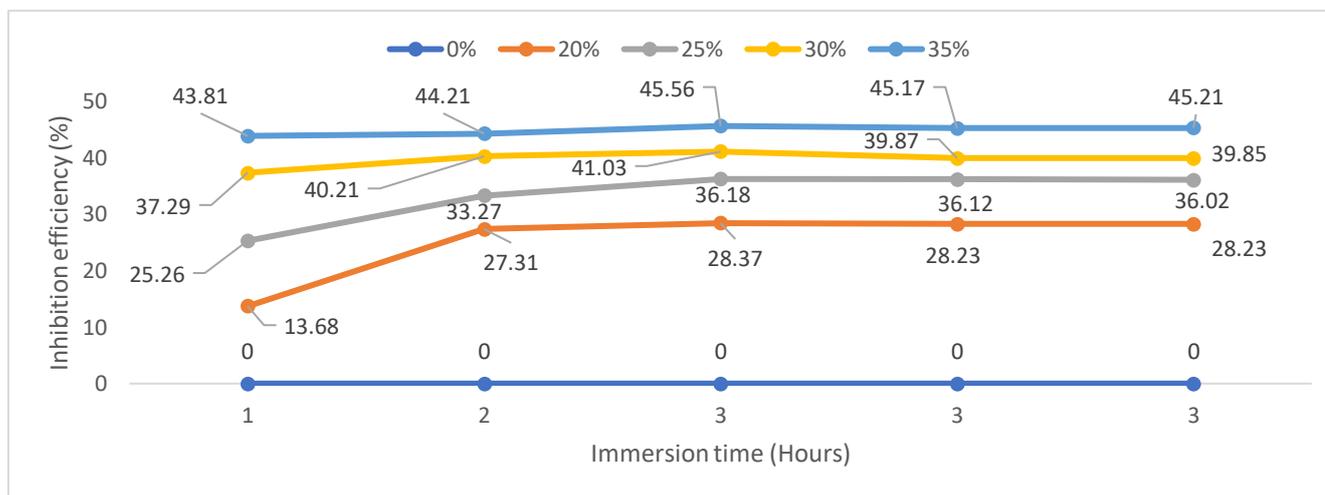


Fig.8b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in acidic media of 2.7-pH and temperature of 70°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

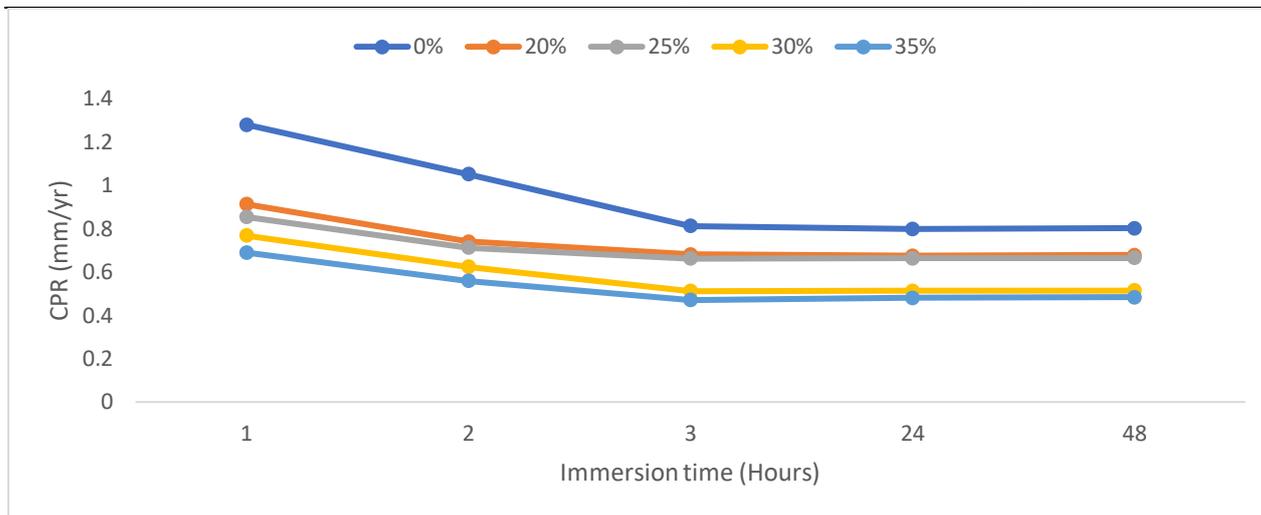


Fig.9a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various acidic media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 3

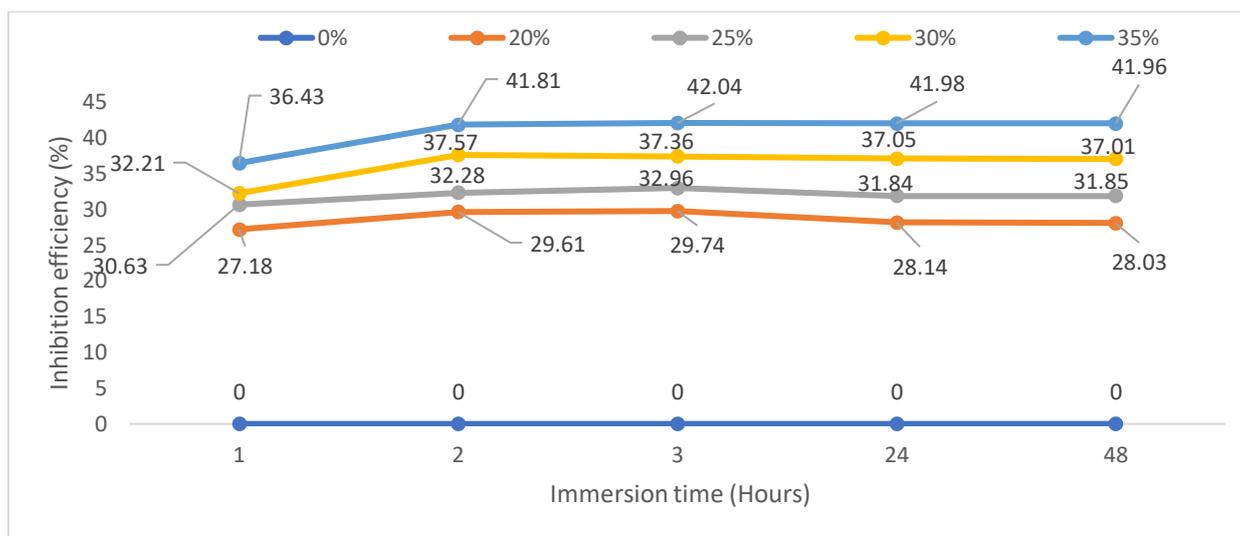


Fig.9b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various acidic media of 3-pH and temperature of 70°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

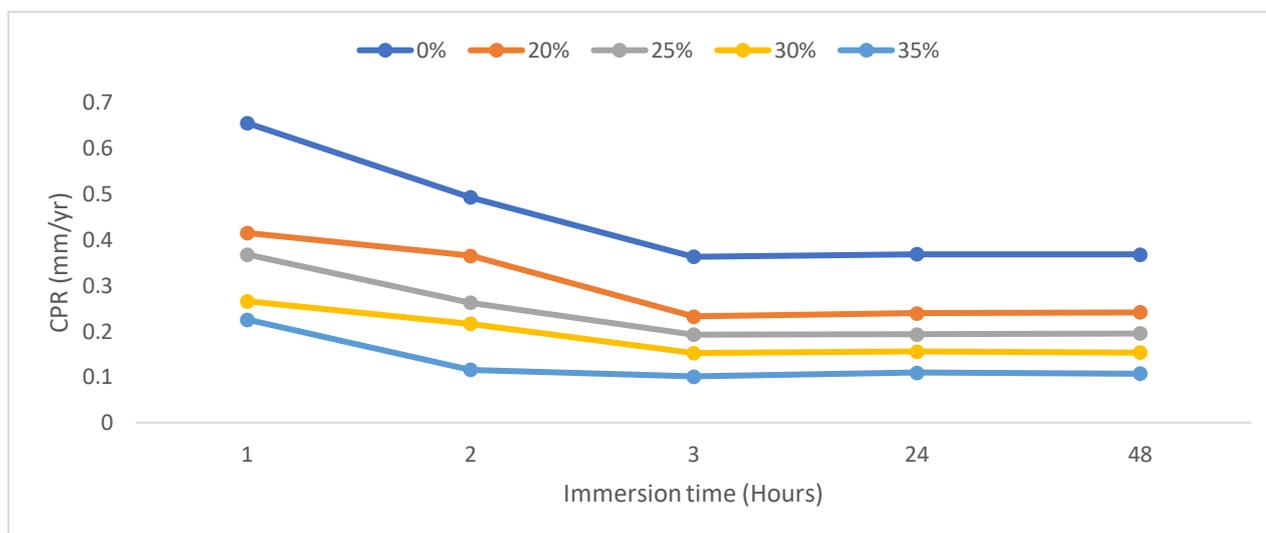


Fig.10a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 10.5

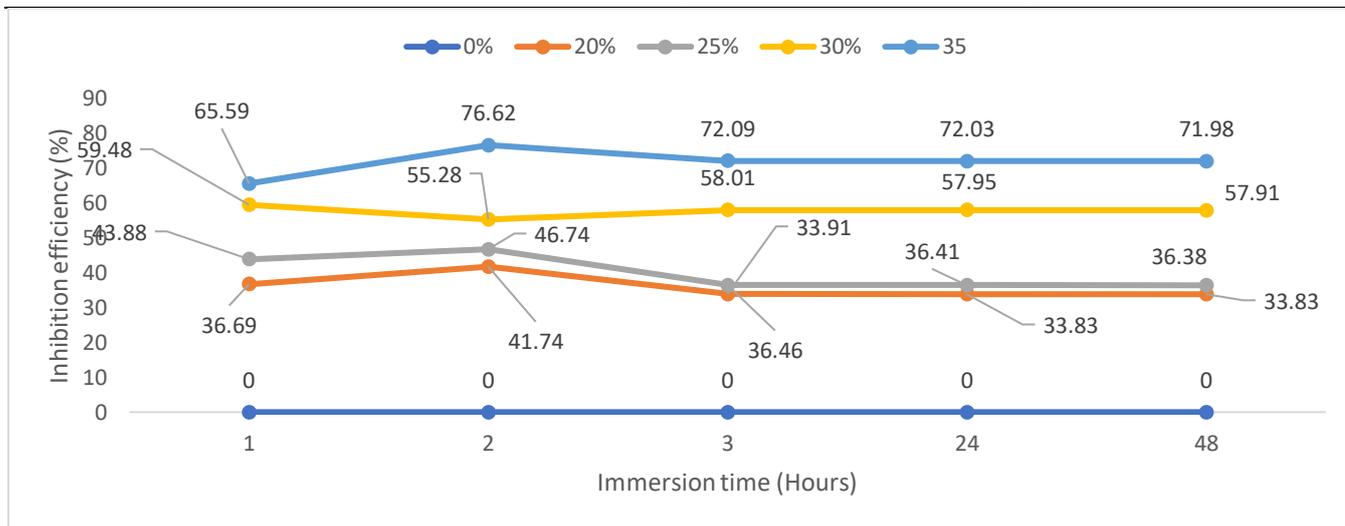


Fig.10b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.5-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

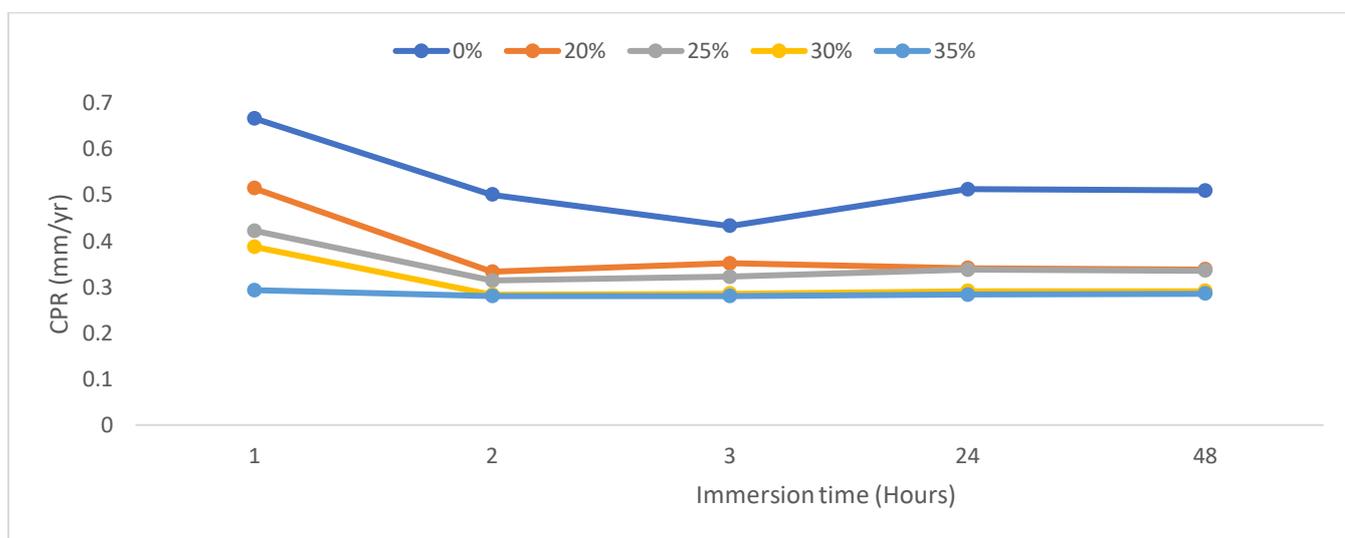


Fig.11a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 10.7

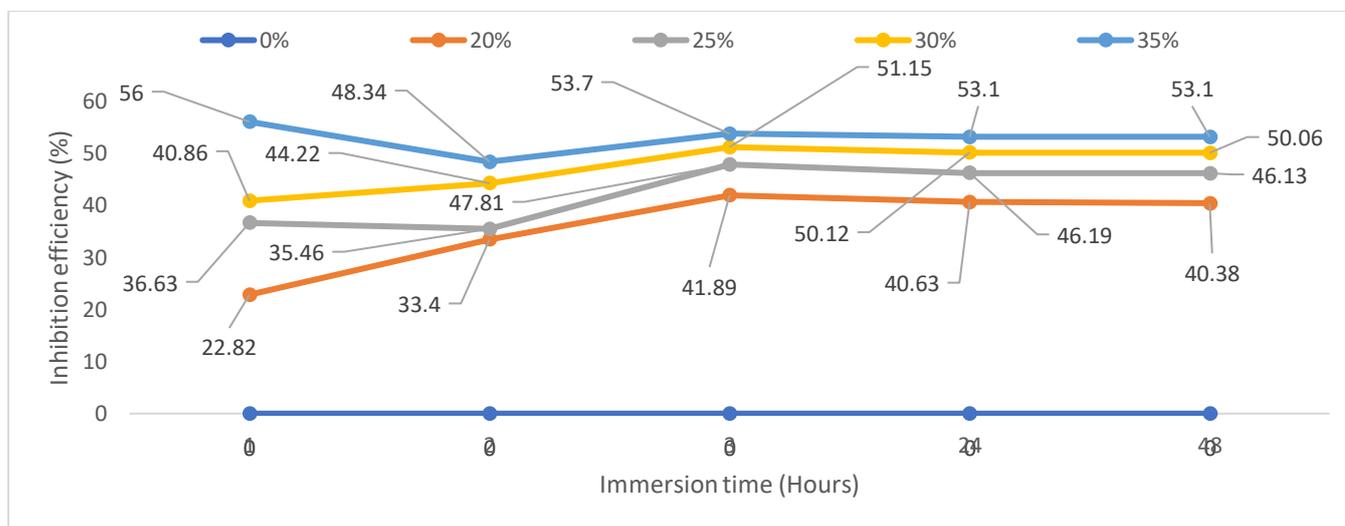


Fig.11b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.7-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

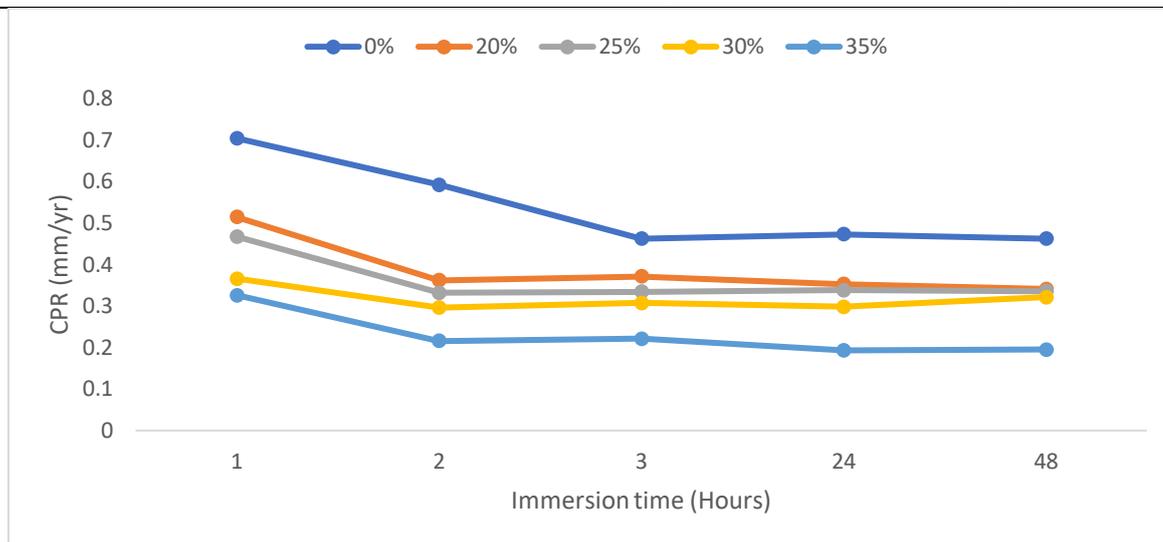


Fig.12a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 30°C and pH of 11

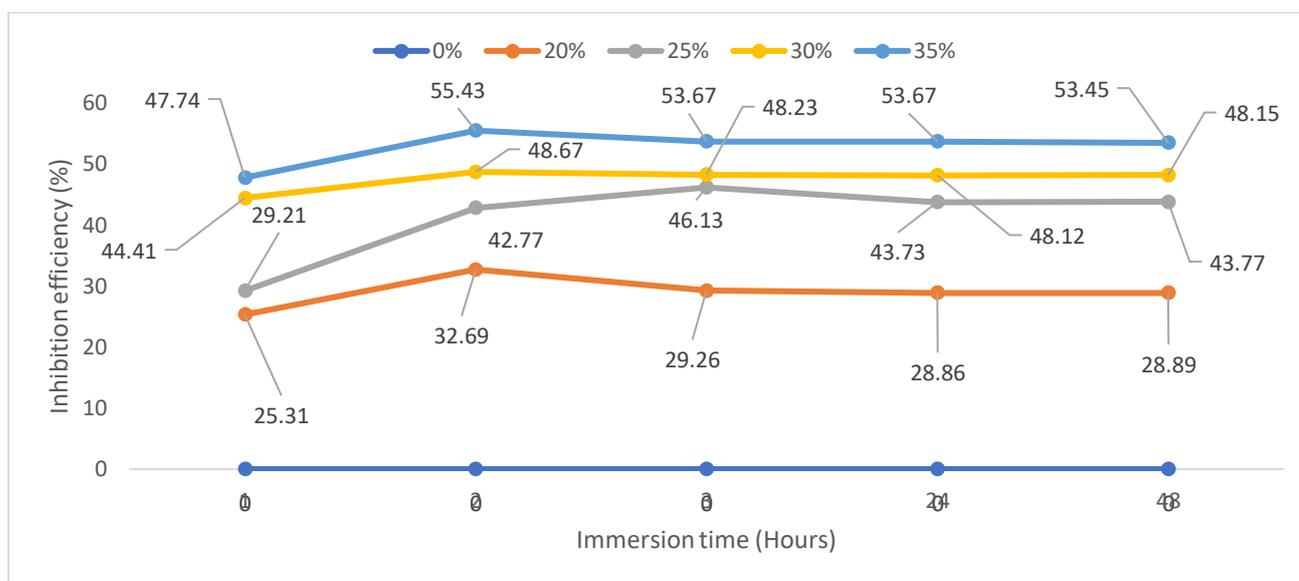


Fig.12b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 11-pH and temperature of 30°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

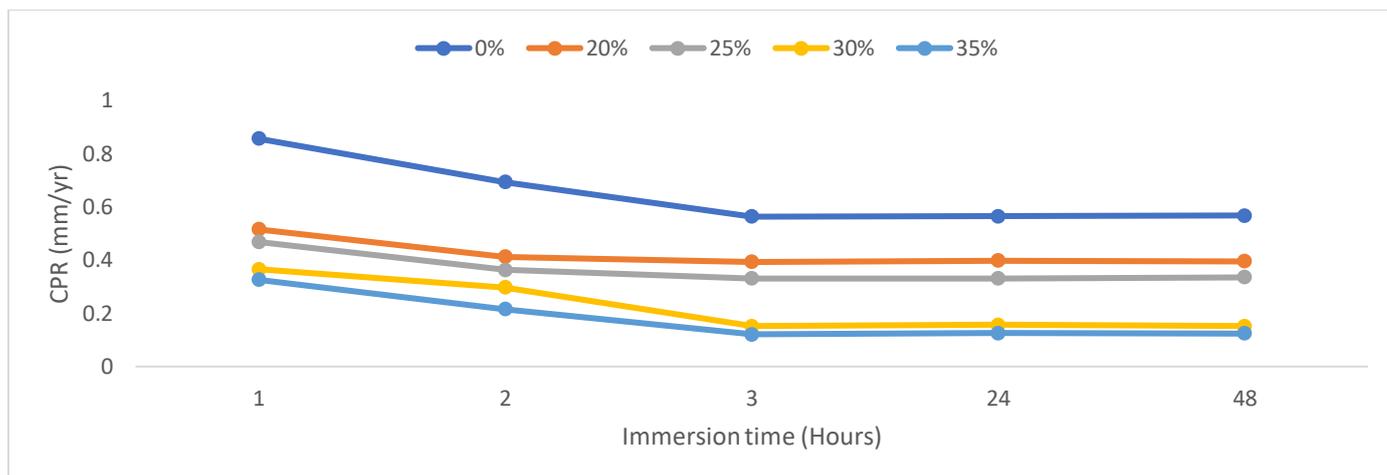


Fig. 13a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 10.5

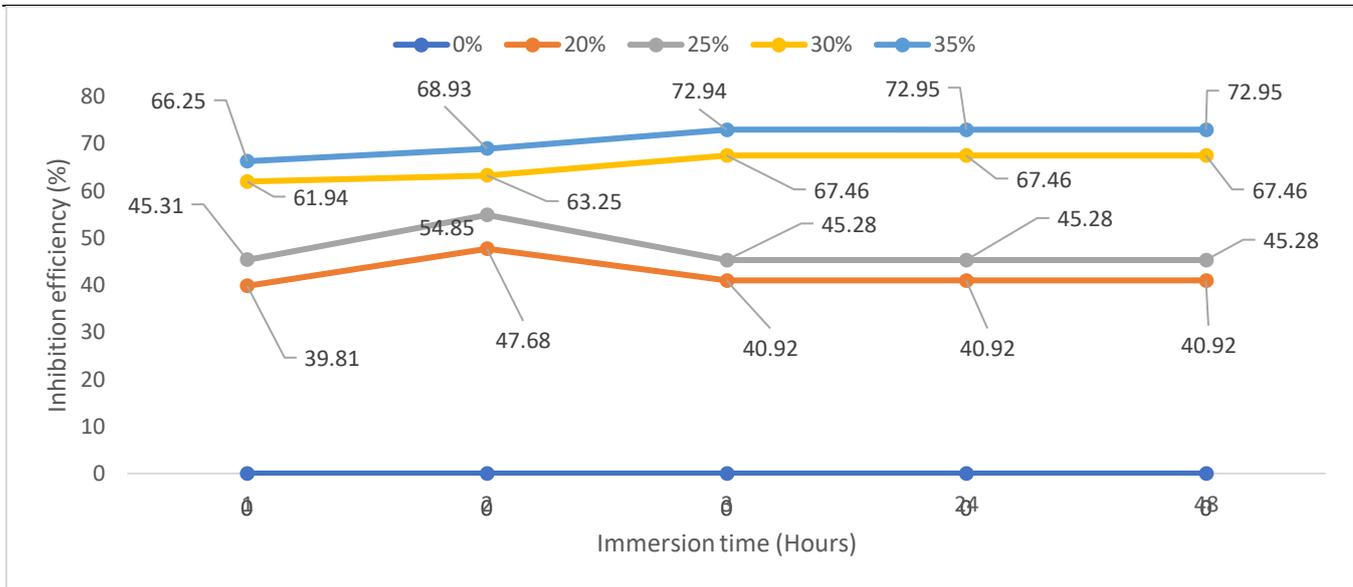


Fig.13b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.5-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

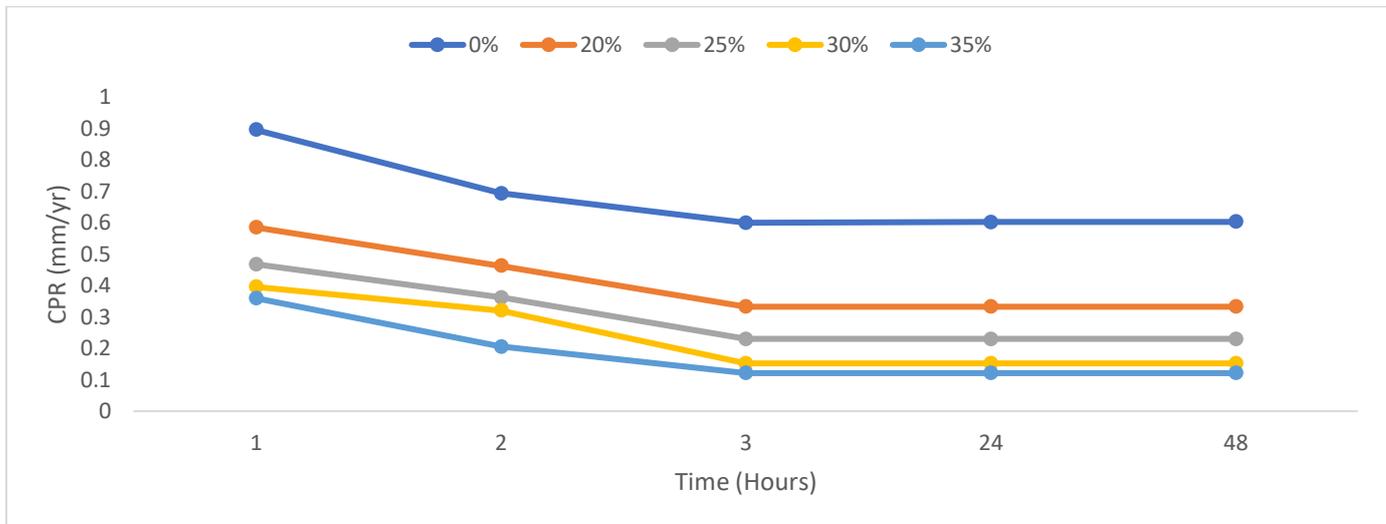


Fig.14a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 10.7

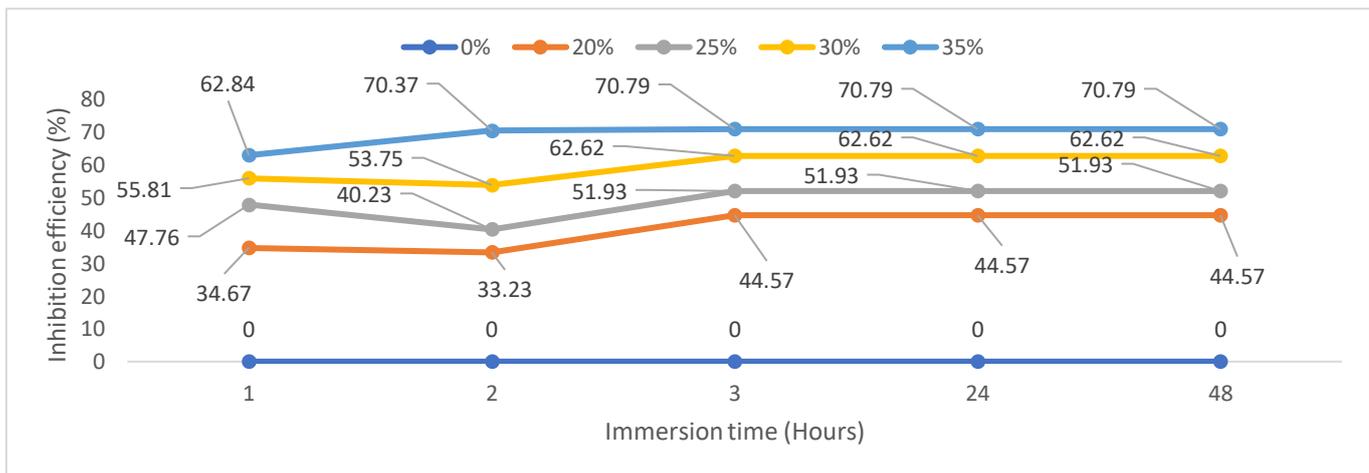


Fig.14b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.7-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

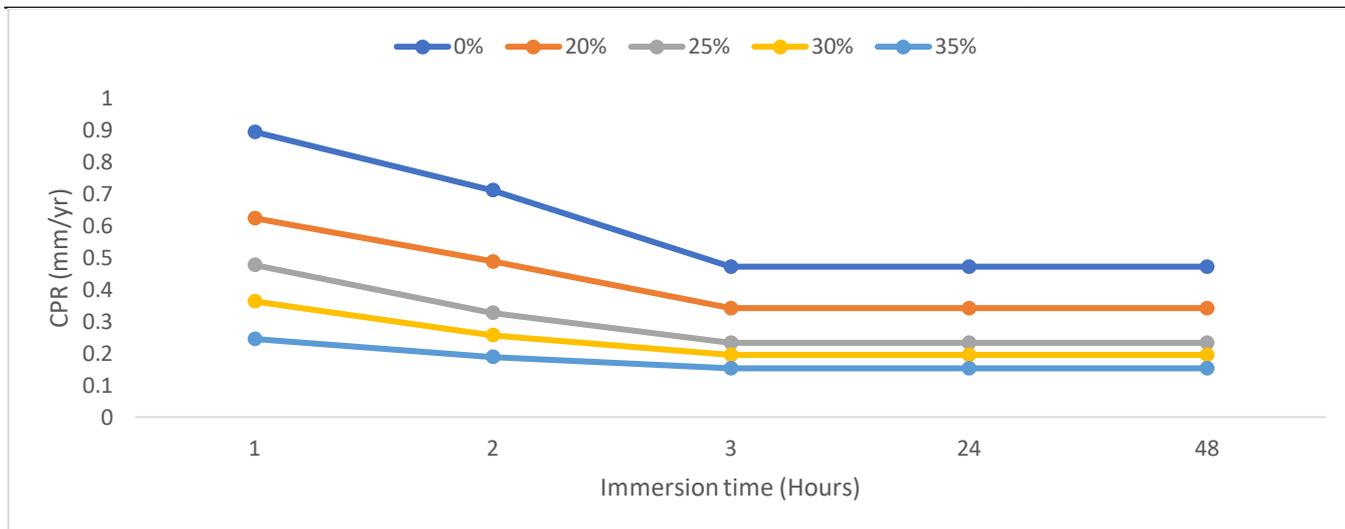


Fig.15a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 50°C and pH of 11

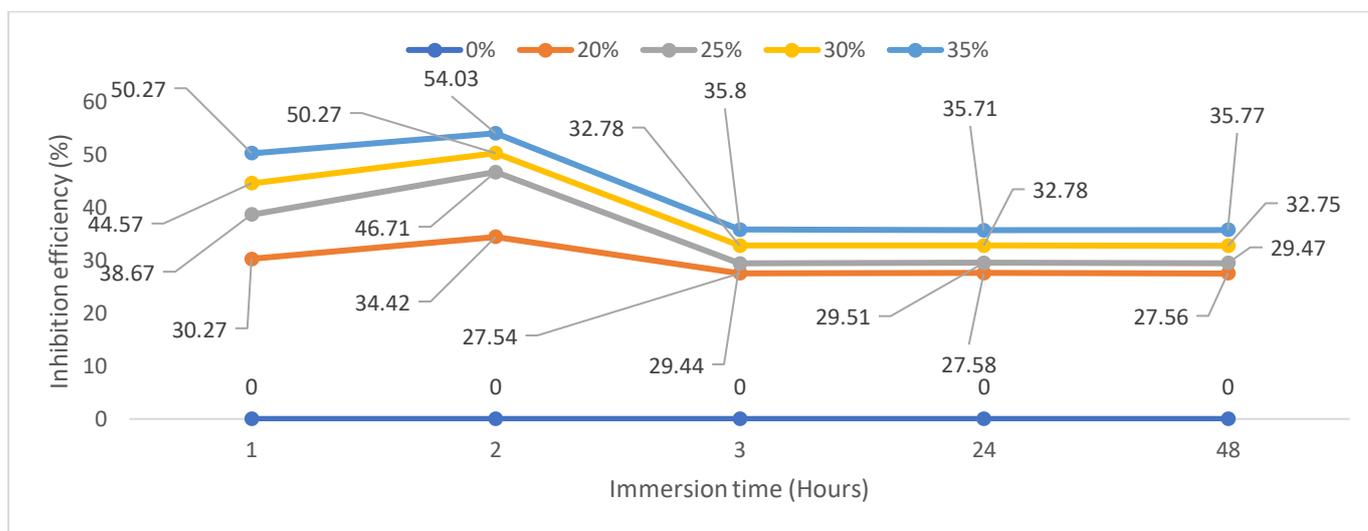


Fig.15b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 11-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

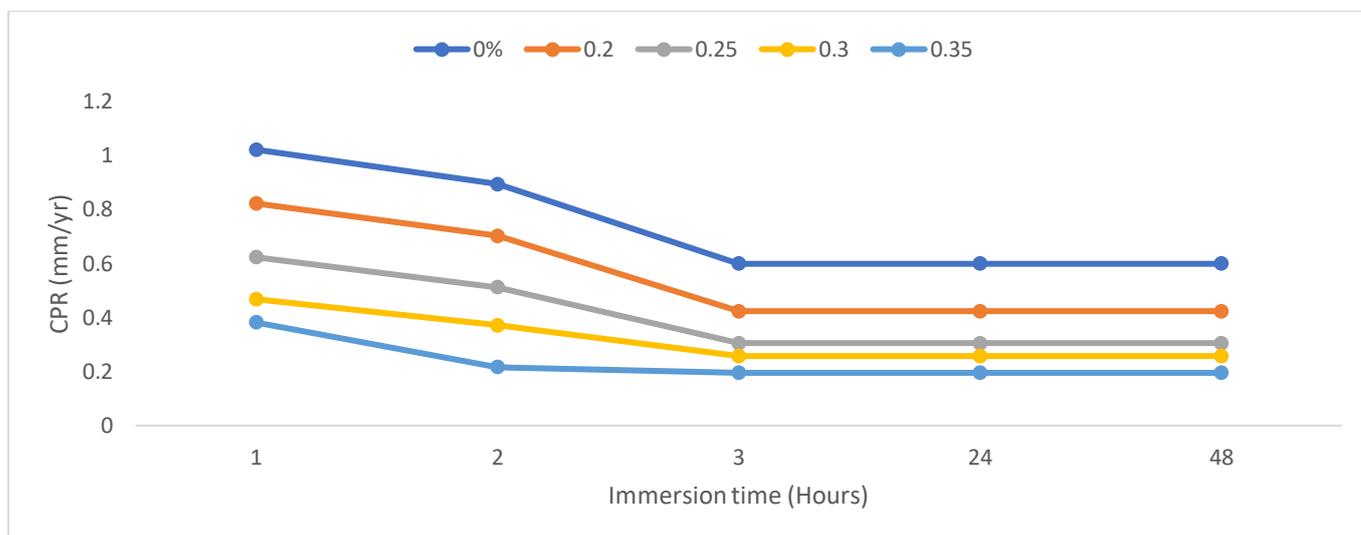


Fig.16a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 10.5

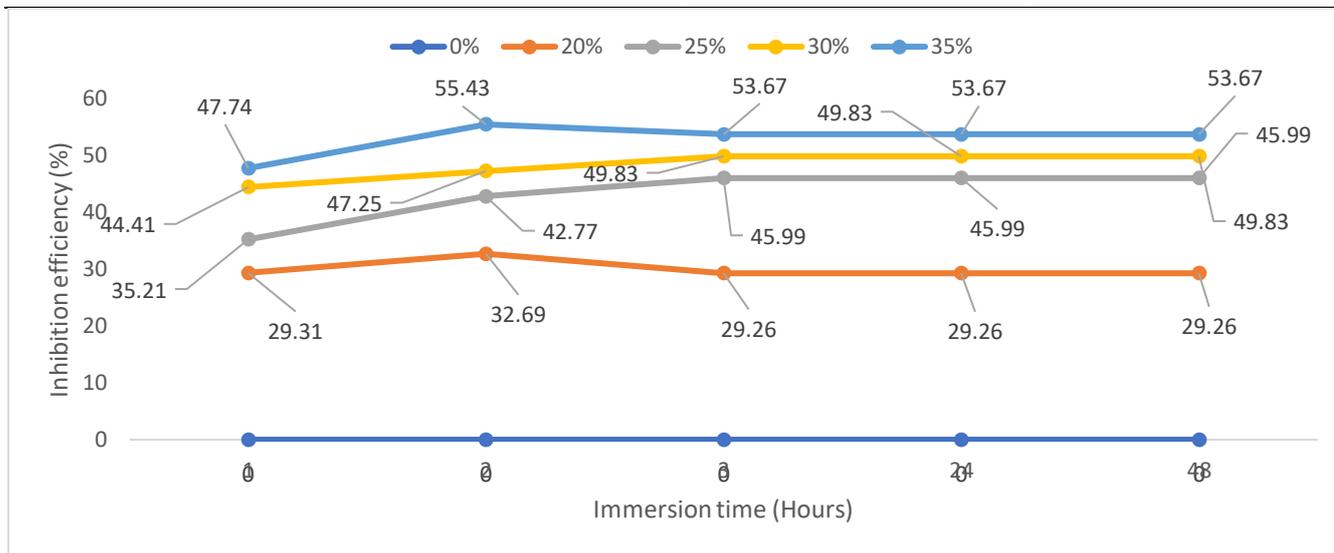


Fig.16b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.7-pH and temperature of 50°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

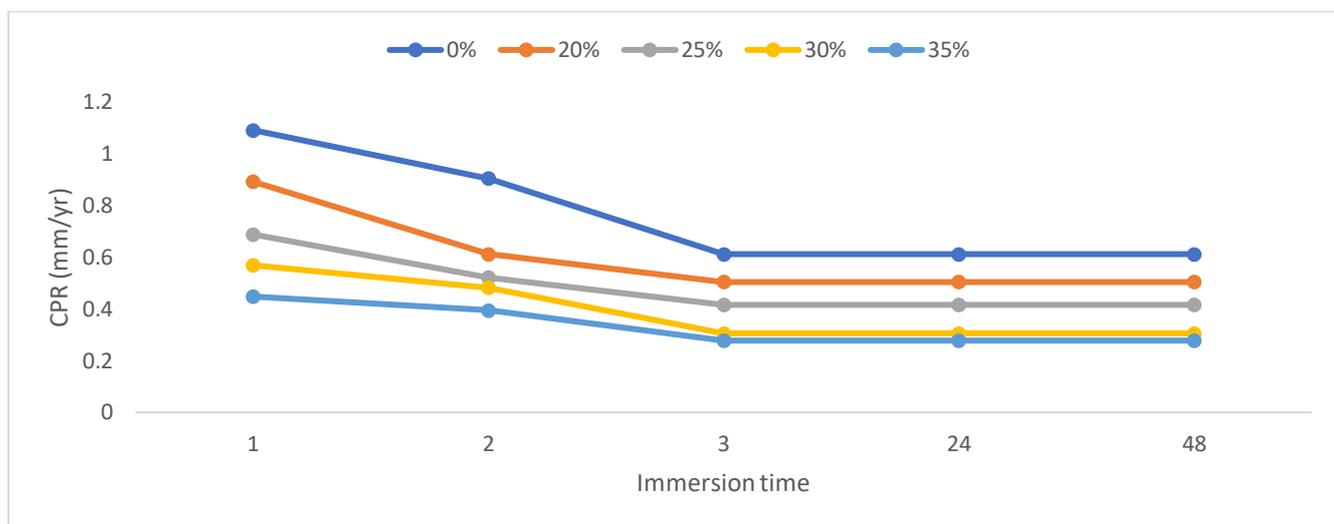


Fig.17a: CPR of the Al 6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 10.7

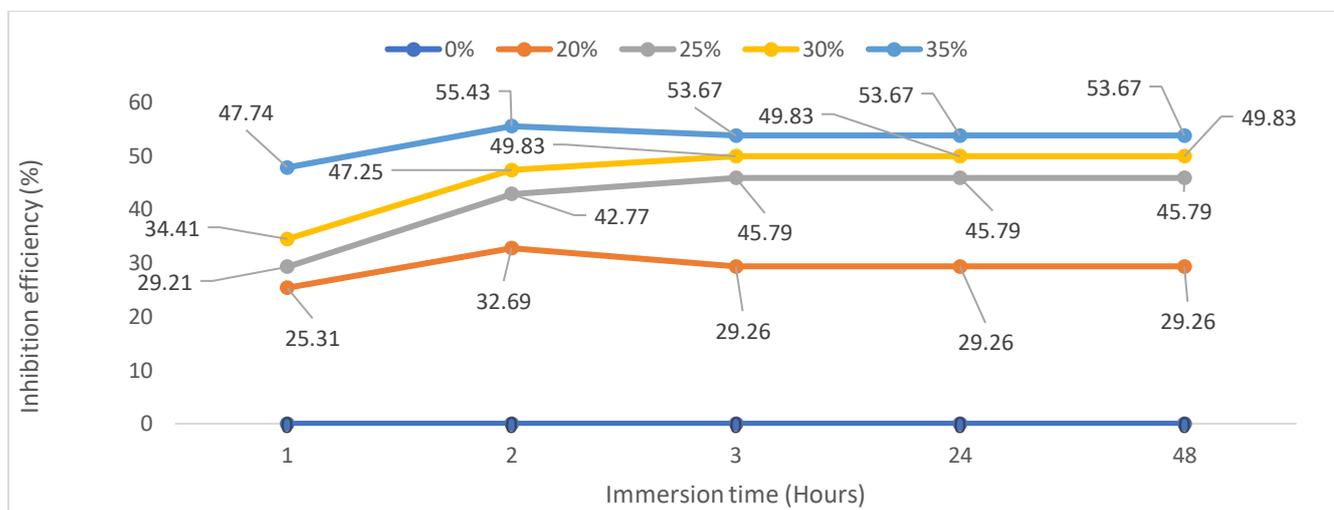


Fig.17b: Corrosion inhibition efficiency (%) of the Al-6061 coupons in various alkaline media of 10.7-pH and temperature of 70°C for immersion time of 1-48 hours by 0-35% calcium sulphate concentrations of the media

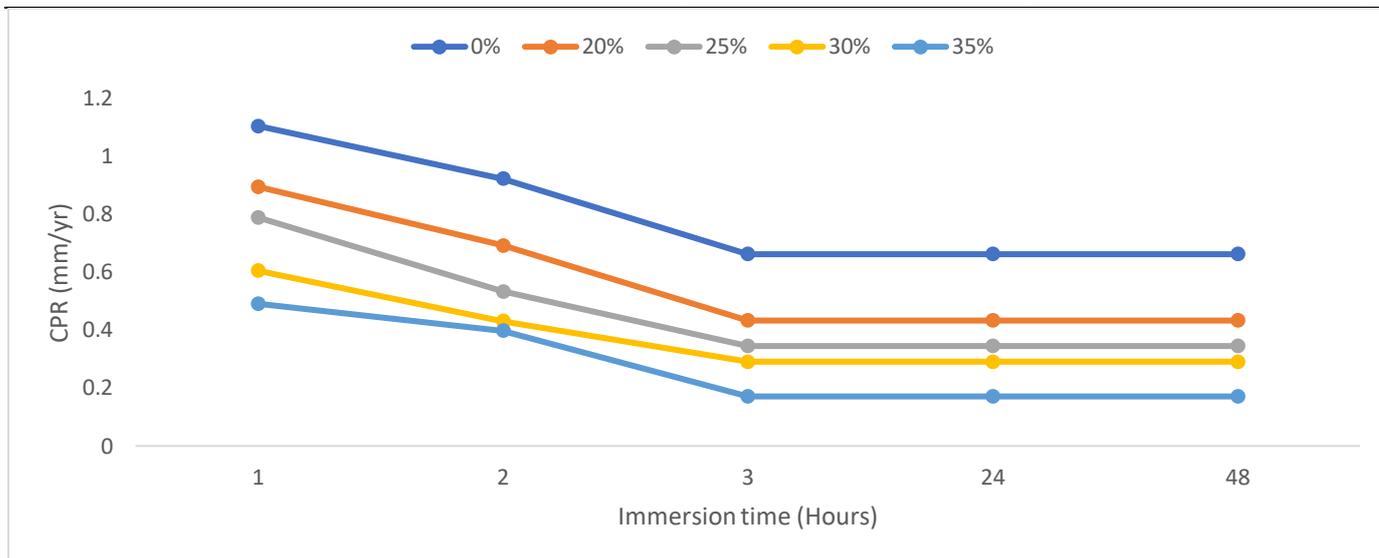


Fig.18a: CPR of the Al-6061 coupons for immersion time of 1-48 hours in various alkaline media containing 0-35% calcium sulphate at a temperature of 70°C and pH of 11

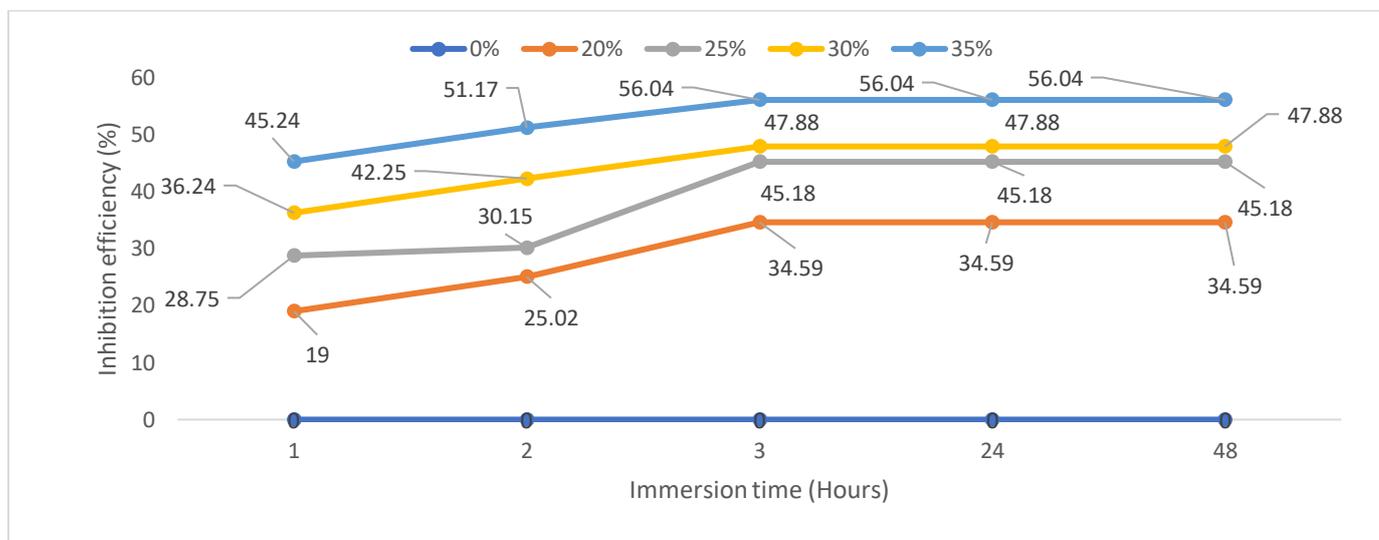
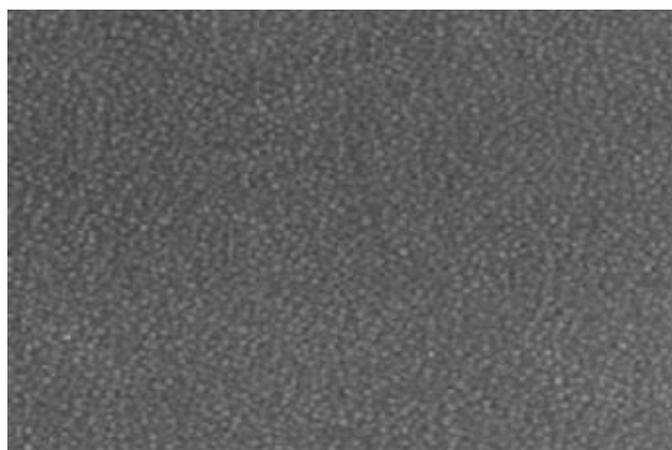
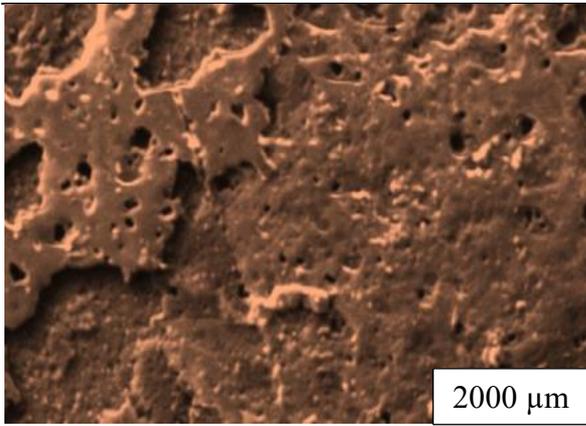


Fig.18b: Corrosion inhibition efficiency of the Al 6061 coupons in various alkaline media of 11-pH at a temperature of 70°C for exposure durations of 1-3 by 0-35% calcium sulphate concentrations of the media

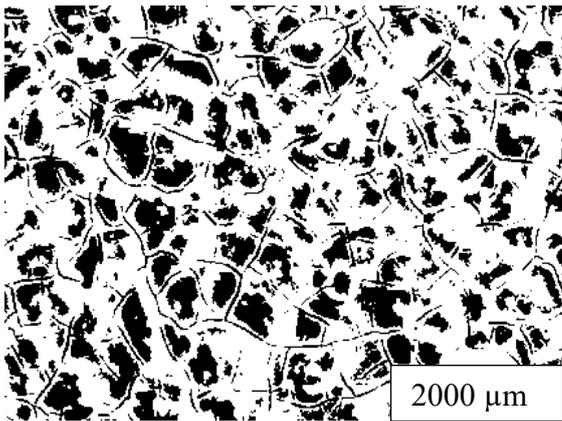


2000 μm

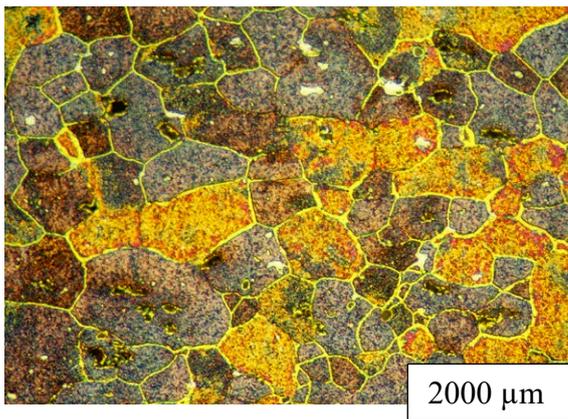
(a). As-prepared uncorroded coupon



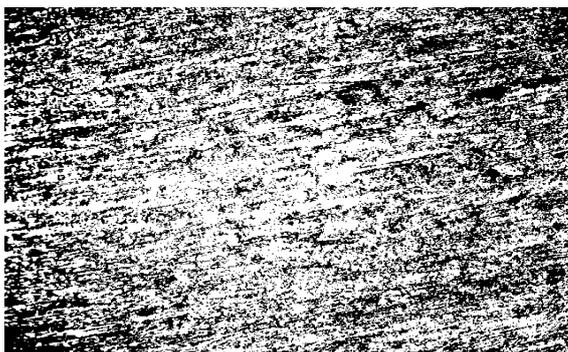
(b). The most corroded uninhibited coupon in acidic media



(c). The most corrosion-inhibited coupon in acidic media



(d). The most corroded uninhibited coupon in alkaline media



(e). The most corrosion-inhibited coupon in alkaline media

Plate II (a-e): Micro-topography statusesque of the aluminum coupons before and after immersion in the various media.

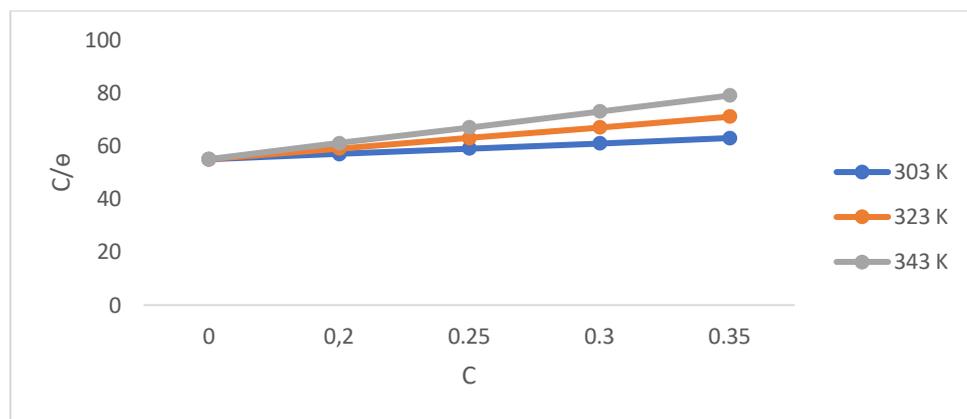


Fig.19a: Patterns of Langmuir isotherm plots of the inhibitory performance in acidic media at various temperatures

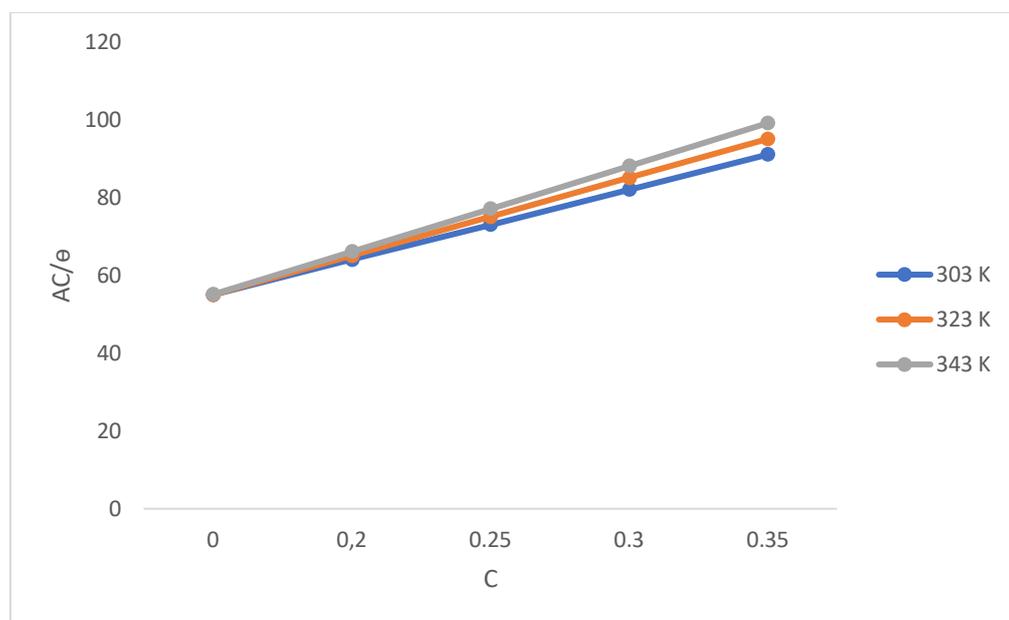


Fig.19b: Patterns of Langmuir isotherm plots of the inhibitory performance in alkaline media at various temperatures

Table 2 Determined adsorption parameters using Langmuir adsorption isotherm model

Medium	Temperature [°K]	K_{ads}	ΔG^*_{ads} [kJ]	ΔH^*_{ads}	ΔS^*_{ads}
Acidic	303	0.01815	-18.37	-30.13 kJ	0.195 kJ/K
	323	0.01817	-22.91		
	343	0.01818	-25.86		
Alkaline	303	0.018166	-20.67		
	323	0.01820	-24.78		
	343	0.01819248	-27.48		

Average value	0.0181764	-23.345		
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DISCUSSION

Table 1 shows that the obtained nominal composition by percentage weights of Cr, Si, Mg, Ti, Mn, Fe, and Cu of the analysed Al 6061 alloy used for the study are all within the allowable maximum content values of 0-0.35, 0-13.5, 0-11, 0-0.3, 0-4, 0-2, and 0-0.6% for the elements, respectively, according to stipulations by the European Food Law standard for cookware aluminium [4, 5, 7].

Figs. 1a-9a show that corrosion rates of the cookware aluminium coupons in the acidic media ranged from 1.528 to 0.47 mm/yr. The maximum corrosion rate value of 1.528 mm/yr in the acidic media occurred in the 2.5-pH medium that was maintained at a temperature of 70°C and not treated with calcium sulphate (Fig. 7a), while the minimum corrosion rate value of 0.121 mm/yr occurred after a 3-day immersion time of the cookware coupon in the 3-pH medium that was maintained at 30°C and treated with 35% anhydrite concentration of the medium (Fig. 3a). Generally, corrosion rates in the acidic media decrease as the media pH increases from 2.5 to 3, and the calcium sulphate concentrations in the media increase from 0% to 35%, but increase as temperature increases from 30°C to 70°C. On the other hand, Figs. 1b-9b depict the corrosion inhibition efficiency values for the results in Figs. 1a-9a, respectively. As shown in Figs. 1b-9b, the highest corrosion inhibition efficiency of 74.34% in the acidic media conditions occurred in the medium with 2.7 pH at a temperature of 30°C, treated with a calcium sulphate concentration of 35% of the medium for a coupon immersion time of 3 hours and remain more less the same for extended immersion durations in the medium, as shown in Fig. 2b. The corrosion inhibition decreases from the highest value with a decrease in the calcium sulphate concentration to 0 value in all the media conditions that were not treated with calcium sulphate, as shown in Figs. 1b-9b. It can also be observed from Figs. 1b-9b that the corrosion inhibition efficiency of the cookware coupons generally decreases with an increase in the operative temperatures of the media but increases with an increase in the coupons' immersion time within 1-3 hours and calcium sulphate concentration of 0%- 35% to a maximum value at the immersion time of 2 hours in the medium condition with a calcium sulphate concentration of 35%. It can also be observed from Figs. 1b-9b that the corrosion inhibition of the coupons generally increases from the 2.5-pH media conditions to higher values in the 2.7-pH media conditions and then decreases in the 3-pH media conditions, indicating that higher acidity media increase corrosion rates and decrease corrosion inhibition efficiency of the cookware aluminium.

As shown in Figs. 10a-18a, corrosion rates of the Al-6061 coupons for immersion durations of 1-48 hours in alkaline media of 10.5, 10.7, and 11 pH at temperatures of 30, 50, and 70°C decreased from the highest value of 1.105 mm/yr in the 11-pH medium that was not treated with calcium sulphate and maintained at a temperature of 70°C (Fig. 18a), to 0.101 mm/yr in the 10.5-pH medium that was maintained at a temperature of 30°C and treated with 35% calcium sulphate at the coupon immersion time of 3 hours (Fig. 10a). The corrosion rates in the alkaline media conditions at the pH values are generally less than the values in the acidic media conditions at the pH values of 2.5, 2.7, and 3. The corrosion rates in the alkaline media gradually decrease as the media pH decreases from 11 to 10.5 and the calcium sulphate concentrations of the media increase from 0 to 35% but increase as the temperature of the media increases from 30 to 70°C. On the other hand, Figs. 10b to 18b show that the highest corrosion inhibition efficiency of the coupons in the alkaline media was 72.95% at a 3-hour immersion time in the 10.5-pH medium maintained at a temperature of 30°C and a calcium sulphate treatment concentration of 30% of the medium, as can be observed from Fig. 13b. The highest corrosion inhibition value of 72.95% decreased to 0 in all the alkaline media conditions that were treated with no calcium sulphate, as can be observed in Figs. 10b-18b. It can also be observed from Figs. 10b-18b that corrosion inhibition of the coupons in alkaline media of 10.5-11 pH by the 0-35% calcium sulphate concentrations of the media generally decrease with an increase in the operative temperatures of the media but increases with an increase in the exposure durations of 1-3 hours of the coupons, and thereafter remain more or less constant. It can also be observed from Figs. 10b to 18b that the corrosion inhibition of the coupons generally decreases from the 11-pH medium conditions to higher values in the 10.7-pH medium conditions and then decreases in the 10.5-pH medium conditions.

According to Raviprabha et al. [34], the corrosion rates of aluminium and its alloys in hydrochloric acid are high, typically in the range of 0.38-24.66 mm/year, depending heavily on the specific alloy, acid concentration, temperature, and exposure time [36-44]. It is also on record that the corrosion rates of aluminium and its alloys in sodium hydroxide are also high with values up to 29.96 mm/yr depending heavily on the specific alloy, sodium hydroxide concentration, temperature, and exposure time [45, 46]. Finally, it can be observed from Figs. 1a-18a that increasing the exposure duration increases the corrosion rates of the cookware coupons in the acidic and alkaline media conditions. For the same immersion time, elevating the temperature of the acidic and alkaline media increases corrosion rates of the coupons therein relative to similar media conditions. This almost results in a decrease in corrosion inhibition efficiencies in similar media conditions treated with similar concentrations of the anhydrite, as shown in Figs. 1b-18b. Generally, an increase in environmental temperature increases the corrosion rate of metals [21-23]. This is because an increase in temperature [21-23]:

- Increase the kinetic energy of molecules and accelerate the reaction rates.
- Provides more energy for chemical reactions and causes molecules and ions to move faster and collide more frequently and effectively to increase the electrochemical corrosion reaction rates.
- Enhances the diffusion rates of corrosive agents, such as oxygen and ions, through the media or protective layers of the coupons, allowing the molecules to reach the coupon surface more quickly.
- Causes thermal expansion of the coupons, which might have widened existing microscopic cracks or fissures on their surfaces, allowing corrosive agents to penetrate deeper and increase their corrosion rates.
- Facilitate the formation of a thermos-galvanic corrosion cell on the coupons' surfaces due to temperature differences across the surfaces in the acidic and alkaline media, such that the hotter spots on their surfaces act as anodes and experience an increase in corrosion rates.
- Enhances the ability of some ions, such as chloride, in acidic media to break down protective natural formations on the coupons, resulting in localized and intensified corrosion, such as crevice or pitting corrosion.

In some cases, an increase in temperature does not cause a significant increase in the corrosion rate or slightly reduces it. This is attributed to a decrease in the solubility of some corrosive gases, such as oxygen, in the aqueous media above a certain temperature threshold, leading to a stagnation or reduction in the corrosion rate due to inadequate oxygen supply [21-23, 47]. According to Arrhenius's rule, the rate of corrosion can nearly double for every 10°C increase in temperature [21-23, 47]. However, the results depicted in Figs. 1a-18a does not indicate so. This can be so because the overall effect in the acidic and alkaline media scenarios might be complex and depend on many other unforeseeable variables.

It can be observed from Figs. 1a-18a and 1b-18b that as the calcium sulphate concentration increases in both the acidic and alkaline media conditions, the corrosion rates of the coupons therein decrease and their corrosion inhibition efficiencies increase for the same media conditions. This trend is attributed to the increased adsorption of the sulphate molecules onto the surface, resulting in the formation of a more compact and stable protective film on the coupons with the physical separation from the media conditions and blockage of active corrosion sites.

Corrosion inhibitor concentrations for aluminium vary significantly depending on the specific inhibitor and the environment's pH level [48-54]. Organic compounds are frequently used to inhibit corrosion in acidic media, such as HCl and H₂SO₄ media. Many of these compounds can be used at very low concentrations of 0.01% to 1% in acidic and alkaline environments to inhibit the corrosion of aluminium with very high corrosion inhibition efficiencies of over 90%. However, the highest corrosion inhibition efficiency of the cookware aluminum coupons by the calcium sulphate concentrations of 20-35% in the acidic and alkaline media in this study is only 74.24%, which is very low. This indicates that for the highest comparable corrosion inhibition of aluminum cookware in acidic and alkaline media with organic corrosion inhibitors, the calcium sulphate concentration in

the media needs to be greater than 35%, and this may not make calcium sulphate an economical corrosion inhibitor for the aluminum cookware. This indicates that calcium sulphate can only be reliably used to protect cookware aluminum in acidic and alkaline media with 100% inhibition efficiency when in much higher concentrations of the media [48-54].

Plate IIa shows that the micro-topography of the as-prepared and uncorroded coupons looks smoother, more uniform, shinier, brighter, and more polished than all the corroded coupons. The micro-topography of the most corroded uninhibited coupon in the acidic media shown in Plate IIb looks roughest, least uniform, least shiny, least bright, and least polished, with signs of deeper pits or craters and greater flaking and blistering than all the other coupons, as can be observed from Plates IIa-IIe, while the surface quality of the most corroded uninhibited coupon in alkaline media shown in Plate Id is comparatively better than that in the acidic media. Plate IIc shows that the micro-topography of the most corrosion-inhibited coupon in the acidic media appears smoother and more uniform than the most corroded coupons in both the acidic and alkaline media but less in quality than the most corrosion-inhibited coupon in the alkaline media. This indicates that differences in the corrosion levels of the aluminium coupons manifested in their surface deterioration levels and the levels of corrosion inhibition by the calcium sulphate concentrations in the media. However, the micro-topography of the most corrosion-inhibited coupon in the alkaline media, has an edge in better surface roughness levelling, uniformity, and consistency than the microstructure of the most corrosion-inhibited coupon in the acidic media.

It is clear from Fig. 19a, Fig. 19b, and Table 2, that the adsorption of calcium sulphate on the cookware coupons in the acidic and alkaline media conditions is in accordance with the Langmuir adsorption isotherm with Gibbs free energy, enthalpy, and entropy of adsorptions of -23.345 kJ, -30.13 kJ, and 0.195 kJ/K, respectively [37, 38]. It is therefore evidential from these that the adsorption is essentially by physisorption [37, 38].

CONCLUSION

The inhibitory effects of calcium sulphate concentrations of 0-35% on the corrosion of cookware aluminium in acidic media of 2.5-3 pH and alkaline media of 10.5-11 pH at temperatures of 30, 50, and 70°C have been systematically investigated experimentally using aluminium 6061-alloy coupons for immersion durations of 0-3 hours. The investigation results indicate that:

- The use of corrosion inhibitors and research progress on the subject have been a major and versatile method of mitigating corrosion of cookware aluminium or supplementing protective coatings for greater protection
- The corrosion inhibition efficiencies of the cookware aluminium by calcium sulphate concentrations increase with the concentrations and immersion time of the aluminium in the media but decrease with an increase in the media acidity or alkalinity levels and temperature of the media, to values up to 74.25% in the acidic media and 72.95% in the alkaline media.
- The corrosion inhibition is by coating formation of calcium sulphate on cookware aluminium and the adsorption of calcium sulphate on the cookware coupons in the media conditions is in accordance with the Langmuir adsorption isotherm with Gibbs free energy, enthalpy, and entropy of adsorption values of -23.345 kJ, -30.13 kJ, and 0.195 kJ/K, respectively
- Only adequate concentrations of calcium sulphate can facilitate the corrosion inhibition of cookware aluminium in the media by forming a coating on its surface, separating it from the media corrodent.
- In comparison with the very low organic corrosion inhibitor concentrations of 0.01%-1% that are usually used in acidic and alkaline environments to inhibit the corrosion of aluminium metal with very high corrosion inhibition efficiencies that can be over 90% in many cases, the corrosion inhibition efficiencies of the cookware aluminium coupons by calcium sulphate concentrations of 20-35% in the acidic and alkaline media used in this study are much lower.

- For the highest comparable corrosion inhibition of cookware aluminium in acidic and alkaline media with highly effective organic corrosion inhibitors, the calcium sulphate concentration in the media needs to be greater than 35%,

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