Wetting behaviour of mixed surfactants on the mild steel surface

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Abstract. Interfacial phenomena such as wetting can be enhanced to a greater extent and mixed surfactant systems are promising candidates for the improvement of wettability of hydrophobic surfaces. In this study, the investigation sought to examine the surface-wetting characteristics of mild steel utilizing a blend of the cationic surfactant cetylpyridinium chloride (CPC) and the anionic surfactant sodium dodecyl sulfate (SDS). The wetting behaviour of mild steel surface is quantified in terms of contact angle corresponding to H_2SO_4, mixed surfactant acid solutions, and methanol for three hours. The contact angles of 0.0008 M, 0.0017 M, and 0.00034 M of mixed surfactant on polished mild steel solution were found to be 38.62°, 39.73°, and 48.50° respectively. The contact angle increases proportionally with the concentration of mixed surfactant in the solution, leading to a visible increase in its hydrophilic nature. On polished mild steel, contact angles of 0%, 10%, 20%, and 30% methanol were measured at 98.21°, 100.39°, 105.29°, and 109.37°, respectively. It shows that the hydrophobic nature increased with an increase in concentration of methanol resulting in less interaction between metal substrate and corrosive species.

1 Introduction

Corrosion can be defined as the ongoing, spontaneous deterioration of metallic structures caused by chemical and electrochemical processes [1, 2]. The cost of corrosion is roughly estimated to be around US $ 2.5 trillion which is roughly equivalent to 3.4% of the world's gross domestic product; GDP) [3]. Mild steel (MS) is widely employed in structural materials due to its superior mechanical properties, easy availability, and low cost. However, as MS is commonly used in corrosive environments, its susceptibility to corrosion has been a longstanding concern [4, 5].

Corrosion inhibitors are classified into three main categories: cathodic, anodic, and barrier based on corrosion inhibitor mechanism. Cathodic inhibitors decrease cathodic reaction while anodic inhibitor inhibits anodic reaction. Barrier inhibitors create a barrier or blocks the reaction sites of the surface. Surfactants are generally considered barrier-type inhibitors with either an N or S atom as a part of a functional group of molecules [6].

Surfactants have an amphiphilic structure consisting of a polar head and a non-polar tail [7]. Surfactants can be classified as gemini-type, cationic, anionic, zwitterionic, nonionic, or bola form. Cationic surfactants possess excellent characteristics such as a high affinity for polar molecules and strong adherence to negatively charged surfaces, while anionic surfactants interact with positively charged surfaces and are extremely soluble in water due to the negative charge of the head group [2, 8, 9]. Surfactants have unique properties due to their hydrophilic and hydrophobic moieties, and they are utilized in a variety of applications, such as corrosion inhibitors. The wettability of metallic surfaces can be modified for various purposes such as corrosion resistance, lubrication, anti-icing, *etc.* [10]. A surface is characterized as hydrophilic if its static water contact angle θ measures less than 90° and hydrophobic if θ exceeds 90° [11]. The hydrophilicity of a surfactant molecule can be increased by diminishing the size or quantity of alkyl chains or by including polar substituents. On the other hand, a surfactant molecule's hydrophobicity can be increased by adding more or larger alkyl chains, or by eliminating the polar substituents. When surfactant interacts with a metal surface, hydrophobic carbon chains point toward the solution side, while hydrophilic heads face the metallic surface [2, 8].

Corrosion researchers focused on discovering, exploring, and synergizing to improve the inhibitive power of inhibitors [12-13]. Synergistic inhibition is a useful strategy for increasing the inhibitive force of an inhibitor with a minimum quantity of usage. Fuchs-Godec et al. [14] reported that the cationic and zwitterionic surfactant mixtures showed significant adsorption properties and good inhibitory properties. Javadian et al. [11] focussed on the synergistic effect of mixed anionic & cationic inhibitors of mild steel in 3.5%NaCl and investigated the inhibition of anodic and cathodic reactions by adsorption on the MS surface. The corrosion inhibition of mixed cationic and anionic inhibitors on mild steel has been studied.

The major goal of our research is to assess the corrosion-inhibiting effect of various concentrations of mixed surfactants (0.0008M, 0.0017M, and 0.0034M) on mild steel in 0.5M H_2SO_4. The test method includes contact angle measurements using the sessile drop method at room temperature. The experiments were performed in polar (water) and less polar (methanol) liquids. Static contact angle was measured with the variation of concentrations of mixed surfactants and methanol by observing the change of contact angle with time.

2 Material and methods

2.1 Materials

AR-grade SDS and CPC, with 98% purity without further purification, were purchased from Hi Media Laboratories and Thermo Fischer Scientific India Pvt Ltd, respectively. Pure methanol (99%) was purchased from Merck's Laboratories, India. AR-grade sulfuric acid from Sigma Aldrich, India, and triply-distilled water from the local market of Nepal was purchased.

2.2 Corrosion study

2.2.1 Specimens Preparation

Mild steel, with chemical composition, as mentioned in[15] was purchased from the local market and was cut into 2 cm by 2 cm rectangles. Each sample specimen was polished to make a mirror image surface with 600, 1000, 1200, and 2000 grades of silicon paper. It was cleaned with hexane, sonicated in ethanol for 25 minutes, and then air-dried by blowing dry air.

2.2.2 Solutions preparation

All the solutions were prepared using triple-distilled water at 298.15 K. The stock solutions of mixed surfactant were prepared at 0.0008 M, 0.0172 M, and 0.0034 M SDS, respectively with 0.0004 M of CPC in the presence and absence of 0.5 M H_2SO_4. Mixed solvent media for 0%, 10%, 20%, and 30% of methanol were performed by using pure methanol.

Thus, the prepared solutions were mixed well and made homogeneous.

2.2.3 Measurement of surface tension

All surfactant solutions were prepared fresh right before measurements, and they were kept in a room with low humidity levels to prevent moisture absorption. The Mansingh Survismeter (Borosilicate) was washed with distilled water to ensure accurate measurements. A contact thermometer, a relay, and a heater were used throughout the experiments to control and maintain the constant temperature. For better accuracy and consistency, experiments were repeated several times. Surface tension data was analyzed and plotted using Easy Plot software.

2.3 Contact angle measurement

The surface wetting behaviour of mild steel surface specimens is quantified in terms of contact angle. A digital microscope camera was used to record the shape of a 5 μL water droplet that was placed on the prepared sample's surface. The drop-analysis plugin was used to analyze the droplet's shape and determine the contact angle in the Image J program. Five repeat readings at different locations on the sample's surface were used to quantify the standard error of the water contact angle (WCA) measurement. [19]. A 0.5 cm diameter opening was drilled in the top to make it easier to enter or remove a syringe. In the CSIR IMMT workshop, mild steel measuring 2 cm by 2 cm was accurately cut. To verify that equilibrium contact angles were being measured, a video recorder was used to take a picture of the drop every 30 seconds while measuring the time dependence of the contact angle. 1 mL of solution was dropped onto the sample surfaces. The mild steel disc was calibrated with the image's center of position to measure the advancing contact angle. Next, a syringe was used to apply the initial drop of solution to the mild steel [15].

Test solutions containing 25 ml of mild steel solution were poured into 50 ml beakers containing mild steel specimens and kept at room temperature for 3 hours. Both the uncontrolled solution and the solution containing mixed surfactant were subjected to the measurements [16]. The contact angle was represented by polished mild steel, corroded mild steel, and inhibitor-adsorbed mild steel. Pure distilled water was used on the surface of polished mild steel at different variation times (0 sec, 30 sec, and 60 sec and it was applied to a variety of mild steel surfaces including polished mild steel, mild steel that had been corroded by 0.5 M H_2SO_4, and mild steel that had been inhibited at varying concentrations of 0.0008 M, 0.0017 M, and 0.0034 M of mixed surfactant. Even very small portions of the mild sheets used in the experiment were found to be non-straight since they were available as rolled sheets. This could have led to differences in the contact angle results. Therefore, a base plate and four screws were used to create an arrangement that would hold the sheet very firmly.

3 Results and discussion

3.1 Nature of pure and mixed solution surfactants

Minimum surface tension values and the critical micelle concentration (CMC) can be obtained by measuring surface tension for cationic and anionic as well as mixed surfactant solutions.

3.2 Surface tension

Surface tension describes a liquid surface's tendency to contract into the smallest possible surface area when it is at rest. Surface tension at liquid-air-contacts arises due to stronger cohesive interactions between liquid molecules compared to adhesive interactions with air molecules. Two forces are seen to be involved. Firstly, the contraction of the liquid due to an inward force acting on the molecules at the surface, and secondly, a tangential force parallel to the liquid's surface, namely surface tension [17]. Water has a surface tension of 72.8 dynes cm^{-1} at 20°C. This higher value of surface tension of water among all liquids is due to the network of hydrogen bonds present between the water molecules. Surface tension has a key role in the capillarity phenomenon. Surface tension is measured in terms of force per unit length or energy per unit area. Although the terms are interchangeable, surface energy is often used to refer to energy per unit of area since it is a more universal term that also applies to solids. Surface tension, surface free energy, and surface stress must all be taken into account when analyzing experimental data on solid surfaces [18].

3.2.1 Cohesive forces

Molecules far from the surface are constantly pulled in all directions by surrounding liquid molecules, resulting in a net zero force. The molecules at the surface are dragged inward on all sides due to the presence of different types of molecules that surround them. As a result, internal pressure is developed which leads to the contraction of liquid molecules to the minimum area [19]. Cohesion is defined as the action or quality of molecules sticking to one another because of mutual attraction. The cohesion force is the force that attracts two similar substances or molecules.

Every molecule of water forms a hydrogen connection with every other molecule. The act of similar molecules sticking together because of their attraction to one another is known as the cohesive force [20].

The equilibrium between the liquid's cohesiveness and adhesion to the material determines a lot of parameters such as the degree of wetting, contact angle, and meniscus morphology. Mercury in a glass container forms a convex meniscus and wetting is non-significant as cohesion energy is larger than adhesion energy. On the contrary, water in a glass container forms a concave meniscus and wetting is significant because adhesion energy is larger than cohesion energy. As mentioned above, surface tension is the force that gives liquid droplets their form. The unequal cohesive forces of the surface layer draw water droplets into a spherical shape, despite their easy deformation. In the absence of outside forces, almost all liquid drops transform into a spherical shape [28]. A molecule's energy level decreases when it interacts with its neighbor. The boundary molecules have more energy than the inner molecules because they lack neighbors, whereas the inner molecules have many neighbors. To decrease the liquid's energy state, it is necessary to reduce the number of molecules at its higher energy boundary. As a result of minimizing the boundary molecules, there is a minimum surface area.

3.3 Calculation of CMC of surfactant by surface tension method

CMC can be determined using the significant breaks in the plot of surface tension or viscosity vs mixed surfactant concentration at a particular temperature. Upon solving the equation, the CMC value can be calculated:

Straight line, $y = -1254x1 + 28.2$

Straight line, $y = -1.95x1 + 23.9$

With distilled water at 298.15 K, Figure 1(a) is the plot of surface tension against concentration of SDS in the presence of CPC whereas Figure 1(b) is the CMC calculation plot of surface tension versus concentration of SDS in the presence of CPC.

The CMC of SDS in the presence of CPC was found to be 0.0034 M. The CMC decreases when two surfactants are mixed. Two oppositely charged surfactants mixed have a larger synergistic effect, resulting in a decrease in CMC [21]. The concentration of SDS that corresponds to the break in the plot of γ vs log [SDS] is considered to be the CMC of SDS, and it is equal to 0.0083 M at 298.15 K [22].

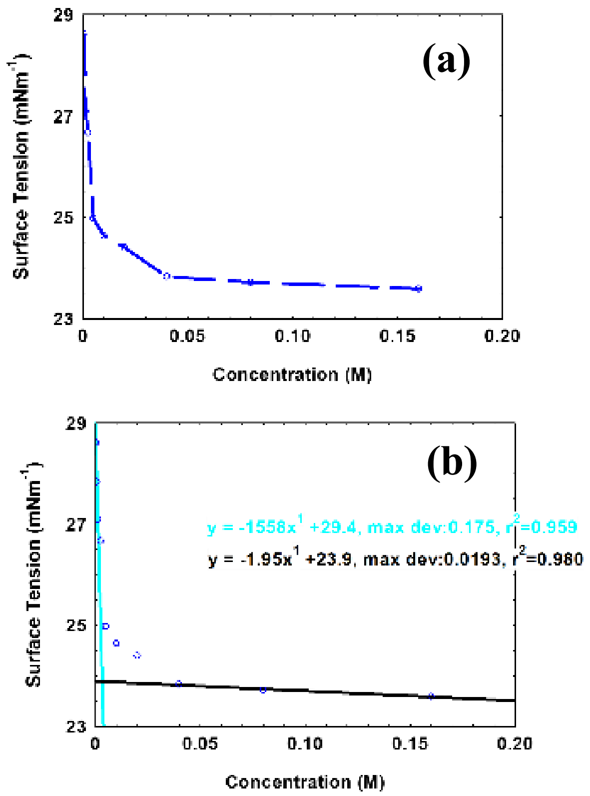

Fig. 1 (a) Surface tension versus concentration (b) CMC calculation by surface tension method

3.4 Contact angle increased with increasing concentration of the surfactant solution

The angle created at the point of contact between a liquid droplet and a solid surface is known as the contact angle. It offers details regarding the liquid's wetting characteristics on the solid surface. When a solid surface is treated with a surfactant solution, the changes in the interfacial tension between the liquid, solid, and gas phases can result in changes to the contact angle. When a surfactant solution is applied to a solid surface, the contact angle may change due to the alteration of the interfacial tension between the liquid, solid, and gas phases. As the surface hydrophobicity increases, the contact angle increases. The contact angle varies with the concentration of the surfactant solution [23].

3.5 Surface wetting

Due to several reasons such as surface roughness or the presence of impurities, water droplets may not completely wet the mild steel surface when they are first applied. The surface becomes more wettable over time when water molecules diffuse into surface imperfections or displace impurities, lowering the contact angle. Figure 2a shows how the contact angle on polished mild steel with pure water was 98.21° at 0 sec. The contact angle with distilled water was measured after 30 sec fall on the polished mild steel surface, and the result is 79.65°, as seen in Figure 2b.
After 60 sec fall on the polished mild steel surface with distilled water, the contact angle was measured and determined to be 76.11°, as shown in Figure 2c. It indicates that on the polished mild steel surface, the contact angle falls as the period increases. Water on mild steel surfaces becomes more hydrophilic over time.

3.6 Contact angle with mild steel, corroded mild steel and inhibited mild steel

Mild steel was used for the measurement of contact angle. However, mild steel was submerged in many kinds of solutions. The contact angle of water on polished mild steel is 98.21° in Figure 3a. Mild steel was immersed in 0.5 m H_2SO_4, 0.0017 M, and 0.0034 M solution for three hours. The contact angle of water was measured on the surface of mild steel, corroded mild steel, and inhibited mild steel. On corroded surface was found to be 36.21° (Figure 3b). 0.0017 M mixed surfactant in solution in an acidic medium was found to be 43.21°(Figure 3c) and 0.0034 M mixed surfactant in solution in an acidic medium was found to be 60.12° (Figure 3d).

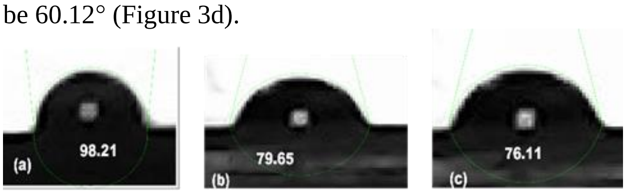

Fig. 2 Contact angle with a variation of time (a) contact angle on mild steel at 0 sec. (b) contact angle on mild steel at 30 sec. (c) contact angle on mild steel at 60 sec.

After three hours of immersion in a 0.5M H_2SO_4 acid solution, polished mild steel exhibits hydrophobic properties of its water molecules. A smaller contact angle is achieved. It displays the hydrophilic character shown in Figure (3 b) when mild steel is immersed in an acidic media containing 0.0017M mixed surfactant solution for 3 hours. About the 0.5M H_2SO_4 solution, the contact angle is enhanced. This shows that the surfactant has been adsorbed into the mild steel. This, in comparison to the acid solution, adsorption of surfactant on mild steel increases the contact angle and exhibits less hydrophilicity shown in Figure (3c). In comparison to a 0.5M H_2SO_4 solution and a 0.0017 M mixed surfactant solution in an acidic media, the contact angle of the mixed surfactant concentration is enhanced. In the same way, the contact angle increases at 0.0034M shown in Figure 3d. It shows that compared to pure mild steel, mixed surfactant has more adsorption on mild steel and a higher contact angle becomes higher than pure mild steel [24]. It demonstrated that mild steel dipped in 0.0017 M mixed surfactant is less hydrophilic than acidic mild steel.

3.7 Effect of concentration of the mixed surfactant solution

The type of solute and solvent involved determines how concentration affects surface tension. The cohesive forces between molecules at a liquid's surface give rise to a phenomenon known as surface tension. When a solute is added to a solvent, the surface tension may be affected in the ways described in the literature [18]. Non-surfactant solutes may also have some effect on surface tension, however surfactants usually lower surface tension by adsorbing at the liquid-air interface. Surface tension is more complicated to understand and is dependent on several variables when it comes to salts and electrolytes.

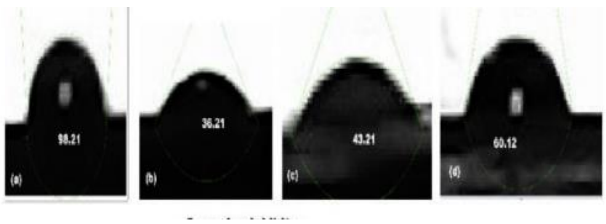

Fig. 3 Contact angle with (a) polished mild steel, (b) corroded mild steel, (c) inhibited mild steel at 0.0017 M mixed surfactant solution, and (d) inhibited mild steel at 0.0034 M mixed surfactant solution.

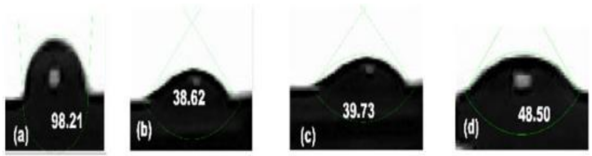

Fig. 4 Contact angle variation of concentration of mixed surfactant solution on polished mild steel plate: (a)water on polished mild steel. (b)0.0008M, (c) 0.0017M, (d) 0.0034M mixed surfactant solutions on polished mild steel.

3.8. Variation contact angle with the concentration of the surfactant solution

The angle created at the point of contact between a liquid droplet and a solid surface is known as the contact angle. It offers details regarding the liquid's wetting characteristics on the solid surface. The contact angle is altered when a surfactant solution is applied to a solid surface because of changes in the interfacial tension between the liquid, solid, and gas phases [32]. As the surfactant solution concentration rises, the contact angle may also increase.

The contact angle of water on polished mild steel is 98.21° in Figure 4a, while the contact angle of 0.0008 M mixed surfactant solution on polished mild steel is 38.62° in Figure 4b. For 0.0017 M mixed surfactant on polished steel, the contact angle is 39.73° in Figure 4c, and for 0.0034 M mixed surfactant solution on polished mild steel, the contact angle is 48.50° in Figure 4d.

In the case of pure water, mild still had the maximum contact angle. As the concentration of the mixed surfactant solution increases, so does the contact angle. It demonstrates that as the amount of surfactant in the solution increases, the hydrophilic nature decreases [27].

The contact angle of a surfactant solution on mild steel increases with the increase in the concentration of the surfactant due to several factors related to the behavior and properties of surfactants at different concentrations. At low concentrations, surfactants adsorb at the solid-liquid interface, reducing the surface tension and leading to a lower contact angle. As the concentration increases, more surfactant molecules adsorb onto the mild steel surface, leading to a more uniform and dense surfactant layer. At higher concentrations, surfactants start forming micelles in the solution. This reduces the number of free surfactant molecules available to adsorb on the surface, leading to an increase in contact angle because the surface becomes less effectively wetted by the solution. With higher surfactant concentrations, the surfactant molecules on the surface may reorient such that their hydrophobic tails are more exposed to the air, increasing the contact angle. This orientation happens because the surfactant molecules pack more densely on the surface and the interactions between the tails dominate.

Beyond the critical micelle concentration, further addition of surfactant does not increase the number of surfactant molecules adsorbing to the surface. Instead, it increases the number of micelles in the bulk solution, which can lead to a stabilization of the contact angle or even an increase due to the reasons mentioned above.

The adsorption of surfactants can modify the surface roughness and chemical heterogeneity of mild steel. An increase in roughness or changes in surface energy can result in an increase in the contact angle [25].

These factors collectively contribute to the increase in the contact angle with the increasing concentration of surfactant on mild steel.

3.9 Effect of the percentage of methanol on contact angle

The effect of the percentage of methanol in a solution on the contact angle can vary depending on the system including the solid surface's nature, the liquid mixture's specific properties, and any additives present. Table 1 shows the contact angle variation of the percentage of méthanol.

Table 1. Contact angle variation of percentage of methanol.

S.N.	Solv. composition	Contact angle
1.	0%	98.21⁰
2.	10%	100.39⁰
3.	20%	105.29⁰
4.	30%	109.37⁰

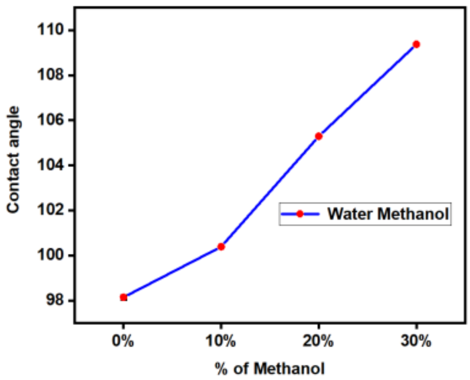

Fig. 5 The contact angle of mixed surfactant at 298.15 K increases with the percentage of methanol.

When the volume fraction of methanol rises, the CMC of mixed surfactant at 298.15 K increases; a similar trend was seen for the contact angle. The surface gets more hydrophobic as the proportion of methanol in the solution rises. The surface's increased hydrophobicity, or decreased wettability, is the cause of the increase in contact angle. After adsorption through tail groups, the surfactant molecules should have comparable orientations on the hydrophilic surface[26].

3.10 Wettability of mixed surfactant system on mild steel

Figure 2 shows variations in the contact angle with time in mild steel and mild steel. Figure 3 shows the variation of contact angle with mild steel, corroded mild steel, and inhibited mild steel respectively. Figures 4 and 5 depict the variation of the contact angle in relation to the concentration of surfactant solution and the percentage of methanol solution on polished mild steel plate. On the polished mild steel surface, the contact angle decreases as the period increases. Water becomes more hydrophilic as the longer it remains on the mild steel surface. Water molecules and mild steel are attracted to one another.

The water contact angle measurement can also be used to assess the corrosion inhibitors' efficacy in an acidic media [27]. Both the chemical composition and the wettability of the mild steel surface are altered when inhibitor molecules are deposited there [28,29]. As a result, both in the presence and absence of inhibitors, the hydrophilic or hydrophobic qualities of the mild steel surface can be directly ascertained by measuring the water contact angle [30,31]. Here, the wetting properties of the mild steel surfaces were examined both in the presence and absence of the maximal inhibitor concentration following a 3-hour immersion in the 0.5M H_2SO_4 solution. With a contact angle of 98.21°, the recently polished mild steel surface demonstrated water-repellent properties. On the other hand, the contact angle dropped to 36.21° after the mild steel specimen was immersed in 0.5M H_2SO_4 medium for three hours (Figure 3). The primary correlation between this hydrophilic attribute and the development of corrosion products on mild steel surfaces is established. The polar nature of the produced corrosion products alters the surface's composition and characteristics [32,33]. This makes water droplets on surfaces readily deformed. However, it is also clear that the addition of inhibitor molecules led to an increase in the aggressive acidic solution's contact angle. In the presence of mixed surfactant inhibitors at concentrations of 0.0017 M and 0.0034 M, respectively, the contact angles shifted to 43.21° and 60.12°. This implies that when inhibitors were present in the aggressive acidic media, a water-repellent coating developed on the mild steel surfaces [34,]. Consequently, the hydrophilic surface was converted to the hydrophobic surface when inhibitor molecules were present in the acidic media [35]. Additionally, it was demonstrated once more that the aromatic moiety has a far greater potential for surface protection than small aliphatic chains [36, 37]

4 Conclusion

The hydrophilic nature of water increases with increased time on the mild steel surface. At concentration is increased it might result in an increase in contact angle. i.e. decrease in wetting, which leads to a higher contact angle. Conversely, at lower concentrations, it might result in good wetting until reaching the CMC concentration of the surfactant. When the concentration of surfactant is higher, at the CMC point surfactant molecules are more spread out, and their effect on reducing surface tension is limited. As a result, less water is in contact with the surface, leading to an increase contact angle. However, at higher concentrations, more surfactant molecules are available for adsorption, altering the surface properties and resulting in a larger contact angle. Similarly, the percentage of methanol affects the surface energy of the mild steel, which in turn influences the contact angle. With an increase in the concentration of methanol, molecules adsorb onto the surface of mild steel, resulting in a decrease in surface energy and, hence, an increase in contact angle.

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