



# A Review of Preparation and Characterization of Sol-Gel coating for Corrosion Mitigation

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## Abstract

Sol-gel technology has emerged as a powerful and versatile tool for synthesizing a wide range of materials such as catalyst, ceramics, coatings, composite, glasses, fibers. Many industries like Automotive, Biomedical, Communications, Construction and Electronics sectors benefit from the use of sol-gel chemistry due to its versatility in fabricating a wide range of materials with different properties. In the sol-gel process, a precursor solution (sol) is used, and the precursor molecules undergo controlled hydrolysis and polycondensation to become a solid substance (gel).

This paper explores the application of the sol-gel synthesis method in coating technology for corrosion prevention. Corrosion presents a notable risk to infrastructure, machinery, and diverse industrial uses, resulting in considerable economic losses and safety hazards. Several methods exist for corrosion prevention using coatings such as chemical vapor deposition and physical vapor deposition but sol-gel stands out due to its low synthesis temperature, cost-effectiveness, simplicity, ability to incorporate and leverage corrosion inhibitors and adaptability to various substrates. Limitations exist in terms of process optimization, and long-term stability, ongoing research efforts are actively addressing these challenges to unlock its full potential. Additionally with our study of related journals we have found that these coatings can be further engineered to exhibit additional functionalities like hydrophobicity, conductivity, or biocompatibility, expanding their applicability across diverse industrial needs.

## 1. Introduction

The sol - gel synthesis process is a flexible method used to produce a vast range of materials from inorganic glasses to complex organic inorganic hybrid materials. It is a wet-chemical process where an inorganic colloidal suspension (sol) is formed and then undergoes gelation in a continuous liquid phase (gel) resulting in the formation of a three-dimensional network structure [1,2]. Sol-gel materials exhibit a wide range of properties which include optical transparency, high surface area, chemical stability, and mechanical strength. Sol-gel materials have applications in various fields like optics [3], electronics [4], construction [5-8], energy storage [9], textile [10], health [11] and coatings [12]. Corrosion is an important issue because it degrades metals mostly in harmful environments like marine etc. The corrosion advancement impacts the metallic structure, potential structural failure, product loss, leaks and environmental harm which results in financial expenses. NACE ( National Association Of Corrosion Engineering ) International released the IMPACT ( International Measures of Prevention, Application and Economics of Corrosion Technology ) study in the year 2016 in which the global cost of corrosion is equal to US 2.5 trillion dollars which is almost equal to 3.4 % of global Gross Domestic Product (GDP). The sol-gel process is one of the best alternatives of toxic pretreatments and coatings which increases corrosion resistance of metals



and is also environmentally friendly [13]. Solgel permits the combination of natural materials and inorganic in a single phase and has the advantage of organic -inorganic (OIH) coatings for a few applications and also for corrosion for a few applications and also for corrosion mitigation. In a report the application of sol-gel methods for coatings for metal corrosion in the year 2001 which concluded that new chromate free materials obtained from sol-gel methods allowed flexible control of morphology coating and oxide materials which led to enhanced properties of the coatings. It showed that Organic-inorganic hybrid (OIH) produced show enhanced properties of adhesion and corrosion protection. The process is easily integrated with some coating like dip coating, spin coating and spray coating [71].

## 2. Sol Gel Process

There are some steps involved in the sol-gel preparation process to achieve the final formula including mixing, gelation, ageing, drying, and sintering [1]. Variations of these processing steps can affect the sol- gel properties by physical and chemical routes.

### 2.1. Mixing

All alkoxide precursors having the general formula  $M-(OR)_n$  (for example,  $Si(OR)_4$ ) when mixed with water, undergo hydrolysis and condensation reactions [14]. Hydrocarbon-based solvents (such as alcohol) are often used to disperse the precursors. By nanoparticles colloidal dispersion in the liquid [15], [1].

### 2.2. Gelation

The colloidal particles in the sol-gel link together via polycondensation to form a network-structured material. The catalyst plays a significant part in this process due to the ionic charge of nanoparticles with a direct effect on the polycondensation rate. For example, at low pH (~1.5), the silica nanoparticles tolerate minimum ionic charge around the silica isoelectric point wherein the silica surface electric charge is zero and thus can collide and connect into chains networks, creating a polymeric gel [14]. On the other hand, at high pH, the dissolution rate is high, and the nanoparticles propagate into the regular size and a decrease in number as the smaller nanoparticles are deposited around, the larger particles [15]. Here colloidal gel is formed. The colloidal gels with the high specific surface area and lower density will be obtained [16].

### 2.3 Drying

The ageing process occurs via three steps, including polymerisation, syneresis, and coarsening. The connectivity and continuity of the gel network increase with polymerisation of unreacted hydroxyl groups. There is some shrinkage during the ageing process. Syneresis is irreversible shrinkage of the gel network caused by the expulsion of the relocating liquid, leading to compressive stresses that draw the solid network to the liquid state. Coarsening is also known as ripening, is considered as a process of reprecipitation and dissolution and is driven by solubility differences between surfaces and other radii of curvature. In the coarsening process, no shrinkage of the network will happen.

### 2.4 Densification

Sintering the dried gel at elevated temperatures will lead to densification of the gel. The reduction in interfacial energy takes place on this process, leading to increased strength and consolidation of the xerogel, thus reducing internal porosity. It is evident that the densification kinetics in gels is considerably complicated as a result of the concurrent reactions of dihydroxylation and structural relaxation [15,1].



### 3. Materials

Sol-gels are mostly manufactured by involving of the organic precursor containing one or more desired organic groups with inorganic precursor like as tetraethyl orthosilicate (TEOS) to achieve the desired properties, such as conductive, hydrophobic, easy to dry, flexibility [17,18]. Synthetic methods for oxide materials often rely on polymerizing metal alkoxides like silicon (Si), such as tetraethoxysilane (TEOS) or tetramethoxysilane (TMOS). Factors like pH, temperature, catalyst, solvent, alkyl group nature, and water to alkoxide ratio influence this process. Alkoxysilanes have slower kinetics compared to titanium, aluminum, or zirconium alkoxides, allowing better chemical control and resulting in materials with diverse properties like pore size, particle dimension, and shape. Silicon's lower reactivity towards chelating groups and redox processes may yield fewer byproducts.

Chemical Name	Abbreviation
Tetraethoxysilane	TEOS
Tetraethyl Orthosilicate	TMOS
Methyltriethoxysilane	MTES
Methyltrimethoxysilane	MTMS
Vinyltrimethoxysilane	VTMS
Phenyltrimethoxysilane	PTMS
3-Aminopropyltrimethoxysilane	APTMS
Aminopropyltriethoxysilane	APTES
N-(2-aminoethyl) 3-aminopropyltrimethoxysilane	AEAPS
3 - Glycidoxypropyltrimethoxysilane	GPTMS
3-Methacryloxypropyltrimethoxysilane	MAPTS
3,3,3-Trifluoro Propyltrimethoxysilane	TFPTMOS
Diethoxymethylsilane	DEDMS
T-Mercaptopropyltrimethoxysilane	MPTMS
Ethyltriacetoxysilane	ETAS
Vinyltriethoxysilane	VTES
Bis(trimethoxysilyl)ethane	BTMSE
2-(3,4-Epoxy cyclohexyl)-ethyltrimethoxysilane	ECHETS
Titanium(IV) tetra 1-propoxide	TIPT
Zirconium(IV) tetra- 1-propoxide	ZrTPO
Diethylphosphonatoethyl-triethoxysilane	PHS



Bis-1,2-[triethoxysilyl]-ethane	BTSE
2,2-Bis-(4-hydroxyphenyl)-propane	BPA
3-Isocyanatopropyltriethoxysilane	ICEPTES
Bis-[3-(triethoxysilyl)-propyl]-tetrasulfide	BTSTS

**Table 1.** Chemical name and Abbreviation of the alkoxy silanes used as precursors for synthesis of OIH materials

#### 4. Influence of the parameters in sol-gel process

Various factors affect how quickly and to what extent certain chemical reactions occur during the synthesis of materials, ultimately influencing their properties. Here are some key factors:

**4.1. Choice of Ingredients :** Different starting materials can lead to different outcomes. The properties of the metal, how easily it can bond with other atoms, the strength of those bonds, and the characteristics of the molecules attached to it all play a role. By selecting specific ingredients, we can control the shape and structure of the final product.

**4.2. pH Level:** The acidity or basicity of the environment affects how fast the reactions happen. For instance, in making silica materials, we often need acidic or basic substances to speed up the reactions because they wouldn't occur quickly enough in a neutral environment. Acids make certain parts of the molecules more reactive, while bases encourage reactions by attacking specific atoms. This difference leads to different final structures: acids produce linear networks with lots of unreacted parts, while bases create more branched, dense structures.

**4.3. Catalysts or Inhibitors:** Adding certain substances can speed up or slow down the reactions. In some cases, we use chelating agents to slow things down, which makes the material less tightly connected.

**4.4. Ratio of Ingredients:** The amount of water we use affects how quickly certain reactions happen and how the material forms.

**4.5. Solvents:** The type of liquid we use can interact with the ingredients in different ways, affecting how they react.

**4.6. Temperature:** Not only does temperature affect how fast reactions occur, but it also impacts the evaporation of liquids during the process, which can influence the formation of cracks in the final product.

#### 5. Corrosion Inhibitor

The term “ corrosion inhibitor “ covers chemical elements and compounds that reduce the rate of corrosion on the material [19]. Incorporating corrosion inhibitors, nanoparticles, or reinforcing agents into the sol allows tailoring properties for specific environments and functionalities. Nanoparticles can enhance barrier properties, while inhibitors impede corrosion reactions. In recent years, significant endeavors have focused on discovering effective corrosion inhibitors. Among



these, inorganic inhibitors, such as those utilizing rare earth metals like cerium and samarium, have garnered attention. These inhibitors are applied to light alloys such as AA2024-T3 to mitigate pitting corrosion. They function by acting as passivation corrosion inhibitors, counterbalancing the release of electrons that could otherwise accelerate corrosion rates and compensating for the loss of ionic charge [20,21]. The other form of inhibitor is the organic inhibitor, which can be used as a source in various corrosive environments. In acid media, nitrogen-based materials and their derivatives, sulfur-containing compounds, aldehydes, thioaldehydes, acetylenic compounds, and various alkaloids, for instance, papaverine, strychnine, quinine, and nicotine are used as inhibitors. Volatile corrosion inhibitors, a subset of organic inhibitors, are employed to safeguard metal alloys, whether ferrous or non-ferrous, from oxidation, often with a low pH as film-forming corrosion inhibitors. When possible, these inhibitors can be administered to metal surfaces via methods like evaporation or diverse deposition techniques, encompassing the use of films, papers, desiccants, coatings, and evaporation chambers [22,23]

. Various types of inorganic inhibitors with minimal toxicity have been employed to enhance the corrosion resistance of hybrid coatings. Inorganic inhibitors, such as phosphates [24], vanadates [25], rare earth elements [26,27], have shown positive influence on the corrosion protection of aluminum alloys. Corrosion inhibitors can be added to the sol-gel system during a synthesis procedure for pretreatments or at the stage of film formation and cross linking. The most straightforward approach to incorporating inhibitors is by directly adding them into hybrid coatings. While inhibitors demonstrate effectiveness in preventing corrosion of bare metal, the impact of directly incorporating inhibitors into hybrid coatings remains uncertain and requires further investigation. Addition of corrosion inhibitors into sol-gel coatings leads to a positive effect in some cases, even increasing the barrier properties of the film [31]. It may seriously deteriorate the stability of the sol-gel matrix [28, 29, 30, 32]. The direct introduction of inhibitors into the protective coatings often raises a lot of adjacent problems such as decrease of barrier properties, osmotic blistering and deadhesion of the coating.

## 6. Studies on Hybrid sol-gel process

Year	Author & Reference	Discussed subject matter
2016	Wazarkar.K ET al. [33]	Principles and purpose of microencapsulation, morphologies of microcapsules ,chemical and physical techniques and healing mechanisms for the coating were spoken.
	Guo.X ET al.[34]	The main goal is to achieve anticorrosion coatings.The progress made in the synthesis of many sol-gel-derived materials was also spoken.
	Figueira.R.B ET al.[35]	Advancements have been made in the development of sol-gel coatings, specifically those containing OIH (organic-inorganic hybrid) components, to mitigate corrosion on steel and aluminum substrates.
	Zvonkina.I Et al. [36]	The overarching characteristics of OIH coatings, along with recent progressions, were outlined.



	Zhang.L Et al.[37]	The most recent accomplishments and tactics concerning the sol-gel process parameters and various factors impacting the corrosion resistance of OIH coatings for safeguarding aluminum-based alloys against corrosion were summarized.
	Ismail[10]	A discussion is provided on the capabilities of sol-gel technology, examining the diverse functions it can achieve in fabric applications, with a focus on its role in producing anticorrosion coatings.
2017	Ulaeto.S.B ET al. [38]	Recent progress in intelligent coatings, such as OIH coatings, was examined in terms of their responsiveness to various stimuli and types of damage. The review highlighted advancements in corrosion detection, self-cleaning, anti-fouling, and self-healing properties within polymeric coating systems.
	Eduok.U ET al. [39]	Recent uses of PDMS polymers for functions such as corrosion prevention, biofouling resistance, ice prevention, flame retardancy, self-cleaning, and reduced reflection were examined in a review.
	Fihri.A ET al.[40]	A summary was provided of superhydrophobic coatings, which encompass OIH sol-gel coatings, documented in literature for safeguarding steel, along with their effectiveness.
2018	Ga siorek. J ET al. [41]	Various approaches to decelerate the corrosion of metal substrates were explored through the utilization of oxides and doped oxides produced via the sol-gel technique.
	Aparicio.M ET al. [42]	Examination was conducted on notable instances where electrochemical methods like EIS, PDP, SVET, SIET, SKP, and LEIS were applied to elucidate the precise mechanism of protection provided by sol-gel coatings on metal surfaces.
	Faustini, M ET al. [43]	An overview was given of the historical understanding of OIH material science, highlighting key periods associated with the origin and development of OIH materials.
2019	Barroso,G ET al. [44]	The primary considerations regarding the utilization of silicon polymers in coatings were examined, covering their pros and cons as well as the evolution of processing techniques devised for these materials.





	Jose Carlos Bernedo Alcazar ET al. [45]	The study successfully synthesized organic-inorganic nanocomposite materials using sol-gel dip coating, characterized them using AFM, SEM, and EDX, and found that the hybrid-coated titanium samples had rough nanostructured surfaces, biocompatible elements, and supported high cellular growth, making the sol-gel dip coating method a viable option for creating stable hybrid layers on titanium surfaces.
	A Bouibed ET al. [46]	The study describes the successful synthesis of SiO <sub>2</sub> nanoparticles on graphene oxide (GO) nanosheets using a one-step in-situ sol-gel process, their incorporation into an epoxy matrix, and demonstrates that the resulting GO-SiO <sub>2</sub> nanohybrids significantly improve the thermal stability and corrosion protection performance of the epoxy coating compared to epoxy/GO and neat epoxy resin.
2020	M Catauro ET al. [47]	The study successfully synthesized and characterized hybrid materials with caffeic acid entrapped in a silica matrix, demonstrating their potential as antioxidant scavengers and antibacterial agents against Escherichia coli and Enterococcus faecalis, with scavenging properties dependent on the caffeic acid content.
	K Ebisike ET al. [48]	Chitosan-silica hybrid aerogel, synthesized from crab shell and bamboo leaf waste, demonstrated a combination of organic and inorganic characteristics, as confirmed by FTIR, SEM, EDX, and TGA analyses, with notable surface area and porosity properties.
	A Salama ET al. [49]	The study presents a three-step synthesis process to create N-guanidinium-chitosan acetate/silica hybrid materials with sulfonate or carboxylate groups, demonstrating their potential as templates for biomimetic calcium phosphate mineralization, which could be valuable for applications in bone tissue engineering.
2021	Michelina Catauro ET al. [50]	This review highlights the potential of bioactive glasses and materials synthesized through the sol-gel technique, including nanoparticles, for various biomedical applications, emphasizing their role in improving the interaction between biomaterials and living tissues while discussing their synthesis and properties.
	Boris Mahtig ET al. [51]	This review discusses the use of hybrid sol-gel coatings for radiation protection, with a focus on materials designed to offer effective protection against UV light while considering various types of electromagnetic radiation.



2022	Mario Basso ET al. [52]	Patterned hybrid organic-inorganic coatings, inspired by the desert beetle's exoskeleton, were successfully deposited on metallic substrates through a sol-gel dewetting process, exhibiting the ability to enhance atmospheric water harvesting and maintaining structural robustness after exposure to humid air.
	Zanurin A ET al. [53]	This paper discusses the versatile and cost-effective sol-gel method for creating ceramic coatings, showcasing its applications in enhancing corrosion resistance, thermal properties, and performance in various fields.
	Tuba Yetim ET al. [54]	This study focuses on enhancing the wear and corrosion resistance of Cp-Ti for biomedical applications by producing Ni-doped Al <sub>2</sub> O <sub>3</sub> nanocomposite coatings using the sol-gel spin coating process and characterizing their structural and performance properties.
2023	Rami K. Suleiman ET al. [55]	They tested how well it protected steel from rust in salty water. Limestone worked best, boosting protection and adhesion while keeping good heat and scratch resistance. Rubber and charcoal hurt the coating, making it porous and weak. Using recycled materials can be cheap, but some work better than others for this coating.
	Srinivasa N.V. ET al. [56]	Tin-doped nickel oxide films were successfully fabricated using sol-gel spin coating, exhibiting tunable structural and optical properties with varying dopant concentration. Optimal doping at 2-4% resulted in high visible light transmittance and slightly increased band gap, suggesting potential applications in gas sensing and optoelectronic devices.
2024	Danqian Wanga ET al. [57]	One-step Portland cement coating with an auto generated oxide film effectively shields magnesium alloys from corrosion. Electrochemical tests confirm significant improvement, highlighting the synergistic effect of the coating and film. Notably, film quality plays a key role, and adding metakaolin and dolomite strengthens the coating while molybdate ions enhance the film itself. This simple method offers promising protection for magnesium alloys.

## 7. Surface and morphology Characterization Techniques and Instrumentation

### 7.1. Electrochemical Impedance Spectroscopy

The study evaluated the corrosion protection performance of parent and waste-modified coating matrices on steel panels exposed to a 3.5 wt.% NaCl corrosive medium. Electrochemical impedance spectroscopy (EIS) was employed to assess the electrical properties of the coatings. Initially, samples modified with limestone and eggshell additives showed higher impedance values and wider semicircles in Nyquist plots compared to the unmodified coating, indicating enhanced barrier properties. However, after 4 weeks of immersion, only the limestone-modified sample maintained improved barrier properties, while others showed deterioration. The experimental data were





modeled using equivalent circuit models, with the limestone-modified sample exhibiting the strongest corrosion protection performance. Potentiodynamic polarization analysis confirmed the EIS results, with the limestone-modified coating showing the lowest corrosion current density. Visual observation also supported these findings, with intact surfaces observed for the limestone-modified and unmodified coatings, while coatings modified with other waste additives showed cracks and defects. Overall, the study demonstrated the efficacy of limestone waste modification in enhancing the corrosion resistance of hybrid sol-gel coatings on steel panels [55].

## 7.2. Scanning electron microscopy (SEM)

The use of electron beams in microscopy dates back to 1932 when Borries and Ruska first reported its application in the Symposium on New Methods for Particle Size Determination [58]. Although the concept of a scanning electron microscope (SEM) was introduced by Zworykin in 1942, it faced resolution limitations [59]. The development of SEM continued, with significant progress achieved in the 1950s at the University of Cambridge, leading to the production of the first commercial scanning microscope by Cambridge Instruments Ltd in 1965. SEM has since become a widely used method for rapid, non-destructive, and cost-effective surface analysis, offering high-resolution topographical images by focusing an electron beam onto the sample surface and detecting resulting secondary or backscattered electrons [60]. SEM utilizes a high-energy electron beam for illumination, providing greater resolution compared to optical microscopes due to the shorter wavelength of electrons. The electron beam is generated at the top of the microscope by an electron gun and then directed through electromagnetic lenses, which focus and concentrate the beam onto the sample surface. Interaction between the electron beam and the sample generates secondary electrons on the surface and backscattered electrons deeper within the material, along with X-rays and heat. Detectors collect these emitted particles and convert them into signals displayed on a computer screen. Additionally, SEMs can be equipped with Energy Dispersive X-Ray (EDX) analyzers for elemental identification and compositional analysis [60,61,62].

## 7.3. Atomic Force Microscopy (AFM)

Atomic force microscopy (AFM) is commonly utilized to assess surface morphology and topography, typically in high-resolution tapping mode. This mode allows for nano-indentation to measure the mechanical properties of the samples being tested [63]. The study investigated the surface morphology of films S1-S3 using AFM analysis and estimated the film thickness using spectroscopic ellipsometry (SE) on PVC substrates. AFM images revealed that the S1 coating (TEOS/MTES/OTES) smoothed substrate irregularities, with a decrease in RMS roughness from 1.67 nm (bare PVC) to 0.41 nm (S1 coating). Introducing VTMS in S2 increased film uniformity, while HDTMES in S3 led to bush-like surface parcels, attributed to surface segregation, with an RMS roughness of 2.51 nm and an Rpv parameter of 12.4 nm. SE showed film thickness ranging from 1288 to 3500 nm, with slight thickness non-uniformity in S2. Increasing silica coating thickness correlated with increased roughness and decreased material hardness. Overall, smooth and continuous silica-based films were obtained by sol-gel on PVC substrates, with variations in morphology and thickness depending on the composition of the coating solution [64].

## 7.4. Contact Angle Goniometer (CA)

The contact angle goniometer was used to measure the angle at the edge of liquid drops on solid surfaces, indicating surface wettability. Young's equation ( $\sigma_{sv} - \sigma_{sl} - \sigma_{lv} \cos \theta_c = 0$ ) was employed to determine the equilibrium contact angle ( $\theta_c$ ) from the interfacial energies of the



solid–vapor (□□□), solid– liquid (□□□), and liquid–vapor (□□□) phases. Hydrophilic surfaces have a contact angle less than 90°, while hydrophobic surfaces have an angle greater than 90° [65]. Super-hydrophobic surfaces can achieve contact angles exceeding 150°. The hydrophobicity of both coated and non-coated samples was assessed by measuring water contact angles using a Dataphysics OCA 15EC Goniometer with deionized water as the solvent. The experiments were conducted at Sheffield Hallam University in MERI labs. Contact angle measurements were performed on both bare and coated samples, with a drop volume size of 1.5 μL and a rate of 0.5 μL/sec. The contact angle was calculated using software provided by the goniometer [66,67,68,69].

## 7.5. Hardness measuring

Hardness, defined as the resistance to plastic deformation, is assessed through indenting a material with a known force for a set time. There are two main classifications: macro hardness (with loads higher than 9.807 N) and microhardness (with loads ranging from 9.807×10<sup>-3</sup> to 9.807 N). Various standard tests, such as Vickers, Knoop, Brinell, and Rockwell, are used, each distributed across different scales. In this project, the DURAMIN-40 AC3 automatic micro/macro universal hardness tester, capable of measuring Vickers, Knoop, and Brinell hardness, was utilized. The Vickers hardness test, employing a diamond pyramid indenter with a penetration angle of 136 degrees, was used to evaluate coating hardness. The Vickers hardness values were determined based on the area of the indentation, calculated using the applied force and the square of the diagonals of the indentation [70].

## 7.6. Thickness measuring

Eddy current testing is a non-destructive technique (NDT) widely used in industries like aerospace and automotive to measure coating and paint thickness on metallic surfaces. It operates on the principle of electromagnetic induction, where an AC current flowing through a coil generates a magnetic field. When brought near a metallic surface, this magnetic field induces eddy currents, which generate their own magnetic fields. Changes in coating thickness or surface cracks disrupt the eddy current pattern, altering the impedance and phase angle in the probe coil. An Elcometer 456 B Coating Thickness Gauge was utilized to measure the thickness of coatings, and SEM cross-sections were utilized to validate the findings [67].

## 8. Conclusion

### 8.1. Corrosion Performance

Bare aluminum alloy 2024-T3 is highly prone to corrosion when immersed in 3.5% NaCl solution. Applying oleic oil solely on the surface provides protection against both atmospheric and saline corrosion, but the resulting film is fragile and susceptible to removal, leading to pitting after three days of immersion. The adsorption of oleic oil on AA2024-T3 is observable due to the change in the carboxylic group's connection to the metal surface. Benzimidazole offers effective protection against corrosion in direct immersion for over five days without pitting, with film formation confirmed visually, as well as through SEM and EDX analysis. The base SBX Sol-gel formula provides good barrier protection without any inhibitor, lasting for at least ten days in 3.5% NaCl solution before cracks and pitting become visually apparent on the coating surface. Fluorinated F-SBX sol-gel coatings offer corrosion protection by enhancing barrier properties through the presence of fluorinated functional groups in the PFOTS precursor, which exhibit hydrophobic behavior. The combination of oleic acid/base SBX sol-gel formula demonstrates corrosion protection lasting up to one month without cracks or pitting. Benzimidazole SBX sol-gel formula is effective for



corrosion protection and can last for one month, with the addition of benzimidazole inhibitor enhancing active protection due to its high electronegativity, resulting in increased impedance. The mixed system of BZI/OA sol-gel provides high impedance protection lasting over three months without exhibiting any signs of corrosion on the coating or metal surface [67].

## 8.2. Formulation and restrictions

Addition of fluorinated precursor PFOTS (1.5% v/v) to the base SBX formula significantly increases water contact angle, attributed to the hydrophobic effect. Adding PFOTS limits the incorporation of other inhibitors due to its hydrophobic nature. The best combination of oleic acid to SBX sol-gel is achieved within the ratio of 0.1 to 0.5% v/v, forming oleic acid enrichment capsules that act as film-forming inhibitors without compromising the coating's strength. The optimal ratio of benzimidazole to SBX sol-gel is 3.5% v/v, enhancing protection without increasing brittleness, which can lead to cracks and delamination on the substrate. Curing for 4 hours at 80°C provides appropriate stability for all sol-gel coating systems [67].

## 8.3. Mechanical Testing

Adding PFOTS to the sol-gel formula results in a reduction in Young's Modulus and hardness levels compared to the base SBX sol-gel, while also reducing post-immersion cracking. The hydrophobic properties of PFOTS enhance the sol-gel coating, making it suitable for use as a monolayer or top coating in a complete system. Sol-gel coatings demonstrate excellent adhesion to AA2024-T3 substrates, as confirmed by cross-cut and pull-off testing techniques. However, the combination of oleic acid/sol-gel coating shows reduced adhesion to the substrate, possibly due to the surface wettability of active molecules. On the other hand, benzimidazole/sol-gel coatings exhibit an increase in hardness and brittleness [67].

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