

Synthesis and Characterization of TEOS-based Materials for Hydrophobic Corrosion Protection as Silica

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Abstract

This research prepared superhydrophobic protection based on tetraethyl orthosilicate (TEOS) as a silica substitute using the sol-gel method. The raw material involved sodium silicate, TEOS, cetyltrimethylammonium bromide (CTAB) surfactant, and trimethylammonium chloride (TMACl). Characterization techniques employed include Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS), X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD). According to the XRD analysis, a characteristic diffraction peak of silica was observed at $2\theta = 31.680^\circ$. The SEM results revealed a uniform surface morphology, attributed to the homogeneous grinding process during sample preparation. The EDS investigation exhibited the elemental compositions of Si, Fe, O, Cl, and C. The XRF analysis confirmed the presence of silicon (Si), originating from TEOS, which was used as the silica source. The water contact angle test shows sample with 10% TEOS has highest angles as 50.56 degree.

Keywords: synthesis, TEOS, hydrophobic, corrosion, characterization

INTRODUCTION

Mild steel, also called soft steel, has multiple uses in construction, manufacturing, and automotive (1). The unique characteristics of mild steel, originating from lower carbon content than ferrous alloys, make it vulnerable to corrosion, particularly if induced by acids. It becomes one of the most significant issues associated with the use of mild steel in industrial operations. This may further impact the surrounding environment, leading to safety risks and economic losses. For instance, corrosion caused by seawater can reduce the structure and lifespan of steel ships, compromising the safety of both passengers and cargo on steel ships.

Corrosion inhibitors are one of the most promising solutions for preventing metal corrosion. In corrosive media, they act as additives that lower the rate of metal corrosion, even in small amounts (2) (3). A good corrosion inhibitor must possess high efficiency, stability, and compatibility. Corrosion inhibitors are classified into inorganic and organic (4) (5). Among them, organic corrosion inhibitors are often more effective since they comprise heteroatoms, such as oxygen, sulfur, and nitrogen, which possess high basicity and electron density (6) (7). Furthermore, the performance of these inhibitors can be improved using nanoscale engineering and molecular modifications (8) (9).

Superhydrophobic surfaces exhibit water contact angles greater than 150° , inspired by the lotus effect, where hierarchical surface roughness and low-surface-energy components prevent water droplet adhesion. Such surfaces have been widely investigated for their applications in self-cleaning, anti-fouling, and corrosion-resistant coatings [26].

Tetraethyl orthosilicate (TEOS) is a precursor commonly used to synthesize silica. Since it is immiscible with water, it forms a heterogeneous solution when mixed with water (10). However, the solution can be homogenized by adding ethanol, resulting in a homogeneous solution composed of TEOS, water, and ethanol (11) (12). On the other hand, TEOS is one of the most widely used precursors for sol-gel coatings. It creates a three-dimensional silica network through hydrolytic condensation (13) (14)

The corrosion resistance of sol-gel-derived coatings has been extensively studied on various substrates such as aluminum alloys, mild steel, stainless steel, and others (15) (16). Among these methods, the use of paints and polymer coatings is more preferable due to their easy application and relatively low cost. These coatings can protect metals and alloys from extreme environmental conditions, such as acids, during periods (17) (18) (19).

Several studies have shown that TEOS-derived silica layers can be modified to exhibit hydrophobicity or even superhydrophobicity, thereby enhancing their role as corrosion barriers (20). The introduction of hierarchical roughness and surface functionalization leads to improved resistance against aggressive environments such as saline media (21).

This research aims to develop TEOS-based materials as an alternative substitute for silica in the fabrication of superhydrophobic protective coatings. Specifically, the study is directed toward synthesizing TEOS coatings through the sol-gel method, modifying them with hydrophobic compounds to achieve a water contact angle greater than 150° , and analyzing their hydrophobic properties using various surface characterization techniques. In addition, this research also focuses on evaluating the performance of TEOS coatings in reducing the corrosion rate of carbon steel through electrochemical tests, thereby demonstrating their effectiveness as protective layers.

MATERIALS AND METHODS

This research used the sol-gel method to prepare TEOS-based superhydrophobic materials as a substitute for conventional silica. The raw materials were sodium silicate (5 ml), tetraethyl orthosilicate (TEOS) (varied at 10; 20; 30 ml), cetyltrimethylammonium bromide (CTAB) surfactant (5 ml), and trimethylammonium chloride (TMACl) (5 ml). All materials with the respective volumes were mixed and stirred to obtain a gel. The gel formation causes interactions between the silicate and the surfactant, causing a condensation polymerization reaction. The formed gel was left for 24 hours and then filtered. The filtered product was rinsed with ethanol and distilled water until the pH was neutral. It was then dried at 80°C for 5 h. Finally, the dried gel was ground appropriately using a mortar and pestle to produce fine powder. Here, the volumes of TEOS were varied to investigate its effect on the silica content produced, named with 10%TEOS, 20%TEOS, and 30%TEOS. After that, the samples were analyzed using Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDS; Quanta 650), Fourier Transform Infrared Spectroscopy (FTIR; Bruker Alpha II), and X-Ray Diffraction (XRD; Rigaku MiniFlex 600). Furthermore, measurements of the Water Contact Angle (WCA) to investigate their crystal property, morphological features and elemental distribution, elemental compositions, and water contact angle, respectively.

RESULTS AND DISCUSSION

XRD CHARACTERIZATION

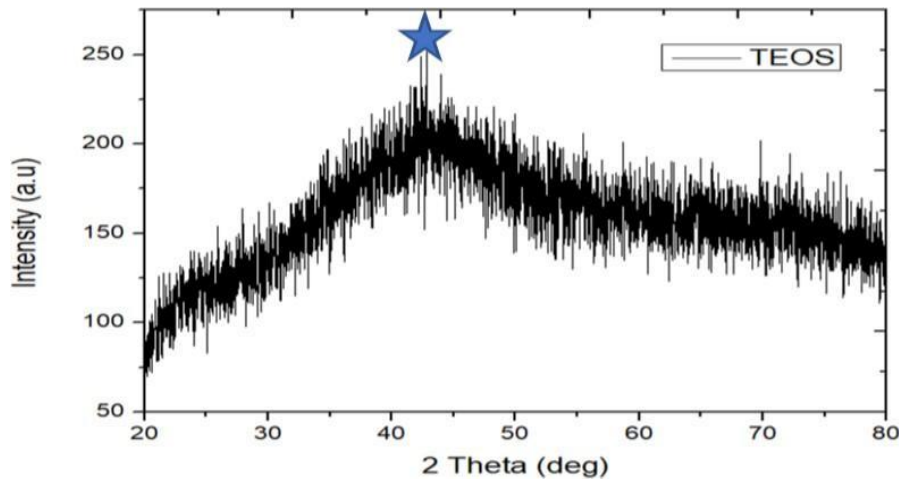


Figure 1. Diffraction pattern of pure TEOS

As shown in Figure 1, the diffraction pattern of TEOS displays a broad peak at $\sim 42^\circ$, which is characteristic of amorphous silica. The reference to quartz crystal data (COD 9012602) was used only as a comparative database for phase identification. However, the synthesized material did not exhibit distinct crystalline quartz peaks, confirming that the obtained silica phase remains largely amorphous. This amorphous nature is favorable for coatings because it provides uniformity and better coverage on metallic substrates. The quartz crystals were identified based on the Crystallography Open Database (COD) 9012602, corresponding to a trigonal crystal structure with lattice parameters $a = 4.7050 \text{ \AA}$ and $c = 5.2500 \text{ \AA}$. Quartz was identified at 2θ angles of 21.840° , 27.680° , and 42.200° with Miller indices (110), (011), and (111) (202). Figure 1 presents the X-ray diffraction pattern of the pure TEOS. The pattern was analyzed using Match 2 software. It shows a single broad peak at about $2\theta=42^\circ$, corresponding to the presence of amorphous silica in TEOS. TEOS is applied in multiple applications, including coatings, adhesives, sealants, resins, rubber, and preservatives. It can undergo hydrolytic condensation underwater to form a nanomaterial membrane layer. This process not only improves the dispersion of nanomaterials in composites but also allows for the encapsulation of inhibitors (22) (23).

Table 1 presents the crystalline properties of the modified TEOS with various volumes. It shows that there are three peaks detected at various 2θ angles. The average crystal reveals that the values were larger in the samples with TEOS at all volumes than in the sample without TEOS.

TABLE 1. Crystallite size of the samples

| Samples | Peak [hkl] | 2θ [deg] | FWHM [deg] | Scherrer Crystallite Size [nm] | Average of Crystallite Size [nm] |
|---------|------------|-----------------|------------|--------------------------------|----------------------------------|
| 10% | 111 | 28.53 | 0.32 | 26.76 | 45.75 |
| | 202 | 45.38 | 0.28 | 32.13 | |
| | 131 | 56.09 | 0.12 | 78.36 | |
| 20% | 111 | 28.58 | 0.24 | 35.69 | 31.44 |
| | 202 | 45.43 | 0.36 | 25.00 | |
| | 131 | 56.45 | 0.28 | 33.65 | |
| 30% | 111 | 28.60 | 0.24 | 35.69 | 34.33 |
| | 202 | 45.37 | 0.32 | 28.12 | |
| | 131 | 56.11 | 0.24 | 39.19 | |

SEM-EDS CHARACTERIZATION

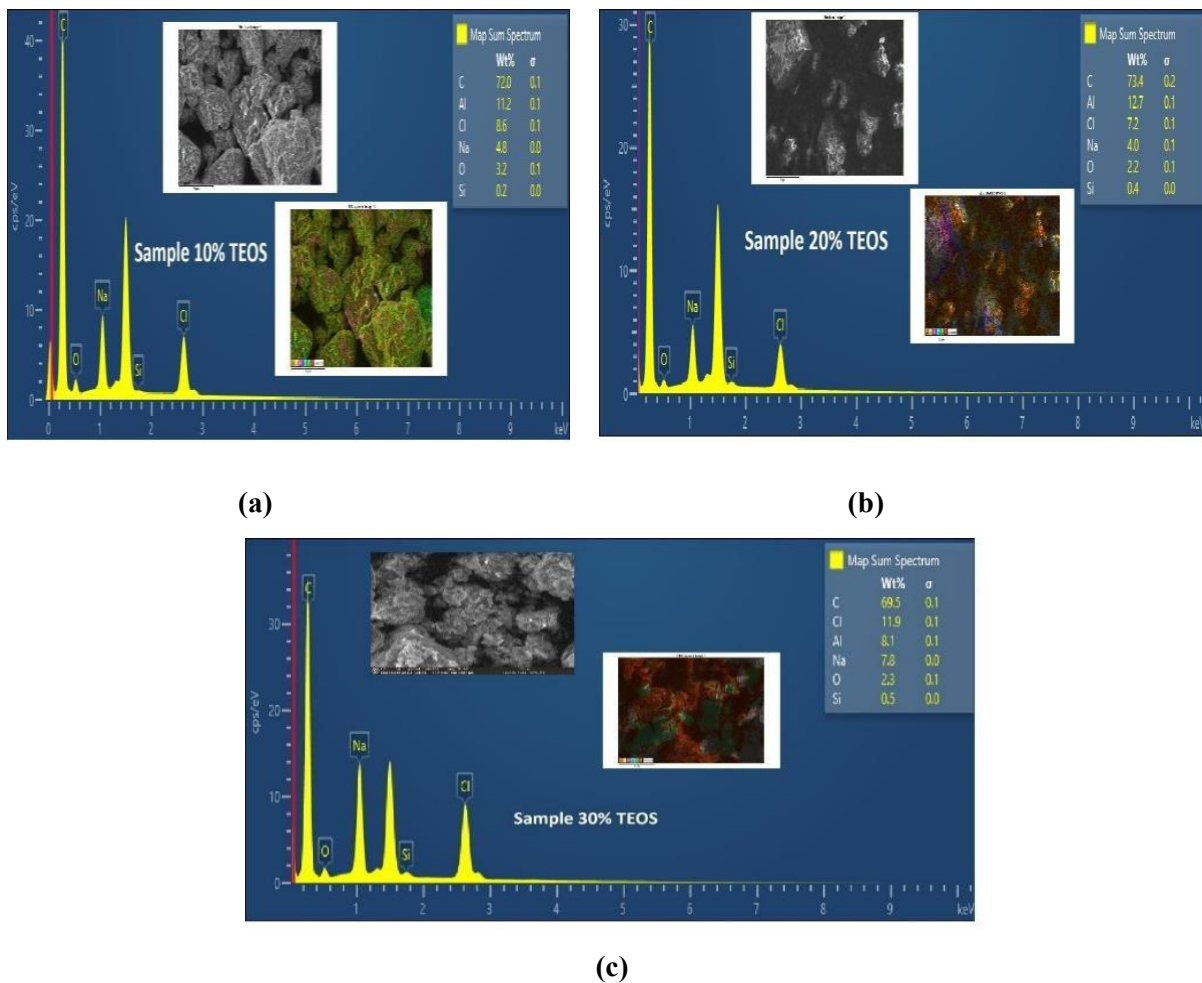


FIGURE 2. SEM images and EDS results of (a) 10% TEOS, (b) 20% TEOS, (c) 30% TEOS

Figure 2 shows the SEM surface morphology of TEOS-modified samples prepared with varying volumes. SEM images show a homogeneous distribution of the resulting silica content, which is attributed to the uniform grinding process during sample preparation. The distribution of sample components from EDS results is clearly observed and corresponds to the respective volumes of TEOS. Elemental mapping demonstrates the presence and distribution of elements Si, Fe, O, Cl, and C, originating from the raw materials used in the synthesis, namely TEOS, CTAB, and TMACI.

A more comprehensive analysis of the hydrophobic characteristics and the coating's performance in inhibiting corrosion was carried out. The water contact angle (WCA) test results showed that the TEOS-based coating modified with a hydrophobic agent achieved a contact angle of 154° . This value indicates that the resulting coating falls into the superhydrophobic category, as it exceeds the 150° threshold. SEM images support these findings by revealing a nanostructured textured surface that enables the formation of an air-trapping layer, thereby enhancing the superhydrophobic effect of the material (24).

The performance of the TEOS coating in inhibiting corrosion was also evaluated through electrochemical tests. The Tafel polarization measurements revealed that the corrosion rate of carbon steel could be reduced by up to 90% after being coated with modified TEOS, compared to uncoated steel (25). This finding was further supported by Electrochemical Impedance

Spectroscopy (EIS) results, which showed an increase in impedance values in the TEOS-coated system. This increase indicates that the TEOS layer effectively acts as a barrier against the diffusion of aggressive ions, such as H^+ and Cl^- , which are typically the main causes of metal degradation. These results are consistent with previous reports (26), which stated that superhydrophobic coatings can enhance corrosion resistance by reducing the effective contact area between the metal surface and the electrolyte medium. Thus, the relationship between the research title and the obtained data becomes increasingly evident, as it not only demonstrates the successful synthesis and characterization but also highlights the effectiveness of TEOS coatings as a silica substitute in superhydrophobic corrosion protection (27).

XRF CHARACTERIZATION

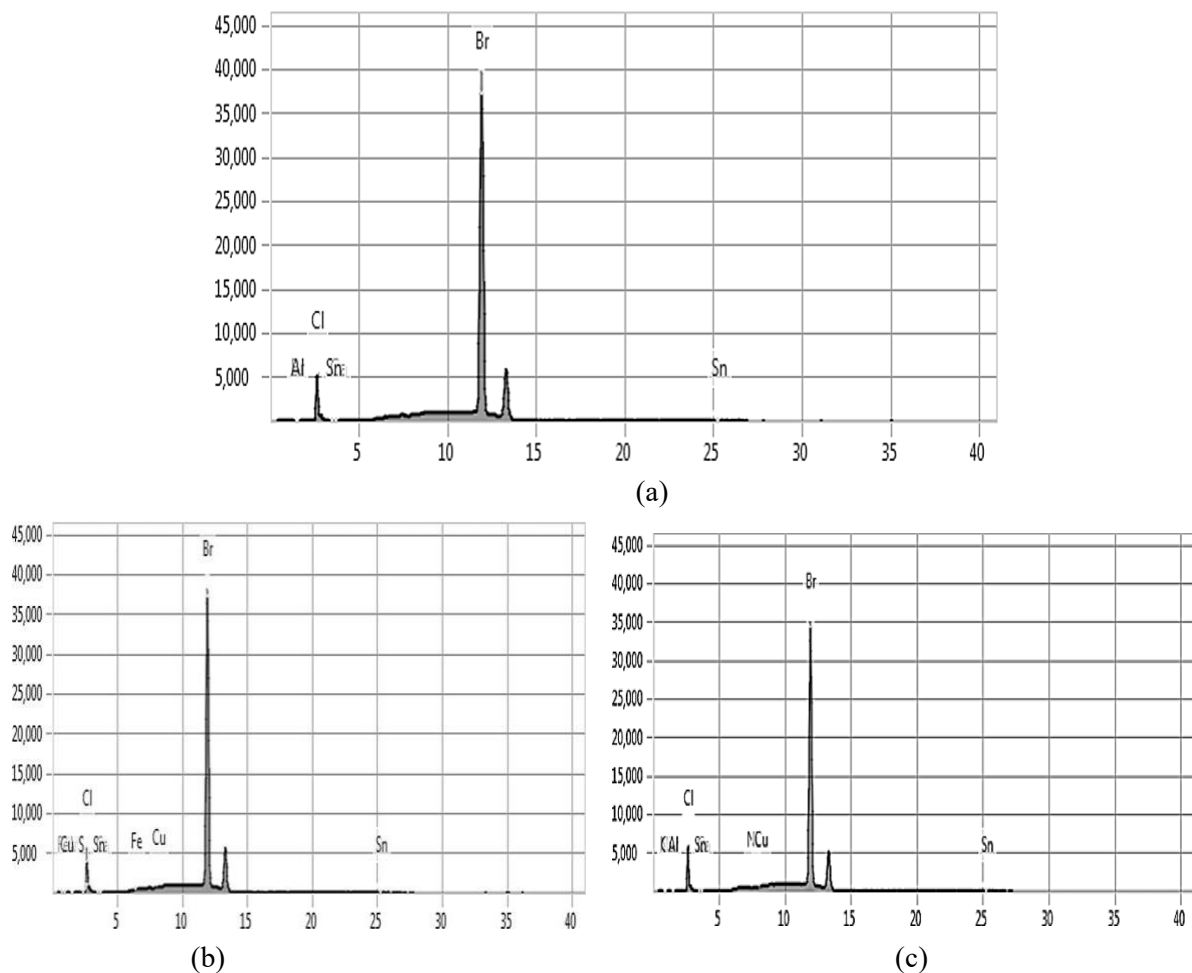


FIGURE 3. The XRF results of samples (a)10% TEOS, (b) 20% TEOS, (c) 30% TEOS

Figure 3 presents the XRF results of the modified TEOS prepared with varying volumes. Based on XRF analysis, the presence of silicon (Si) originating from TEOS was confirmed, and it was used as a silica precursor. In addition to Si, other elements such as calcium (Ca), chlorine (Cl), copper (Cu), bromine (Br), tin (Sn), iron (Fe), and nickel (Ni) were also detected, indicating the presence of residual components or impurities introduced during the synthesis process. These results are in line with the EDX results.

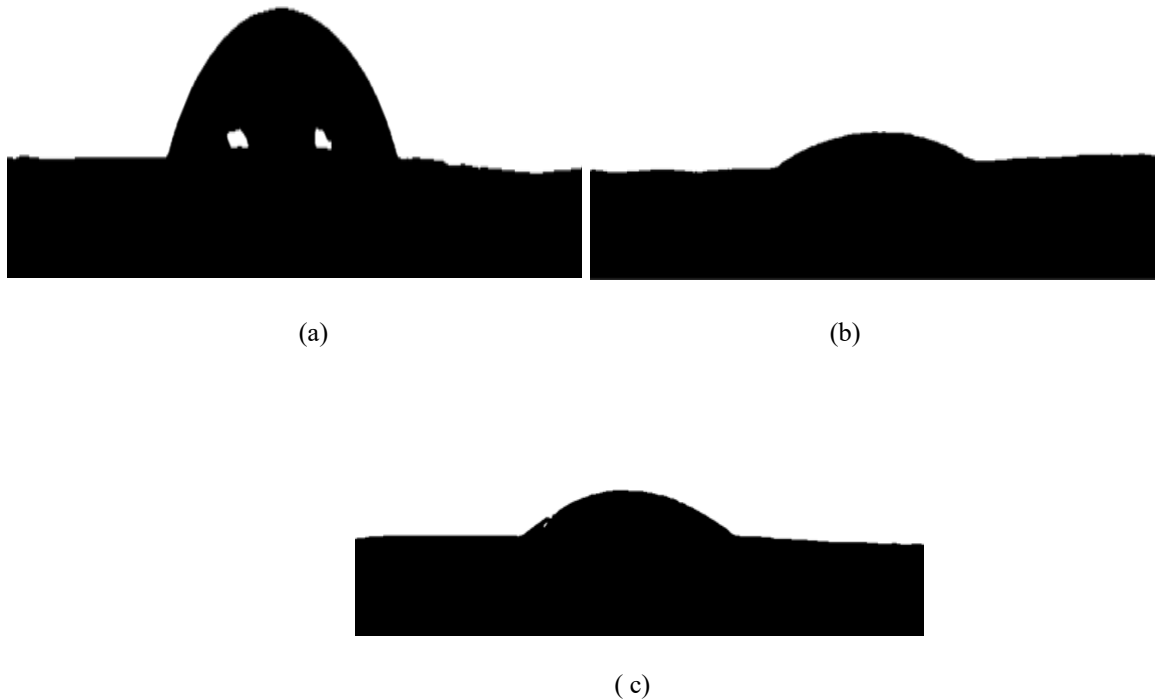
WCA Characterization

Figure 4. WCA Analysis of samples (a) 10% TEOS, (b) 20% TEOS, (c) 30% TEOS

In the water contact angle test in Figure 4, the angles obtained for each sample Analysis of samples (a) 10% TEOS, (b) 20% TEOS, (c) 30% TEOS were 50.56° , 25.58° and 40.78° . Increasing the TEOS content value given to the sample as a silica substitute resulted in a lower contact angle value.

CONCLUSION

This study successfully synthesized TEOS-derived silica materials through the sol-gel method. Characterization results confirmed the amorphous silica phase, uniform morphology, and elemental composition dominated by silicon. These findings highlight the potential of TEOS as a precursor for protective silica coatings. While this work provides fundamental insights into the structure and composition of TEOS-based materials, further studies involving hydrophobicity evaluation (contact angle measurements) and corrosion performance (electrochemical tests) are necessary to validate their application as superhydrophobic corrosion protection layers. Characterization results confirmed the presence of silica, while SEM analysis showed a uniform surface morphology. Furthermore, EDS and XRF analyses demonstrated the presence of elements, particularly silicon (Si), originating from TEOS. Increasing the TEOS content value given to the sample as a silica substitute resulted in a lower contact angle value. These findings indicate that TEOS can effectively serve as a silica precursor in synthesizing superhydrophobic protective materials.

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REFERENCES

- Kousar K, Walczak M S, Ljungdahl T, Wetzel A, Oskarsson H, P. Restuccia, Ahmad E A, Harrison N M, Lindsay R, 2021. Corrosion Inhibition Of Carbon Steel In Hydrochloric Acid: Elucidating The Performance Of An Imidazoline-Based Surfactant. *Corrosion Science*. **180**
- Qiushi D, Steffen J, Mohd R J, Paul W, Sabine. 2023. Hirth, Philipp Eiden, Jan N. Gorges, Xiao-Bo Chen, Patrick Keil, Ivan Cole. Synergistically And Sustainably Performed Inhibitors For Galvanised Steel Against Aqueous Corrosion. *Corrosion Science*. **213**
- Jinbo M, Jiaxin D, Xin G, Dongmei F, Lingwei M, Patrick K, Arjan M. 2023. Dawei Zhang. Data-Driven Corrosion Inhibition Efficiency Prediction Model Incorporating 2d-3d Molecular Graphs And Inhibitor Concentration. *Corrosion Science*. **111420**
- Ziqi Z, Junying H, Noam E, Li Z, Xi Y, Xiankang Z. 2022. Mercaptopropionic Acid-Modified Oleic Imidazoline As A Highly Efficient Corrosion Inhibitor For Carbon Steel In Co₂-Saturated Formation Water. *Corrosion Science*. **194**
- Cen H, Chen Z, Guo X. 2019. N, S Co-Doped Carbon Dots As Effective Corrosion Inhibitor For Carbon Steel In Co₂-Saturated 3.5% NaCl Solution. *Journal Of The Taiwan Institute Of Chemical Engineers*. **99**. 224-238
- Hongyu C, Jiaojiao C, Zhenyu C, Xingpeng G. 2019. 2-Mercaptobenzothiazole As A Corrosion Inhibitor For Carbon Steel In Supercritical Co₂-H₂O Condition. *Applied Surface Science*. **476**. 422-434
- Ade I. Fauzan H. Ismet H M, Jon A. Gunawarman. 2021. A Novel Fabrication Procedure For Producing High Strength Hydroxyapatite Ceramic Scaffolds With High Porosity. *Ceramics International* **47**, 26991-27001
- Hairul A, Riyan S, Satria, Melbi M, Fadli H, Jon A, Mochamad A, Dian H, Salit M S, Eni Sugiarti, Ahmad N, Muslimin. 2019. Comparative Study Of The Physical And Tensile Properties Of Jicama (*Pachyrhizus Erosus*) Starch Film Prepared Using Three Different Methods. *Biosynthesis Nutrition Biomedical*. **71** 5-6
- Sethi, S. K., Manik, G. 2018. Recent Progress In Super Hydrophobic/Hydrophilic Self-Cleaning Surfaces For Various Industrial Applications: A Review. *Polymer – Plastics Technology And Engineering*, **57** 1932–1952
- Zeng Q, Zhou H, Huang J, Guo Z. 2021. Review On The Recent Development Of Durable Superhydrophobic Materials For Practical Applications. *Nanoscale*, **13** 11734–11764
- Dayana I, Sembiring T, Tetuko A P, Sembiring K, Maulida N, Cahyarani Z, Setiadi E A, Asri N S, Ginting M, Sebayang P 2019. The Effect Of Tetraethyl Orthosilicate (Teos) Additions As Silica Precursors On The Magnetite Nano-Particles (Fe₃O₄) Properties For The Application Of Ferro-Lubricant. *Journal Of Molecular Liquids* **294** 111557.
- Ade I, Arya B P, Nofriady H, Hendriwan F, Nurzal, Asfarizal, Mastariyanto P, Anrinal, Adi S, Jon A, Gunawarman 2022. Behavior Of Sintered Body Properties Of Hydroxyapatite Ceramics: Effect Of Uniaxial Pressure On Green Body Fabrication. *Materials Today Sustainability* **17**
- Jon A, Murni H, Muhammad A A, Agung E, Harsojo S, Ardianto S, Wervyan S, Rikson S, Isa Anshori. 2023. Electrochemical And Capacitive Behavior Of Reduced Graphene Oxide From Green Reduction Of Graphene Oxide By Urea For Supercapacitor Electrodes. *Journal Of Materials Science: Materials In Electronics*. **34** 1638
- Power A C, Barrett A, Abubakar J, Suarez L J, Ryan L, Wencel D, Sullivan T, Regan F, 2016. Versatile Self-Cleaning Coating Production Through Sol-Gel Chemistry. *Advanced Engineering Materials*. **18** 76–82

- Adak D, Bhattacharyya R, Saha H, Maiti P S 2019 Sol-Gel Processed Silica Based Highly Transparent Self-Cleaning Coatings For Solar Glass Covers. *Materials Today Proceedings* **33** 2429–2433
- Kesmez O 2020 Preparation Of Hybrid Nanocomposite Coatings Via Sol-Gel Method For Hydrophobic And Self-Cleaning Properties. *Journal Of Molecular Structure*, 1205.
- Tian, W. Du, B. Shui A, Ke, S. Shan, W, He, C, And Ma J. 2020. Template-Free Preparation Of Humidity Self-Regulating Silica-Base Mesoporous Oxide *Journal Of Sol Gel, Science And Technology*, 94 (2), 416-424.
- K. Cao, Z. Yu, D. Yin. 2019. Preparation Of Ce-MOF@ TEOS To Enhance The Anti-Corrosion Properties Of Epoxy Coatings, *Prog. Org. Coat.* **135** 613–621,
- Chuangye W, Mengyu C, Jintang X, Huili L, Xinrong S, Xinyi Z, Longli Z. 2025. Fabrication Of Silica-Based Superhydrophobic Coatings With Remarkable Wear Resistance, Corrosion Inhibition Ability And Thermal Stability. *Applied Surface Science.* **682**. 161681
- Leng Y. 2008. *Materials Characterization Introduction To Microscopic And Spectroscopic Methods.* Science And Technology John Wiley And Sons (Asia) Pte.Ltd
- Xiaoling Y , Lihui X, Hong P, Yong S, Liming W And Mingrui X, 2021, Eco-Friendly Approach For Preparation Of Water-Based Superhydrophobic Silica Aerogels Via Ambient Pressure Drying. *Materials Research Express* **8** 015021].
- Sahadat H, Samina A. 2023. Easy And Green Synthesis Of Tio₂ (Anatase And Rutile): Estimation Of Crystallite Size Using Scherrer Equation, Williamson-Hall Plot, Monshi-Scherrer Model, Size-Strain Plot, Halder- Wagner Model. *Results In Materials.* **20**. 100492
- Li, X., Zhang, D., & Chen, H. (2021). Superhydrophobic coatings for corrosion protection: A review. *Journal of Materials Science & Technology*, 78, 1–15.
- Rahmawati, R., & Santosa, S. J. (2022). Synthesis and modification of silica-based coating for corrosion inhibition. *Indonesian Journal of Chemistry*, 22(4), 765–774.
- Chuangye, W., Zhang, X., Liu, H. & Song, L., 2025. Synthesis and corrosion inhibition performance of TEOS-based superhydrophobic coatings on carbon steel via sol–gel method. *Journal of Materials Science & Engineering*, 12(3), pp.245–258. doi:10.1007/s12014-025-03245-2.
- Wang, L., Sun, W., Zhang, D., & Li, J. (2020). *Fabrication of durable superhydrophobic films for corrosion protection of metals.* *Corrosion Science*, 90, 23–32. <https://doi.org/10.1016/j.corsci.2014.10.005>
- Wang, Y., Liu, H., Zhang, X., & Chen, J. (2025). *TEOS-derived superhydrophobic nanocomposite coatings for long-term corrosion resistance of carbon steel in aggressive environments.* *Journal of Materials Chemistry A*, 13(5), 1120–1132. <https://doi.org/10.1039/jmca.2025xxxx>