# RICE HULL ASH NANOSILICA: CHARACTERIZATION AND ITS POTENTIAL AS SILICATE COATING FOR CARBON STEEL

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**ABSTRACT:** Corrosion of steel structures is one of the main causes of destruction and loss of materials due to the damages it can bring to operating, storage, and structural equipment. Anti-corrosion inhibitors available in the market frequently contain chromium and lead, which are hazardous to human health and the environment. This study focused on the synthesis of nanosilica from the agro-industrial byproduct rice hull ash (RHA) and examined its potential as a corrosion barrier. Steel specimens were prepared in the form of 4mm thick disks cut from 16mm steel bars. RHA nanosilica was synthesized via acid precipitation method and characterized using x-ray diffraction (XRD), dynamic light scattering (DLS), atomic force microscopy (AFM), and Fourier Transform Infrared (FTIR) Spectroscopy. After characterization, 2.5% w/v RHA nanosilica was used to formulate sodium silicate as a coating for the steel specimens. The coated specimens were then subjected to Complex Impedance Spectroscopy (CIS) at 20Hz to 20MHz frequency range. The generated Nyquist plot showed that the specimen coated with sodium silicate with RHA nanosilica has a much larger diameter than red oxide indicating better protection against corrosion. The generated Bode plots confirmed this result as it showed that the specimens coated with sodium silicate with RHA nanosilica had higher impedances than those coated with red oxide, especially in the low-frequency regions.

Keywords: Corrosion, Nanosilica, Rice Hull Ash, Coating, Carbon Steel

# 1. INTRODUCTION

The demand for steel renovation and retrofitting projects is mainly caused by the corrosion and deterioration of previously installed steel products. Thus, it is of great interest for scientists and steel manufacturers to develop ways to inhibit and prevent the deterioration of these steel products, mainly caused by corrosion.

Several techniques and methods have been employed to prevent or protect metal products from corrosion. Techniques like barrier protection [1,2], and cathodic or anodic protection [3,4] have been employed by steel manufacturers and developers.

One technique currently employed to protect against corrosion is the use of inhibitors. Inhibitors are substances added in relatively low concentrations on the metal surface. The high corrosion inhibition activities of chromate and leadbased [5] inhibitors, such as molybdates [6], organic thioglycolates [7], and phosphonates [8], make them one of the most effective types of commercial corrosion inhibitors available today. Despite its efficacy, the inclusion of highly toxic chromateand lead-based compounds in the formulation of corrosion inhibitors make it an environmental and health hazard [9]. Due to the increasing presence of these inhibitors in the market, there is a great interest in developing alternative, non-toxic formulations, and techniques that remediate the effects of corrosion.

The emergence of novel techniques in enables the production nanotechnology of submicron-scale materials with excellent mechanical, physical, and chemical capabilities. Due to its extremely fine-grained size and highgrain boundary volume fraction, significant progress has been made in nanostructure manipulation for the development of coatings with wear and corrosion resistance. Some categories of materials that are recently developed for corrosion inhibition applications on carbon steel are: (i) nanocomposite alloys; (ii) polymer and polymercomposite coatings; (iii) self-assembled nanophase coatings; (iv) layer-by-layer nanoscale assembly; (v) self-healing paints and biocidal coatings; (vi) hydrophobic coatings; and (vii) ceramic-based anticorrosion coatings [10].

Silicate coatings are a class of corrosion inhibitors that can be produced by sol-gel techniques. These coatings act as a cathodic inhibitor that can be adsorbed onto the metal surface to form a thin protective layer. Furthermore, silicate corrosion inhibitors are non-toxic and considered natural [11,12]. Cathodic inhibitors are materials and compounds that produce a protective film that decreases the cathodic reaction rate by limiting the diffusion of oxygen to the metal surface. However, the disadvantage of silicate coatings is their tendency to form clusters that leave areas without coating.

Nanosilica can provide more effective and homogenous coating compared to ordinary silicatebased coatings due to its size and high reaction activities [13]. One of the most important characteristics of nanosilica as a component of anticorrosion coatings is the high surface area due to its small size. The high surface area allows a more effective cathodic inhibition which further limits the diffusion of oxygen to the metal surface thus preventing corrosion [14]. Furthermore, unlike native silicates, its nano-scale size has been reported to inhibit the propagation of existing corrosion on metal surfaces caused by pitting and crevice corrosion by inserting and filling these pits, holes, and crevices, thereby halting the spontaneous progression and electrochemical reaction of corrosion [15].

Ruzgal (2020) and his colleagues were able to develop a nanosilica-based surface coating for cold rolled steels using the nanosilica obtained from rice hull ash (RHA). SEM analysis of the steel samples were able to show the presence of sodium silicate hydrates on the microscopic crevices of the steel. The presence of the nanosilica-based coating on CRS decreased the soil-to-metal friction and adhesion coefficient of the steel which indicates the wide applicability of the coating not only on steel but also on farm and tillage implements [16].

The nanosilica used in this study was derived from rice hulls, an agro-industrial byproduct of rice milling. The rice hulls contain over 90% by weight silica depending on climate, rice variety, and soil chemistry [17]. Using RHA as source for nanosilica is ideal since it has little to no purpose in agricultural lands. Traditionally, rice hulls are disposed through open field burning which produces RHA [18]. This disposal method generates more unwanted air pollutants therefore, using RHA as a low-value source to generate a high-value product is highly desirable.

### 2. RESEARCH SIGNIFICANCE

Corrosion is considered as one of the major industrial problems, mainly in the systems involving water (e.g., cooling systems, steel pipelines, farm implements, and farm structures). The corrosion of steel structures is one of the predominant causes of destruction and loss of material due to the damages it can bring to operating, storage, and structural equipment. Since carbon steel or mild steel is a common construction material, its protection against corrosion is important for prolonging its life and structural integrity.

Rice hull ash, an agro-industrial waste from rice production, contains over 90% nanosilica, making extraction of nanosilica from RHA cost-efficient [19,20]. However, research on the use of nanosilica, synthesized from RHA, as protection against corrosion is scarce. This study aims to assess the potential capability of sodium silicate, with nanosilica from RHA, in protecting steel against corrosion. The specific objectives of the study are: (i) to characterize the nanosilica synthesized from RHA, and (ii) to determine the potential of sodium silicate coating with RHA nanosilica as an anticorrosion coating for steel by examining the generated impedance plots.

## 3. MATERIALS AND METHODS

The main materials in this study are nanosilica from RHA and carbon steel specimens. In this section, the characterization tests for nanosilica synthesized from RHA and carbon steel specimen are discussed. In addition, the methodology for complex impedance spectroscopy is explained.

### 3.1 Materials Used

### 3.1.1 Nanosilica

Hydrothermal nanosilica, the product of the proprietary technology of the UPLB Nanotechnology Subprogram for Engineering Applications, was obtained directly from ground rice hulls. The amorphous nanosilica used in this study is shown in Fig. 1.



Fig. 1. Amorphous nanosilica synthesized from rice hull ash.

#### 3.1.2. Carbon Steel

Commercially available round steel bars were used in this study. The size of steel bars used were 16 mm in diameter. These were cut into 4 mm thick samples and subjected to surface preparation in accordance with the ASTM G1 standard method. Existing rust, grit, and oil were removed using sandpaper with grades of 100 and 240. The steel specimens were subsequently washed with acetone. The sample steel specimen used in this study is shown in Fig. 2.





### 3.2 Characterization Tests for Nanosilica

The RHA nanosilica was subjected to various characterization tests to determine and evaluate its surface morphological characteristics. The characterization tests conducted in this study were summarized as follows:

# 3.2.1 Structural Characterization of Nanosilica by X-Ray Diffraction

X-ray diffraction (XRD) analysis of nanosilica was conducted using the Shimadzu XRD-6000 powder x-ray diffractometer equipped with a copper target with wavelength of 1.540598Å and accelerating voltage of 30kW. Analyses were done with a scan rate of  $2^{\circ}$  per minute and scan range of  $2^{\circ}$  to 90°. Post run analysis was done using X'Pert HighScore Plus software.

### 3.2.2 Hydrodynamic diameter of nanosilica

The hydrodynamic diameter of the purified nanosilica was determined in accordance with the ISO 22412:2017 using the Malvern Zetasizer Nano-ZS90 for the Dynamic Light Scattering (DLS) analysis. The RHA nanosilica powder was reconstituted in PEG/water and the resultant solution was sonicated for 5 minutes at room temperature. After sonication, the sample was subjected to DLS analysis. The average hydrodynamic diameter was calculated based on 6 runs with 12 readings each.

### 3.2.3 Atomic Force Microscopy of RHA Nanosilica

Morphological and topographic analysis of the RHA nanosilica powder was done using Park Systems XE-70 atomic force microscope in noncontact mode. A non-contact cantilever with a resonant frequency of 330kHz, force constant of 42N/m, length of 125µm, mean width of 30µm, and thickness of 4µm was used in the analysis. The sample was prepared by suspending a small amount of the RHA Nanosilica in absolute ethanol. The suspended nanosilica was then ultrasonicated for 20 minutes before depositing a small amount of the suspension to the mica surface. The sample was then air-dried prior to analysis. During atomic force microscopy (AFM) analysis, various scanning areas of 45 µm x 45 µm in size at different portions of the mica surface were obtained at a scan rate of 0.30 Hz. A scanning area of 0.62 µm x 0.62 µm was used to capture an image of a single RHA nanosilica particle.

## 3.2.4 Fourier Transform-Infrared Spectroscopy Analysis of RHA Nanosilica

The RHA nanosilica powder was subjected to the Attenuated Total Reflectance Fourier Transform-Infrared Spectroscopy analysis (ATR-FTIR) using the Shimadzu IR Affinity-1S Fourier Transform Infrared Spectrophotometer with the MIRacle 10 single-reflection ATR accessory. The RHA nanosilica powder sample was placed on top of the stage and was pressed firmly by the diamond/KRS-5 prism. The percent transmittance (%T) of the sample was then measured from  $400 \text{cm}^{-1}$ to 4000cm<sup>-1</sup>. The obtained FTIR spectrum was then transferred to the Lab Solutions IR software for atmosphere correction, baseline correction, and peak picking.

### **3.3 Characterization Tests for Steel**

### 3.3.1 AFM Analysis of the Steel Specimens

Morphological and topographic analyses of the steel specimens were conducted using the equipment and configurations used in method 3.2.3.

# *3.3.2 X-Ray Diffraction Analysis of Carbon Steel* Samples

X-ray diffraction (XRD) analysis of coated and uncoated carbon steel samples were conducted using the equipment and configurations used in method 3.2.1.

### 3.4 Complex Impedance Spectroscopy

The test specimen's real and imaginary impedance components were measured and recorded at varying frequencies (20 Hz to 20 MHz). The total impedance was measured and noted which follows Ohm's law defined by Eq. 1.

$$\mathbf{Z}(\mathbf{w}) = \frac{\mathbf{V}(\mathbf{w})}{\mathbf{I}(\mathbf{w})} = \mathbf{Z}_{\text{real}}(\mathbf{w}) + \mathbf{Z}_{\text{img}}(\mathbf{w}) \mathbf{j}$$
(1)

CIS was used to generate the Nyquist and Bode plot of the nanosilica-based coating. The Nyquist plot was formed by plotting the imaginary impedance component ( $Z_{img}$ ) in the y-direction and the real impedance component ( $Z_{real}$ ) on the xdirection. On the other hand, the Bode plot was created by graphing the total impedance (Z) recorded at frequencies ranging from 20 Hz to 20 MHz on a logarithmic scale. The total impedance was computed using Eq. 2.

$$|\mathbf{Z}(\mathbf{w})| = \sqrt{\mathbf{Z}_{real} (\mathbf{w})^2 + \mathbf{Z}_{img} (\mathbf{w})^2}$$
(2)

# 4. RESULTS AND DISCUSSION

### 4.1 AFM Analysis of Nanosilica

The surface morphology of the prepared RHA nanosilica powder was also examined using atomic force microscopy. Based on the topographic and 3D profile obtained (Fig. 3), the nanosilica sample was observed to be uniformly spherical. In addition, the surface topographic profile of the synthesized nanosilica showed the amorphous nature of the material due to the absence of lattice fringes in the generated AFM image.



Fig. 3. Surface topographic and 3D profile of the RHA nanosilica powder captured using atomic force microscopy.

### 4.2 XRD Analysis of Nanosilica

Since the structure of nanosilica formed from RHA depends on its treatment and strict temperature control during burning, it is essential to determine if the synthesis generated the amorphous nanosilica product sought for coating applications. Based on the results shown in Fig 4, a broad peak was observed at  $2\theta$  angle of  $22^\circ$ , which is a characteristic peak of amorphous nanosilica [21]. This result has indicated that the phase transformation and crystallization of nanosilica did not occur during synthesis and purification.



Fig. 4. X-ray diffractogram of purified nanosilica.

The amorphous character of nanosilica in this study can be attributed to the controlled thermal treatment via calcination of ground rice hull. The calcination temperature at 650°C is below the temperature at which phase transformation and crystallization of silica take place. Careful control of combustion of ground rice hull samples was important since uncontrolled calcination may result in the retention of contaminants which can affect the crystal characteristics of nanosilica [22].

### 4.3 DLS Analysis of Nanosilica

Based on the DLS analysis of purified RHA nanosilica (Fig. 5), the average hydrodynamic diameter of the nanoparticle dispersion was found to be  $17.815 \pm 0.7894$  nm with a polydispersity index (PDI) of  $0.366 \pm 0.1443$ . The PDI depicts the intensity of light scattered by a fraction of nanoparticles of different sizes [23]. For a nanoparticle dispersion to be considered highly monodisperse, the PDI must be ≤0.10. A nanoparticle dispersion categorized is as moderately polydisperse when the PDI has a value of 0.10 - 0.40. On the other hand, a nanoparticle dispersion with PDI greater than 0.40 is classified as highly polydisperse. The nanosilica surface is mainly composed of hydroxyl moieties which contribute to the high polarity of the nanosilica surface. This property contributes to the high

susceptibility of nanosilica particle aggregation and agglomeration, which can account for the presence of larger particle distribution.



Fig. 5. Size distribution of nanosilica in PEG/ethanol solution.

### 4.4 FTIR Analysis of Nanosilica

The rice hull ash nanosilica powder was also subjected to FTIR analysis. The FTIR spectra obtained (Fig. 6) showed multiple peaks that were indicative of nanosilica. Table 1 shows the FTIR peak assignment for the RHA nanosilica. The broad medium peak at 3100-3700 cm<sup>-1</sup> denotes the O-H stretching of the silanol group as well as the water present in the sample. On the other hand, the medium peak at 1643.35 cm<sup>-1</sup> indicates the H-O-H bending of water. The presence of the silanol group was also confirmed by the presence of a medium peak at 956.69 cm<sup>-1</sup> that indicates the bending of the Si-O-H moiety. Lastly, the peaks at 790.81 cm<sup>-1</sup> and 443.63 cm<sup>-1</sup> denote the Si-O-Si stretching and bending, respectively.



Fig. 6. FTIR spectra of the rice hull ash nanosilica powder.

Table 1. FTIR	spectra peak	assignment	for rice	hull
ash nanosilica	powder.			

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Frequency	Specific Bond Intens	
$(cm^{-1})$	Vibration	
3100-3700	O-H Stretching	Medium
1643.35	O-H Bending	Medium
1058.92	Si-O-Si Stretching	Strong
956.69	Si-O-H Bending Mediu	
790.81	Si-O-Si Stretching	Medium
		<b>a</b> .
443.63	S1-O-S1 Bending	Strong

### 4.5 AFM Analysis of Carbon Steel

Atomic force microscopy was used to visualize the topography of the steel disc surface. In atomic force microscopy, the nanoscale tip of the cantilever scans through the surface of the sample to probe its topography. A piezoelectric actuator controls the movement of the cantilever horizontally at XY scanning direction and vertically at Z motion direction. During the XY scanning direction, the cantilever bends while probing the topography of the sample due to the interaction force caused usually by van der Waals and electrostatic interactions between the tip of the cantilever and surface of the sample. To maintain the consistent oscillation of the tip while scanning, the Z piezo must move correspondingly [24].

In the AFM analysis of carbon steel after surface preparation (Fig. 7), it was observed that there were notable grooves present on its surface possibly caused by surface preparation and abrasion of SiC abrasive paper. Presence of bulbous material was also observed immediately after surface preparation which may indicate that corrosion formation on the carbon steel surface is slowly taking place. This phenomenon is frequently observed on air-exposed metal surfaces.



Fig. 7. AFM analysis of carbon steel specimen.

### 4.6 XRD Analysis of Carbon Steel

X-ray diffraction analysis was done to determine the presence and possible identities of corrosion products. Fig. 8 shows representative peaks of the steel discs samples that were analyzed.



Fig. 8. XRD of carbon steel specimen.

The XRD pattern obtained from the carbon steel show peaks at  $2\Theta = 44^{\circ}$ , 65°, and 82° which denote the three characteristic peaks of iron (Fe). However, small peaks at  $2\Theta = 24^{\circ}$ , 43°, 57°, and 62° suggest the beginning of corrosion product formation on the surface of carbon steel, which was verified by AFM analysis as the appearance of bulbous materials. Because the steel surface is exposed to air and moisture, the iron in the surface undergoes electrochemical reactions to produce Fe(OH)<sub>2</sub>. This will then be further oxidized to Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>, and  $\alpha$ -FeOOH [25]. After surface preparation, Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub> immediately form a thin film on the carbon steel surface which was seen in the obtained XRD pattern.

### 4.7 Nyquist and Bode Plots

CIS was used to evaluate the coating performance of sodium silicate hydrate coating with a nanosilica concentration of 2.5 % w/v versus red oxide primer. The components of the total impedance of each coating at different excitation frequencies were measured and their corresponding Nyquist and Bode plots were generated.

The coating performance can be easily interpreted by observing the diameter of the semicircle formed [26], wherein a larger diameter corresponds to a coating with better performance. The diameter of the semi-circle can be related to the charge transfer at the interface of the steel [27].

As shown in Fig. 9, the nanosilica coated specimen has a much larger diameter compared to the almost non-visible plot for the red oxide. This means that the nanosilica has the better coating protection performance. More information on the behavior of each coating with respect to varying frequencies were provided through the use of Bode plots. The total impedance at the low frequency region was used to interpret the coating's performance. The coating performance is represented by the plateau formed at the low frequency region. Higher plateaus represent better coating performance [28,29].



Fig. 9. Nyquist plots of different coatings

As shown in Fig. 10, the nanosilica coated specimen showed higher impedances especially at the lower frequency region. This further reaffirms the observation that the nanosilica coating performed better than the red oxide coating.



Fig. 10. Bode plots of different coatings

### 5. SUMMARY AND CONCLUSIONS

The notable conclusions from this study are enumerated as follows:

1. Rice hull ash can be used as cost-efficient precursor for amorphous nanosilica with high purity.

2. The synthesized nanosilica from RHA was spherical, and amorphous. Its hydrodynamic diameter was found to be  $17.815 \pm 0.7894$  nm with a PDI of  $0.366 \pm 0.1443$ . FTIR analysis led to spectra that showed multiple peaks that were indicative of nanosilica.

3. The characterized RHA nanosilica can be incorporated in a silicate-based eco-friendly and non-toxic coating for effective corrosion protection.

4. CIS results suggested that the formulated silicate coating performed significantly better than red oxide based on the generated Nyquist and Bode plots.

5. The generated Nyquist plot showed that the specimens coated with RHA nanosilica has larger diameter than red oxide which indicate better protection performance against corrosion.

6. The Bode Plot for specimens coated with RHA nanosilica showed higher impedances compared to red oxide coated specimens, especially at the low frequency regions denoting a better corrosion protection performance.

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