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Special Issue
A Themed Issue in Honour of Professor Onukwuli Okechukwu Dominic (FAS).

This special issue is dedicated to Professor Onukwuli Okechukwu Dominic (FAS), marking his retirement and celebrating a remarkable career. His legacy of exemplary scholarship, mentorship, and commitment to advancing knowledge is commemorated in this collection of works.

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Electrochemical Studies of Hybrid Botanical Extracts on Mild Steel Corrosion in Hydrochloric Acid Medium

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Abstract

Corrosion's detrimental effects on materials and structures compromise their functionality, leading to economic burdens through replacement, maintenance, and environmental degradation, thereby sparking interest in eco-friendly corrosion inhibitors. Although plants deemed weeds exhibit limited corrosion inhibition, hybridizing them with potent extracts enhances their efficacy. This study investigated hybrid extracts from water hyacinth, bitter leaf, and Moringa oleifera for controlling mild steel corrosion in 2M HCl solution, utilizing gravimetric and electrochemical techniques at 25°C ± 2°C. A 70:30 Eichhornia crassipes-Moringa oleifera hybrid extract demonstrated exceptional corrosion inhibition, achieving 65% gravimetric efficiency, 92.5% linear polarization resistance, and 94.7% potentiodynamic polarization efficiency. Surface-active components strengthened the protective metal coating, aligning with the study's primary goal and revealing a synergistic interaction between hybrid and individual inhibitors.

Keywords: Corrosion Inhibitor, Plant Extract, Mild Steel, HCl Solution, Green Inhibitor and Hybrid Corrosion Inhibitors

1. Introduction

Corrosion, a pervasive and debilitating issue across multiple industries, imposes considerable challenges due to its detrimental impact on metals, resulting in exorbitant costs for inspection, repair, and replacement, as well as risks to public safety and environmental well-being (Enekwe et al., 2024; Udoisoh et al., 2024). Corrosion, as defined by the International Union of Pure and Applied Chemistry (IUPAC), is a persistent degradation process that occurs when materials interact with their environment (Minniti et al., 2023). This natural phenomenon causes metals to revert to their original state, resulting in significant financial losses for industries like energy, manufacturing, and transportation (Salimi et al., 2023). The widespread use of corrosion inhibitors has raised concerns about their environmental impact and toxicity, emphasizing the importance of developing safer, more eco-friendly alternatives (Ullah et al., 2024).

The far-reaching consequences of corrosion necessitate urgent attention, with a staggering 5% annual loss of global GDP, according to the National Association of Corrosion Engineers. Corrosion poses significant risks to public safety and economic stability, threatening catastrophic infrastructure failures and devastating industrial accidents, with India alone facing estimated annual losses of $275 billion (Rechenchoski et al., 2020; Saleh et al., 2024; Ullah et al., 2024). Fortunately, eco-friendly corrosion inhibitors derived from organic materials offer a promising solution, providing effective metal surface protection while reducing reliance on toxic chemicals (Okediran et al., 2023; Omran et al., 2022). However, their development and adoption require addressing efficacy, scalability, regulation, and awareness challenges through collaborative research, industry, and policy efforts. By prioritizing sustainable corrosion mitigation, we can minimize environmental harm, protect infrastructure, enhance productivity, and foster innovation, ultimately paving the way for a more resilient and sustainable future (Schmitzhaus et al., 2024; Udoisoh et al., 2024).

Plant-based compounds, boasting an array of bioactive constituents such as alkaloids and flavonoids, have demonstrated remarkable corrosion-mitigating capabilities, heralding a new era in the development of eco-friendly corrosion inhibitors (Yadav et al., 2024). Leveraging their innate biodegradability, these innovative inhibitors promise a toxic-free alternative, streamlining disposal processes and aligning industrial practices with environmental stewardship (Okediran et al., 2023; Omran et al., 2022). Derivable from various plant components, including fruits, leaves, and roots, these extracts offer a promising alternative. Given the widespread industrial use of acidic solutions, particularly hydrochloric acid, due to its cost-effectiveness and efficiency, identifying environmentally friendly to safeguard metals against corrosion in acidic environments, especially hydrochloric acid, is of paramount importance (Oyewo, 2024; Oyewo et al., 2023; Oyewo et al., 2022; Ullah et al., 2024).

Corrosion's detrimental impact on material integrity necessitates urgent attention across various engineering disciplines, due to its far-reaching environmental consequences, including costly repairs and safety risks (Azeez et al., 2021; Azevedo et al., 2019). Although Eichhornia crassipes is readily available, its inherent limitations as a corrosion inhibitor highlight the need for enhancement. Fortunately, recent studies have shifted focus towards developing innovative, low-toxicity, eco-friendly corrosion inhibitors, with numerous plants demonstrating potential as rich sources of novel inhibitors. Leveraging phytochemicals such as alkaloids and flavonoids, these natural compounds effectively mitigate corrosion by adsorbing onto metal surfaces, underscoring the vast potential of plant-based solutions in combating corrosion-related challenges (Hoang et al., 2021; Li et al., 2022; Wu et al., 2019).

Given the potential of phytochemicals in plant extracts to combat corrosion, researchers have explored various botanical sources for their corrosion-suppressing properties. To maximize the utility of available plant resources, combining extracts with varying efficacy levels may offer a synergistic approach. This investigation employs weight loss and electrochemical methods to assess the corrosion inhibition efficiency of hybrid extracts derived from Eichhornia crassipes, Vernonia amygdalina, and Moringa oleifera on mild steel. Most corrosion inhibition studies have focused on individual botanical extracts, with limited research on hybrid extracts. Additionally, the electrochemical behavior of hybrid botanical extracts in hydrochloric acid medium is not well understood. This study seeks to fill this knowledge gap by investigating the corrosion inhibition efficiency and electrochemical behavior of hybrid botanical extracts. The objectives of this study are to evaluate the corrosion inhibition efficiency of hybrid botanical extracts on mild steel in hydrochloric acid medium, elucidate the synergistic effects of combining different plant extracts, and characterize the electrochemical behavior of hybrid botanical extracts. The study also aims to identify the most effective hybrid extract for corrosion inhibition and contribute to the development of sustainable and eco-friendly corrosion inhibitors for industrial applications.

2.0. Experimental Methods

2.1. Specimen Preparation

The techniques outlined by Omran et al. (2022) serve as the foundation for the stock solution procedure. After being gathered in their natural condition, fresh water hyacinth, moringa, and bitter leaf leaves were cleaned under running water, allowed to dry in the shade for 17 days, and then milled into a powder. A Soxhlet extractor was used to extract 300g of dried powdered water hyacinth leaves and 150g of moringa and bitter leaves each using 600 ml of ethanol. A condenser that gathered water from a nearby faucet was attached to the Soxhlet extractor. The ethanol was evaporated by the condenser, then it condensed and trickled onto the plant leaves that were pulverized and placed in filter paper inside the extractor. This cycle continued, allowing the ethanol to soak into the powdered leaves until saturated, after which the extract was discharged back into the round-bottom flask, yielding the desired extract.

2.2 Electrochemical Measurement

2.2.1 Linear Polarization Resistance Measurement

An electrochemical investigation employed a three-electrode setup, featuring a platinum counter electrode, a saturated calomel reference electrode, and a mild steel working electrode with a 1 cm^2 exposed surface. Conducted at ambient temperature (25 ± 2°C) in a 2M hydrochloric acid electrolyte solution, the study utilized a Potentiostat Galvanostat Instrument controlled by NOVA 1.10.1.9 software. The research evaluated two scenarios: corrosion behavior without inhibitors and with extracted inhibitors, providing valuable insights into the efficacy of these compounds in mitigating corrosion.

To evaluate the corrosion resistance of the specimens, a comprehensive electrochemical analysis was conducted. This involved sweeping the potential from -25 mV to +25 mV relative to the corrosion potential (E_corr) at a scan rate of 0.125 mV/s, generating linear polarization curves that enabled the calculation of polarization resistance (Rp). Furthermore, cyclic sweep polarization tests were performed, commencing at a cathodic potential and progressing to an anodic potential, while monitoring the open circuit potentials of the working electrode. Subsequent calculations utilizing a standardized formula yielded the inhibitory efficiency (IE) of the extracted inhibitors, providing valuable insights into their effectiveness in mitigating corrosion is presented in Equation (1).

$$I.E = \frac{\mathrm{Rpi} - \mathrm{Rpo} \quad \times 100}{\mathrm{Rpi}} \qquad (1)$$

The inhibitory efficiency calculation utilizes polarization resistance values, denoted as Rpi (with inhibitors) and Rpo (without inhibitors), representing the specimen's resistance to corrosion in the presence and absence of inhibiting agents, respectively.

2.2.2 Corrosion rate

The corrosion penetration rate can be quantitatively determined utilizing the following equation:

$$\mathrm{CR} = \frac{\mu\mathrm{eqKp\ x\ Icorr}}{\rho} \qquad (2)$$

The corrosion rate calculation incorporates the following constants:
- μeq: the equivalent weight constant, equal to 27.93 atomic mass units (amu)
- Kp: a proportionality constant, valued at 3.272×10^{-3} (μA·cm/year)
- ρ (Theta): the density of the metal, expressed in grams per cubic centimeter (g/cm^3).

2.2.3 Potentiodynamic Polarization Measurement

To evaluate the inhibitor's effectiveness in preventing mild steel corrosion, a Tafel polarization analysis was conducted. This involved applying a controlled voltage sweep, ranging from -250 mV to +250 mV, at a rate of 1 mV/s. By analyzing the resulting polarization curves, the corrosion current density (I_corr) was determined by extrapolating the linear segments to the corrosion potential (E_corr). The inhibitor's efficiency was then quantified by calculating the inhibition efficiency (IE) using a standard formula (Equation #3) (Fernandez et al., 2016), providing a clear understanding of the inhibitor's corrosion-mitigating properties:

$$I.E = \frac{\mathrm{Icorro} - \mathrm{Icorr\ I} \times 100}{\mathrm{Icorro}} \qquad (3)$$

Icorri and Icorro represent the corrosion current densities under two conditions:
1. Icorri: in the presence of inhibitors
2. Icorro: in the absence of inhibitors.

2.2.4. Impedance Spectroscopy Study

Impedance spectroscopy is a potent tool for investigating corrosion in materials, as it illuminates the electrochemical interactions occurring at the material-environment interface. Understanding resistance and impedance is crucial in this context. While Ohm's Law (E = IR) defines resistance in DC circuits as the voltage-to-current ratio, AC circuits introduce impedance (Z), encompassing both resistance and reactance – the opposition to current caused by capacitance and inductance. This relationship is expressed as in Equation (4).

$$E = IZ \qquad (4)$$

In this context, impedance (Z) is represented as a complex quantity, typically expressed in the form: Z = R + jX. Here, R denotes the resistance, X represents the reactance, and j signifies the imaginary unit.
Impedance spectroscopy offers a powerful tool for unraveling the complex frequency-dependent behavior of electrochemical systems, revealing key factors driving corrosion rates, including double layer capacitance and charge transfer resistance. By examining impedance data, researchers can elucidate the underlying corrosion

processes and evaluate the efficacy of protective coatings and mitigation strategies. As formulated by Enekwe et al. (2024), the total impedance (Ztotal) can be described by the Equation (5):

$$Z\ (Total) = Rs\ + ZI\ -\ ZiL \qquad (5)$$

where Rs represents the solution resistance. Notably, at high frequencies, impedance (Z) converges to Rs. A direct relationship exists between increased solution resistance, polarization resistance, and enhanced corrosion resistance. To elucidate the inhibition mechanisms, Nyquist plots were generated for mild steel interfaces with and without hybridized plant extracts at various concentrations.

3.0 Results and Discussion

This section describes the procedure and combination of different inhibitor used in this study. This is presented in Table 1. The 70:30, 90:10, and 80:20 ratios of Water Hyacinth (WH), Moringa (M), and Bitter Leaf (B) were chosen to investigate the optimal blend of bioactive compounds for corrosion inhibition. These ratios were selected to evaluate the synergistic effects of combining WH's phenolic compounds, M's isothiocyanates, and B's saponins and flavonoids. The varying proportions of WH, M, and B in each ratio allow for the identification of the most effective combination, with the 70:30 ratio expected to provide a balanced blend, while the 90:10 and 80:20 ratios examine the effects of dominant WH and varying M and B proportions.

While the control experiment did not contain any inhibitor (blank), all experiments, including those with the hybrid extracts in 70:30, 90:10, and 80:20 ratios of Water Hyacinth, Moringa, and Bitter Leaf, were repeated three times to ensure accuracy and reliability of the results.

Table 1: Experimental samples description

S/N	Abbreviation	Water hyacinth leaf extract (WH) %	Moringa leaf extract (M) %	Bitter leaf extract (B)%
1	Blank	0	0	0
2	$WH_{90}M_{10}$	90	10	
3	$WH_{80}M_{20}$	80	20	
4	$WH_{90}M_{30}$	70	30	
5	$WH_{90}B_{10}$	90	-	10
6	$WH_{80}B_{20}$	80	-	20
7	$WH_{70}B_{30}$	70	-	30

3.1. Linear Polarization Measurement

Table 1 and Figure 1 demonstrate that various concentrations of Moringa and bitter leaf extracts effectively mitigated both cathodic and anodic reactions, indicating their role as mixed-type inhibitors. By reducing corrosion densities on both sides of the polarization resistance, these plant extracts decrease the overall corrosion rate as current density decreases. Notably, in aggressive 2 M HCl environments, the W3 inhibitor exhibited exceptional corrosion protection for mild steel. Increasing Moringa concentrations enhanced inhibitory efficiency, likely due to increased phytomolecule adsorption onto the metal-solution interface, amplifying the protective effect as reported by Ezugha and Aralu (2023).

Table 2: Electrochemical parameters from linear polarization measurements for mild steel corrosion in 2M hydrochloric acid with and without hybridized plant extract inhibitors

Mixed inhibitors	Ba (mVdec1)	Bc (mVdec1)	Ecorr (mV)	Icorr (μA)	IE%	CR (mm/yr)	Rp (Ωcm^2)
Blank	99.8	75.5	-486.2	214.0		-1.48	55.5
$WH_{90}M_{10}$	91.7	77.8	-486.0	42.0	80.5	0.49	266.8
$WH_{80}M_{20}$	84.6	88.1	-479.8	35.0	82.7	0.41	266.8
$WH_{90}M_{30}$	65.2	77.1	-461.0	12.3	93.5	0.14	731.3
$WH_{90}B_{10}$	76.0	81.4	-483.9	48.6	77.1	0.51	228.0
$WH_{80}B_{20}$	81.5	86.3	-479.2	41.7	79.4	0.48	252.5
$WH_{70}B_{30}$	91.7	77.8	-484.0	42.0	80.5	0.41	265.8

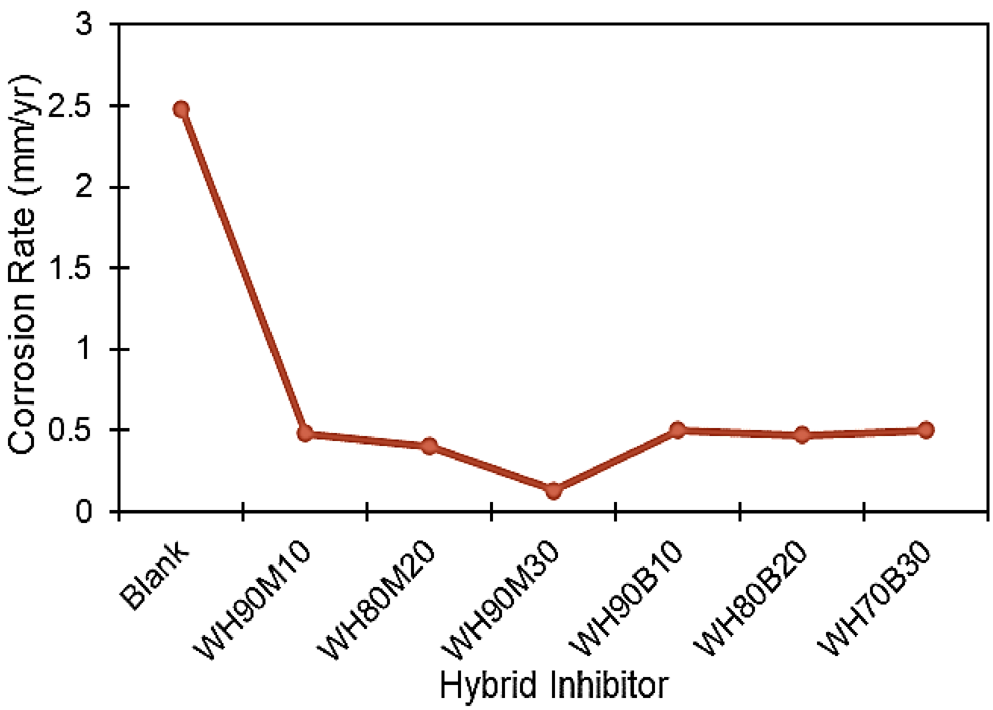

Figure 1: Graph of corrosion rate against the inhibitors for electrochemical test

3.2 Potentiodynamic Polarization

Table 2 reveals a consistent decrease in corrosion current density (Icorr) and a corresponding increase in polarization resistance (Rp) with rising concentrations of Moringa and Bitter leaf extract. The inverse relationship reveals that adsorption of phytomolecules from inhibitors onto the metal surface is enhanced, forming a protective physical barrier that restricts mass and charge transfer, thereby safeguarding the metal surface by blocking access to reactive sites. Furthermore, since the corrosion potential shift remains below 85mV, the extracts are categorized as mixed-type inhibitors, effectively mitigating both anodic and cathodic reaction rates.

Table 3: Corrosion behavior of mild steel in 2M HCl: Potentiodynamic polarization parameters in the absence and presence of hybridized plant extract-derived inhibitors

Mixed inhibitors	Ba (mVdec1)	Bc (mVdec1)	Ecorr (mV)	Icorr (µA)	IE%	Rp (Ωcm^2)
Blank	99.8	75.5	-484.2	214.0	-	55.5
$WH_{90}M_{10}$	91.7	77.8	-484.0	42.0	81.8	266.8
$WH_{80}M_{20}$	84.6	88.1	-479.8	35.0	85.0	299.0
$WH_{90}M_{30}$	65.2	77.1	-461.0	12.3	95.7	731.3
$WH_{90}B_{10}$	76.0	81.4	-483.9	46.6	76.7	229.0
$WH_{80}B_{20}$	81.5	86.3	-479.2	41.7	81.9	253.5
$WH_{70}B_{30}$	91.7	77.8	-484.0	42.0	81.8	266.8

3.3. Impedance Spectroscopy Study

Table 4 presents the corrosion characteristics of mild steel in 2M hydrochloric acid derived from Nyquist Curves with and without inhibitor addition. Figure2 reveals a progressive enlargement of the Nyquist plot's semi-circle diameter as the inhibitive molecule concentration increases from 10% to 30% in the extract. This corresponds to an enhancement in the compactness of the adsorbed barrier layer, resulting in a steady decline in acid corrosion rates of mild steel. Notably, the Nyquist plots exhibit depressed semi-circles with centers below the real X-axis, indicative of solid electrode characteristics and frequency dispersion effects. This dispersion is attributed to surface roughness and inhomogeneity, underscoring the complex interplay between inhibitive molecules and the metal surface. Meanwhile Figure 4 presents the corrosion characteristics of mild steel in 2m hydrochloric acid derived from Nyquist curves with and without inhibitor addition.

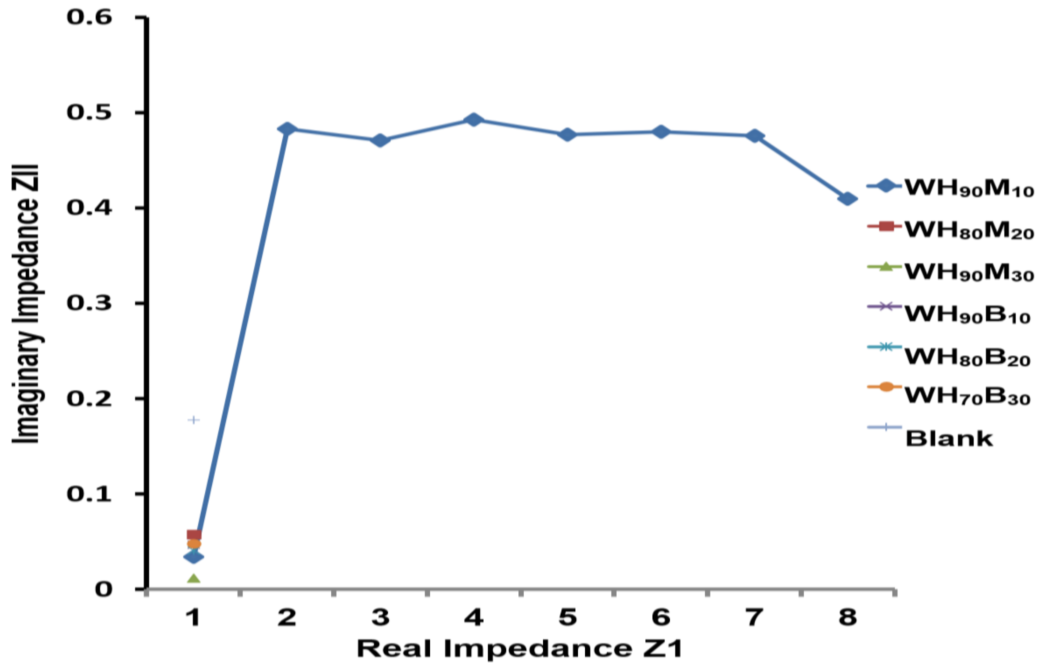

Figure 2: Nyquist plot in the presence and absence of inhibitors

Table 4. Corrosion characteristics of mild steel in 2M hydrochloric acid derived from Nyquist curves with and without inhibitor addition

Mixed inhibitors	ZI (Ωcm^2)	ZII (Ωcm^2)
$WH_{90}M_{10}$	0.035	0.484
$WH_{80}M_{20}$	0.059	0.472
$WH_{90}M_{30}$	0.013	0.494
$WH_{90}B_{10}$	0.048	0.478
$WH_{80}B_{20}$	0.042	0.481
$WH_{70}B_{30}$	0.049	0.477
Blank	0.179	0.411

The powdered extracts were then mixed in varying ratios to create different combinations. The focus was on the 70% Water Hyacinth and 30% Moringa (WH70M30) combination. The mixtures were prepared by weighing the required amounts of each extract and mixing them thoroughly. The inhibition efficiency of each extract mixture was evaluated using a standardized assay. The assay measured the ability of the extract mixture to inhibit the growth of a specific microorganism. The standout result of this study was the impressive inhibition efficiency achieved by the WH70M30 combination. This combination demonstrated a significant enhancement in inhibitory activity compared to the individual extracts. The synergy between the Water Hyacinth and Moringa leaf extracts in this ratio highlights the potential for developing novel, plant-based inhibitors. Notably, the WH70M30 combination outperformed other extract mixtures, including WH80M20 and WH90M10, which achieved inhibition efficiencies ranging from 60% to 80%. The remarkable inhibition efficiency of the WH70M30 combination has significant implications for the development of sustainable, plant-based inhibitors. This finding suggests that optimizing extract ratios can lead to enhanced inhibitory activity. Future studies can focus on scaling up the extraction process, optimizing the formulation of the extract mixture, and evaluating its efficacy in various applications. Overall, this study demonstrates the potential of combining Water Hyacinth and Moringa leaf extracts to develop effective and sustainable inhibitors.

4.0 Conclusions

The combined plant extracts demonstrated exceptional inhibition capabilities, achieving efficiencies exceeding 50% in 2M HCl across various concentrations. Notably, hybrid extract $WH_{90}M_{30}$ exhibited superior inhibition of 66%, outperforming other hybrid materials, while $WH_{90}M_{10}$ and $WH_{80}M_{20}$ also displayed significant inhibition. Electrochemical assessments demonstrated the efficacy of all inhibitors in mitigating mild steel corrosion within a 2M HCl environment over the duration of the experiment at room temperature. The outstanding performance of $WH_{70}M_{30}$ was primarily attributed to its pronounced tendency to form a protective layer on the mild steel surface. Furthermore, the synergistic combination of Moringa oleifera and Eichhornia crassipes extracts in a 30:70 ratio yielded a mixed-mode inhibitor, achieving a notable inhibition efficiency of 65%. This remarkable outcome underscores the potential of these plants, often regarded as nuisance species, to be repurposed as valuable resources in various industrial contexts.

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