Research Article

Journal of
Environmental
Nanotechnology

Biogenic Silver Oxide Nanoparticles for Inhibition of TMT Rod Corrosion in Marine Environment

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Received: 01.03.2024 Accepted: 01.06.2024 Published: 30.06.2024
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ABSTRACT

The current study explores the utilization of *Thespesia populnea* mediated biogenically synthesized silver oxide using as a protective coating for TMT rods in a marine environment. The process commenced with the extraction of the inhibitor from the leaves of the plant using ethanol followed by the preparation of silver oxide nanoparticles (AgONPs). Subsequently, TMT rods were coated with these AgONPs and subjected to the corrosive conditions of marine environment. In this investigation, AgONPs were synthesized employing chemical precipitation method and their size effects were thoroughly examined using techniques such as, Fourier Transform Infrared Spectroscopy, Fourier Ultraviolet-visible Spectroscopy and SEM. Remarkably, when TMT rods were coated with 10 layers of AgONPs, the corrosion rate efficiency escalated to 97.1% for 8 mm rods, 95.2% for 10 mm rods and 98.7% for 16 mm rods. Furthermore, the inhibition data conform to the Langmuir adsorption isotherm suggesting that the action of AgONPs followed a physical adsorption mechanism.

Keywords: Silver oxide nanoparticles; Biogenic nanoparticle; Corrosion inhibition; Marine environment; Brackish water.

1. INTRODUCTION

Corrosion refers to the degradation of mechanical properties induced by chemical or electrochemical reactions between a material and its environment (Li *et al.* 2015; Jeffrey *et al.* 2024). Corrosion is a global problem impacting materials, especially metals and alloys, which need efficient mitigation to prevent the negative impacts. Furthermore, corrosion is commonly regarded as a widespread issue (Fernandes *et al.* 2015). Since most metals reside in the form of oxides, they require a significant amount of energy to be extracted into their free state. When exposed to an external environment such as, moisture or oxygen, they may return to their combined state using the energy provided (Jeffrey *et al.* 2024). Electrochemical corrosion causes metals to revert to their original state and lose their properties, compromising their performance and durability (Fernandes *et al.* 2015). Metallic corrosion can take several forms based on the type of metal damage. Corrosion may attack the metal’s surface and progressively reduce its thickness.

Corrosion diminishes around 20% of the total production of iron annually. Corrosion of materials causes significant economic loss as well as loss of life globally each year. Corrosion can cause losses anywhere from 3% to 5% of a nation’s GDP (Koushik *et al.* 2021). The annual economic loss caused by different forms of corrosion in the United States is expected to be roughly 3.2% of its GDP (Harsimran *et al.* 2021; Koushik *et al.* 2021; Maaß and Peter, 2011). The 2016 corrosion survey by the American Society of Corrosion Engineers predicted a global cost of USD 2.5 trillion (Xhanari *et al.* 2019; Soltis *et al.* 2018). In 2015, the Chinese Academy of Engineering initiated a consultancy project titled "Research on Chinese corrosion status and control strategy". In 2014, China's yearly corrosion cost was around 3.34% of its GDP (Li *et al.* 2015; Zhang *et al.* 2015; Kuznetsov *et al.* 2020; Xhanari *et al.* 2020; Soltis *et al.* 2018; Hou *et al.* 2018). In India, the corrosion damage costs millions of USD annually in various sectors such as, agriculture, industrial and other service sectors accounting for 4.2% of its GNP (Gretchen *et al.* 2016). Corrosion control strategies might save 15-35% of corrosion cost, amounting to a global savings of USD 375 to 875 billion per year (Gretchen *et al.* 2016). Marine corrosion loss accounts for approximately 30% of overall corrosion loss. Seawater corrosion poses a significant risk to marine platforms such as, ships, bridges, and offshore structures that have been in service for more than 15-20 years (Wang *et al.* 2015). There is an urgent requirement to investigate corrosion behaviour and design corrosion-resistant materials for marine environments (Xian-man *et al.* 2022).

Several approaches exist for retarding or preventing corrosion in metallic constructions. Metals are typically protected by protective coatings made of organic molecules, plastics or polymers as well as cathodic or anodic protection with organic or inorganic inhibitors. Compounds based on chromium have been shown to be efficient corrosion inhibitors. Nonetheless, the issue of toxicity linked with chromium-based compounds is gaining attention (Sakunthala *et al.* 2013). Corrosion inhibitors function by producing a protective barrier film on the metal's surface thereby decreasing corrosion. In the metal sector, both inorganic and organic inhibitors are commonly used to combat corrosion. However, inorganic inhibitors are rarely used because of their high cost and probable harm to the people and the environment.

Not all engineering alloys, even those with desired qualities, are corrosion resistant. It is also unlikely that their inherent corrosion resistance can be increased to the appropriate level. Surface treatments and coatings enable the use of such metals in the intended applications. At the same time, increased environmental awareness has rendered some surface treatment methods and coatings outdated. As a result, developing science and technology for ecologically friendly multifunctional and smart coatings is critical (Raja *et al.* 2022).

Steel is one of the largest and oldest materials utilized in engineering applications. It has been employed in structural and engineering applications for decades due to its excellent mechanical qualities, both on land and in sea. The purpose of delivering suitable steel is to strengthen the member. Steel of proper quality and quantity can offer strength to a member during its projected life. Structural steel is prone to corrosion from environmental factors (Aminul *et al.* 2015). Thermomechanically treated (TMT) rods are important structural elements used in major buildings, water retention structures, ports, harbours and bridges. These rods are prone to corrosion, particularly under unfavourable conditions (Donatello *et al.* 2013). Corrosion of bars placed in concrete is a major factor affecting the durability of marine structures (Vedalakshmi *et al.* 2008). Nonetheless, TMT rod corrosion is a key contributor to structural concrete failures. Several solutions exist to address this issue, including surface treatments, the addition of inhibitors to the concrete and concrete coatings (Pandian *et al.* 2015). In order to identify the most effective organic inhibitor, a large number of organic inhibitors were examined (Gopiraman *et al.* 2011; Kavitha *et al.* 2016; Kalaiselvi *et al.* 2020; Kesavan *et al.* 2012; Pandian *et al.* 2015; Rajeswari *et al.* 2017; Kandaswamy *et al.* 2021)

Nanotechnology is a rapidly developing science with a wide range of applications. Nanoparticles are used in a variety of industries due to their excellent physical and chemical features such as, enhanced surface area, mechanical strength, optical activity and chemical reactivity (Mohammad Shafiee *et al.* 2018; Parthasarathy *et al.* 2014; Rai *et al.* 2012). Nanoparticles are particularly useful in industrial settings due to their extraordinary ability to prevent metal corrosion in a variety of environments. Nanomaterials with their high surface-to-volume ratio are far more effective corrosion inhibitors than standard macroscopic materials. In today's ever-changing scientific situation, researchers have made significant advances in the production of new nanoscale materials that show great potential in a wide range of fields (Mohammad Shafiee *et al.* 2018). The exact design of experimental procedures to produce nanoparticles with a wide range of chemical compositions, sizes, morphologies and behaviours is an important component of nanoscience. Traditionally, these nanoparticles are synthesized using chemical or physical processes. However, current efforts have focused towards biological techniques to produce nanoparticles, which provide various benefits such as, safety, environmental friendliness, cost-effectiveness and reduction in toxic byproducts (Rai *et al.* 2011; Rai *et al.* 2012; Salem *et al.* 2021).

In this respect, the current study looks into the domain of nanoscale corrosion prevention with particular emphasis on the use of green-synthesized silver oxide nanoparticles to reduce marine corrosion of important structural components such as TMT rods. Silver oxide nanoparticles hold great promise for increasing the durability and operational longevity of TMT rods in marine environments due to their environmentally acceptable production techniques and intrinsic corrosion resistance. This study provides a substantial step towards long-term corrosion mitigation strategies that are consistent with the current necessity of environmental management.

2. MATERIALS AND METHODS

2.1 Preparation of the Inhibitor

Two kilograms of fresh *Thespesia populnea* (TP) leaves from Nagapattinam District were used to prepare the inhibitor. These leaves were dried for two weeks under shade. The shade-dried TP leaves were finely pulverised and extracted using Soxhlet technique with 85% methanol as the solvent (four rounds of 500 mL each) (Gopiraman *et al.* 2011; Kavitha *et al.* 2016; Kalaiselvi *et al.* 2020; Kesavan *et al.* 2012; Nadaro *et al.* 2017; Pandian *et al.* 2015; Rajeswari *et al.* 2017). The solution was filtered using a Whatman filter paper. The resultant filtrate was concentrated in a vacuum chamber yielding a material resembling green oil that was then refrigerated for future use.

2.2 Synthesis of AgONPs

In the first step, 100 mL of 0.01 M silver nitrate solution using 70% water and 30% ethanol. Subsequently, 50 mL of 0.01% crude TP extract solution was added to this solution and thoroughly stirred. Following a three-hour reaction period, an additional 50 mL of the 0.01% extract was incrementally introduced drop by drop and the mixture was stirred for an extra 25 minutes. The resulting particles were then separated through centrifugation followed by thorough washing with double distilled water and ethanol. The AgONPs were subsequently subjected to vacuum drying at room temperature for 24 hours and were subsequently stored in ethanol for future use.

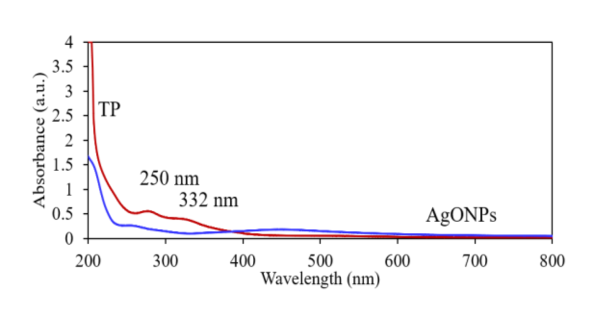

Fig. 1: UV-visible spectra of TP and AgONPs

2.3 Characterization of AgONPs

The green-synthesized silver nanoparticles were characterized using FT-IR (JASCO, FTIR-4100) and UV-visible spectrophotometry (JASCO, V-630). Furthermore, the surface morphology and elemental composition of the particles were assessed using scanning electron microscopy combined with energy dispersive spectroscopy (SEM-EDS) (Hitachi-S 3400N).

2.4 Weight Loss Measurements

In line with previous research (Kavitha *et al.* 2016; Kalaiselvi *et al.* 2020; Kesavan *et al.* 2012; Pandian *et al.* 2015), weight loss assessments were conducted. Thermomechanically treated rods of three different diameters, 8 mm, 10 mm and 16 mm with a length of 5 cm were abraded using emery sheets of varying grades (spanning from 600 to 1200). Subsequently, the rods were meticulously cleansed with distilled water and then treated with acetone. Each rod was then weighed and allowed to air dry at room temperature. A 100 ppm solution of AgONPs was prepared. The AgONPs was subsequently applied as 1, 5 and 10 coatings, separately on each of the TMT rods. As per the calculations, each coating for the TMT rod required 1 mL of 100 ppm AgONPs solution. Following each coating, a 10 minute drying period at room temperature was provided for all the rods. In this study, TMT rod specimens were utilized with the following chemical composition: C, 4.93%; Mn, 1.09%; Si, 1.78%; and the remaining constituent was predominantly Fe. Equations 1 and 2 were employed to quantify the inhibition efficiency (%IE), corrosion rate (CR) and surface coverage (θ).

$$IE(\%) = \frac{(W_2 - W_1)}{W_2} \times 100 \qquad \text{(1)}$$

$$\theta = \frac{(W_2 - W_1)}{W_2} \qquad \text{(2)}$$

The weight of the specimen before and after incubation in the marine environment is represented by W2 and W1, respectively.

3. RESULTS AND DISCUSSION

3.1 Analysis of Crude Extract and AgONPs

The properties of both the crude extract and AgONPs were assessed by UV-visible spectroscopy. Notably the crude extract exhibited two distinct spectral bands around 250 nm and 332 nm, indicative of the presence of flavones (Fig. 1) (Gopiraman *et al.* 2011; Kavitha *et al.* 2016; Kalaiselvi *et al.* 2020; Kesavan *et al.* 2012; Pandian *et al.* 2015; Rajeswari *et al.* 2017). On the other hand, AgONPs displayed a unique band at 219 nm suggesting that this feature might be attributed to the nanoparticles themselves. The FT-IR spectrum as illustrated in Fig. 2 revealed distinct peaks in the crude extract corresponding to the stretching vibrations of -O-H, -C-H, -C=O and -C-O observed at 3432 cm^{-1}, 3212 cm^{-1}, 1635 cm^{-1} and 1384 cm^{-1}, respectively. Distinctive peaks of AgONPs detected at 615 cm^{-1} indicated metal oxygen interactions (Meena *et al.* 2020).

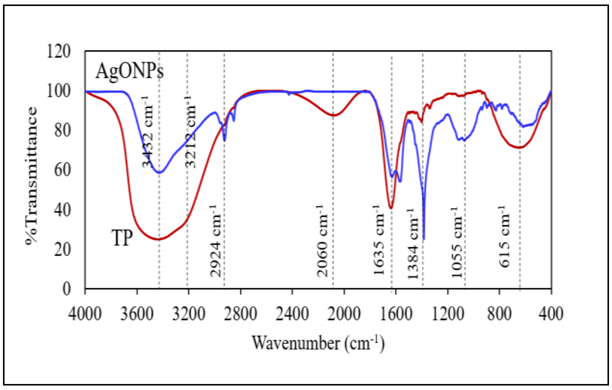

Fig. 2: FT-IR spectra of TP and AgONPs

Fig. 3 displays the SEM morphologies of AgONPs. The AgONPs showed an average diameter of 155 ± 23 nm. The SEM-ED spectrum was used to identify the elemental composition of nanoparticles, which showed 18.92% carbon, 17.26% oxygen and 63.81% silver as the main constituents. The effectiveness of AgONPs was meticulously assessed through their application on TMT rods of varying diameters (8 mm, 10 mm and 16 mm) with differing number of coatings (1, 5 and 10). This evaluation was conducted within a brackish water environment characterized by a salinity of 5 ppt. Both the TMT rods with AgONPs coatings and their uncoated counterparts were subjected to a thorough examination. The results as depicted in Fig. 4 and Table 1 underscore a noticeable correlation between the number of AgONPs coatings and the percentage of inhibition efficiency (%IE).

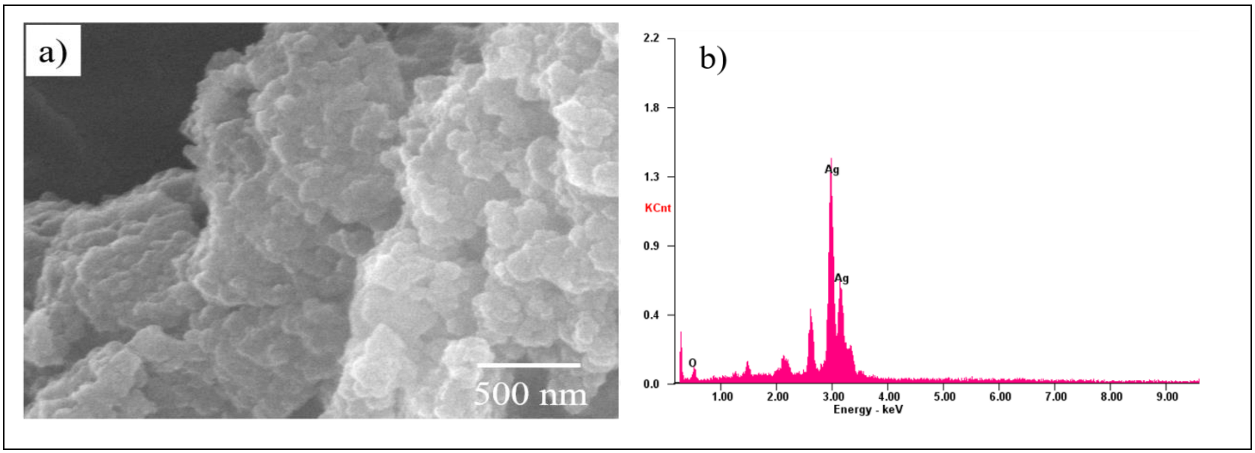

Fig. 3: SEM morphology and (b) EDS of AgONPs

Table. 1. Corrosion inhibition effectiveness of biogenic AgONPs and thermochemical parameters

TMT Rod	Coating number	IE%	Surface Coverage (q)	Corrosion Rate (mm/y)	K (kJ/mol)	ΔG_{ads} (kJ/mol)
	1	70.6	0.7059	0.9961	1200	-17684
8 mm	5	76.5	0.7647	1.0791	325	-14426
	10	97.1	0.9706	1.3697	1650	-18478
	1	70.2	0.7023	0.9910	1179	-17641
10 mm	5	74.8	0.7481	1.0556	296	-14201
	10	95.2	0.9519	1.3433	989	-17203
	1	76.5	0.7652	1.0798	1629	-18447
16 mm	5	85.7	0.8565	1.2086	596	-15943
	10	98.7	0.9870	1.392759406	3783.3333	-20548

ΔG_{ads} -The free energy of adsorption; K – adsorption equilibrium constant.

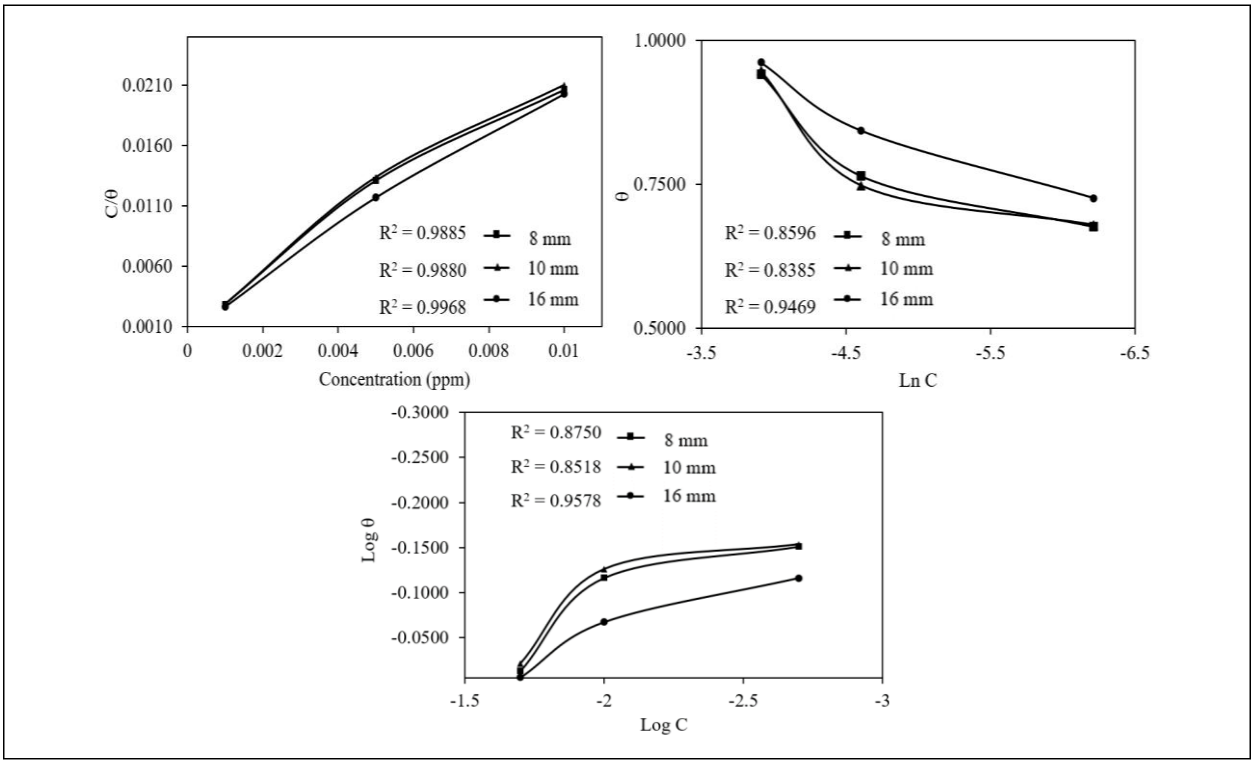

Fig. 5: (a) Langmuir, (b) Temkin and (c) Freundlich adsorption isotherms of corrosion inhibition of TMT rod in brackish water using biogenic AgONPs

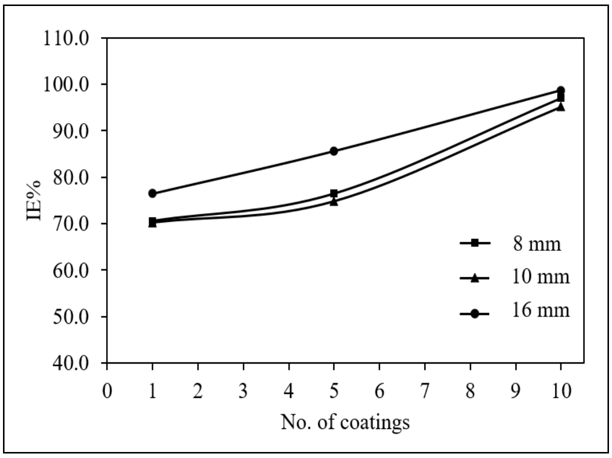

Fig. 4: Effect of biogenic AgONPs on corrosion inhibition of TMT rods in brackish water

For 8 mm TMT rods, the inhibitor initially displayed a %IE of 70.6% which subsequently increased to 76.5% after 5 coatings. Impressively, after 10 coatings, the TMT rod showed an exceptional %IE of 97.1%. For 10 mm TMT rods, the efficiency of AgONPs started at 70.2% for the initial coatings and this figure steadily increased to an impressive 74.8% after 5 coatings, culminating in a remarkable 95.2% inhibition after 10 coatings. The inhibition efficiency of AgONPs on 16 mm TMT rods exhibited a range from 76.5% after 1 coating to 85.7% following 5 coatings ultimately reaching an outstanding 98.7% inhibition effectiveness after 10 coatings. It was observed that AgONPs effectively covered the entire surface of the TMT rods. Furthermore, the diameter of the rod increased the surface area and correspondingly the corrosion inhibition capability of AgONPs appeared to enhance. However, it is noteworthy that no significant improvement in %IE was observed with the addition of more AgONPs coatings. The behavior of AgONPs during adsorption offers valuable insights into the mechanism of corrosion inhibition. The weight loss data subsequent to adsorption isotherms is presented in Fig. 3. Notably, correlation coefficient values provide insight into the effectiveness of AgONPs.

Fig. 5a shows that AgONPs from TP exhibited a superior fit for the Langmuir adsorption isotherm with correlation coefficient values of 0.9885 for 8 mm rod, 0.9980 for 10 mm rod and 0.9968 for 16 mm TMT rod. This suggests a more effective adsorption mechanism for AgONPs when following the Langmuir model. In Fig. 5b, the Temkin adsorption isotherm for weight loss data is depicted illustrating non-linear correlations. The correlation coefficient values were found to be 0.8596 for 8 mm TMT rod, 0.8385 for 10 mm rod and 0.9469 for 16 mm rod. Fig. 5c presents the non-linear correlation of the Freundlich adsorption isotherm with correlation coefficient values of 0.8750 for 8 mm TMT rod, 0.8385 for 10 mm and 0.9469 for 16 mm.

The mechanism of inhibition can be attributed to the adsorption of the inhibitor on the TMT surface as suggested by previous studies (Aminul Islam *et al.* 2015; Sakunthala *et al.* 2013; Kalaiselvi *et al.* 2020; Kesavan *et al.* 2012). The enhanced inhibitive effectiveness of the inhibitor is likely due to the presence of heteroatoms and lone pair electrons in natural products along with their larger molecule size which facilitates better coverage of the metallic surface.

4. CONCLUSION

Amid growing concerns regarding the environmental impact and cost of corrosion, there is a focus on finding sustainable, affordable and effective corrosion inhibitors. This study explores the realm of eco-friendly inhibitors highlighting the remarkable potential of silver oxide nanoparticles derived from *Thespesia populnea* leaves. The environmentally friendly and cost-effective nature of green inhibitors, combined with their concentration-dependent efficacy, adherence to the Langmuir adsorption isotherm, mixed-mode inhibition mechanism, and empirical validation through advanced microscopy, collectively position these AgONPs as a potent asset for safeguarding critical marine infrastructure. By prolonging the lifespan and resilience of such infrastructure while promoting sustainable corrosion protection practices, these AgONPs hold the promise of being a game-changer in the field of corrosion protection.

ACKNOWLEDGEMENT

Monikandon Sukumaran thank Annamalai University, Chidambaram and Tamil Nadu Dr.J.Jayalalithaa Fisheries University, Nagapattinam, for encouragement towards research.

FUNDING

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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