



Nanocoating Is a New Way for Biofouling Prevention

Santosh Kumar^{1,2}, Fei Ye¹, Sergey Dobretsov^{3,4*} and Joydeep Dutta^{1*}

¹Functional Materials, Department of Applied Physics, School of Engineering Sciences, KTH Royal Institute of Technology, Stockholm, Sweden, ²Department of Food Engineering and Technology, Central Institute of Technology Kokrajhar, BTR, Assam, India, ³Department of Marine Science and Fisheries, Sultan Qaboos University, Muscat, Oman, ⁴Center of Excellence in Marine Biotechnology, Sultan Qaboos University, Muscat, Oman

Biofouling is a major concern to the maritime industry. Biofouling increases fuel consumption, accelerates corrosion, clogs membranes and pipes, and reduces the buoyancy of marine installations, such as ships, platforms, and nets. While traditionally marine installations are protected by toxic biocidal coatings, due to recent environmental concerns and legislation, novel nanomaterial-based anti-fouling coatings are being developed. Hybrid nanocomposites of organic-inorganic materials give a possibility to combine the characteristics of both groups of material generating opportunities to prevent biofouling. The development of bio-inspired surface designs, progress in polymer science and advances in nanotechnology is significantly contributing to the development of eco-friendly marine coatings containing photocatalytic nanomaterials. The review mainly discusses photocatalysis, antifouling activity, and formulation of coatings using metal and metal oxide nanomaterials (nanoparticles, nanowires, nanorods). Additionally, applications of nanocomposite coatings for inhibition of micro- and macro-fouling in marine environments are reviewed.

Keywords: nanocoating, antifouling, biocide, polymer, hydrogel, nanomaterial

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*Correspondence:

Joydeep Dutta
Joydeep@kth.se
Sergey Dobretsov
sergey@squ.edu.om

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INTRODUCTION

Biofouling is a process that any substrate in the marine environment is quickly covered by organisms. Biofouling is referred to as undesirable growth on submerged surfaces of micro- (bacteria and protists) and macro-fouling (invertebrates and algae) organisms (Wahl, 1989). Any submerged substratum is quickly covered with organic molecules and particles that can be colonized by microscopic organisms, forming biofilms (Wahl, 1989). Marine biofilms contain multiple species of bacteria, microalgae and diatoms (Salta et al., 2013). Biofilms can subsequently enhance or reduce settlement of larvae and spores of invertebrates and algae (Dobretsov et al., 2006; Dobretsov and Rittschof, 2020).

The maritime industry and naval forces across the world lose billions of US dollars due to biofouling of marine installations (Yebrá et al., 2004; Schultz et al., 2010; Aghajani and Esmaeili, 2021). In order to prevent biofouling, industries apply antifouling coatings on exposed surfaces. The antifouling coatings feature in antimicrobial activity and generally prevent both micro- and macrofouling. The majority of antifouling coatings use toxic inorganic (copper) and organic biocides (isothiazolone) that leach out of the coating and kill biofouling organisms (Yebrá et al., 2004). However, toxic biocides affect non-targeted marine organisms as well and accumulate in the marine environment. In 2008, the most potent antifouling agent triorganotin was banned by the International Maritime Organization (IMO) due to its adverse effects on the marine environment

(Maréchal and Hellio, 2009). The non-toxic antifouling methods available in the market are often costly and are not as effective as traditional biocidal solutions. Thus, it is necessary to develop environmentally friendly solutions and nanotechnology-based applications can be of great help in order to do create non-toxic or low-toxic antifouling coatings.

Nanotechnology involves manipulation of materials at the nanometer scale in order to improve and obtain new properties of materials (Hornyak, 2008). Nanostructures with highly controlled and tunable properties can be synthesized by self-assembling of atoms. These nanostructures can either be zero dimensional (nanoparticles) (Grzelczak et al., 2008; Hornyak, 2008), one dimensional (nanowires) (Sugunan et al., 2006), two dimensional (thin films) (Aoki et al., 2005) or three dimensional (arrays, hierarchical structures) (Von Freymann et al., 2010). At nano-scale sizes, materials possess unique size-dependent properties that differ from their bulk, which can be exploited for diverse applications, in electronics (Lah and Zubir, 2018), medicine (Uskoković and Bertassoni, 2010), food (Kumar et al., 2019b), fuel (Chung and Manthiram, 2019), solar cells (Fei et al., 2012), sensors (Zhu et al., 2014), and water treatment (Baruah et al., 2012).

Nanocoatings have been used for biofouling prevention. Previous work reviewed the antifouling effects of nano- and micro-scale surface patterns which are inspired by nature (Scardino and De Nys, 2011; Bixler and Bhushan, 2012; Myan et al., 2013; Graham and Cady, 2014; Richards et al., 2020). The best example of such biomimetic antifouling coating is Sharklet AF™ which was inspired by the microtopography of the shark skin (Beichert et al., 2000). The C18 coatings with high levels of nano-roughness were found to inhibit the settlement of *Ulva* spores and its antifouling effect was shown to exceed that of a commercially available fouling release coating (Majumdar et al., 2008). Superhydrophobic coatings with nano-scaled roughness prevented the settlement of major micro- and macro-fouling species (Scardino et al., 2009). On another hand, engineered nanomaterials have found extensive application in electronics, pharmaceuticals, renewable energies, and antifouling coatings applications, amongst others (Bandala and Berli, 2019). Hybrid nanocomposites of organic-inorganic materials give an opportunity to combine the characteristics of different materials generating opportunities to prevent biofouling (Wassel et al., 2020; Nathanael and Kumar, 2021). Metal and metal oxide nanoparticles, such as silver (Ag), titanium dioxide (TiO₂) and zinc oxide (ZnO), possess antifouling properties (Kim et al., 2003; Chapman et al., 2010; Das et al., 2013; Song et al., 2020). Additionally, carbon nanotubes (CNTs) incorporated in coatings prevent macrofouling by inhibition of settlement and adhesion of larvae (Carl et al., 2012; Kim et al., 2014; Yang et al., 2016a; Kim et al., 2016; Aghajani and Esmaeili, 2021).

Nanostructured metal oxide, like ZnO and TiO₂, is capable of absorbing visible and ultraviolet light and inhibiting microbial growth due to the photocatalytic process leading to redox reactions (Baruah et al., 2012; Danwittayakul et al., 2018; Spirescu et al., 2021). The generated reactive oxygen species (ROS) like peroxides, superoxides, and hydroxyl radicals that form during photocatalysis, prevent the growth of

microorganisms on the surfaces (Sathe et al., 2017; Ganguly et al., 2018). Metal oxide nanostructures can have advantages in antifouling application than other types of nanocoatings due to the fact that ROS are short-lived and have localized surface toxicity (Bora et al., 2017). Antifouling and anti-algal activities of coatings containing ZnO nanorods and TiO₂ nanoparticles have been recently studied (Al-Fori et al., 2014; Sathe et al., 2016b; Yemmireddy and Hung, 2017; Zhu et al., 2018).

This review is aimed at providing an overview of recent advances in formulation and antifouling activity of nanocoatings containing active ingredients of polymers, inorganic nanoparticles, or organic-inorganic hybrid materials.

NANOTECHNOLOGY-BASED ANTIFOULING SOLUTIONS

Various antifouling strategies, namely fouling-resistant, fouling-release and fouling-degrading, have been explored in preparation of antifouling coatings (**Figure 1**) to cater for treatment of different types of biofoulings. For instance, fouling-resistant coatings with a highly hydrated surface provide a physical and free-energy barrier to prevent adhesion of foulants such as proteins, algae, or bacteria (Thérien-Aubin et al., 2011). In contrast, the fouling-release coatings allow weak adhesion of foulant, which can be removed afterwards by external force like water flushing (Damodaran and Murthy, 2016). These two coating approaches introduce hydrophilic and hydrogen bond-forming characteristics to the surface, where the tightly bound water molecules form a barrier layer preventing adsorption (Chen et al., 2010). The third type, fouling-degrading coatings, incorporates antimicrobial moieties in the coating to degrade the settled bacteria or microorganisms via oxidation or other bactericidal functionalities (Sakala and Reches, 2018).

Antifouling Biocides

Antifouling biocides are chemicals that can destroy or render harmless of the microorganisms responsible for biofouling. The previously widely used tri-substituted organostannic compounds, such as tributyltin (TBT) and triphenyltin (TPT), in antifouling paints on ships are no longer permitted for use as biocides within the EU since July 2010 (EU Commission Regulation, 2010), due to the risk of leaching into the aquatic environment and toxicity to aquatic organisms through endocrine disruptive effects. On the other hand, copper compounds are used in antifouling paints for centuries to control hard fouling, such as barnacles, mussels and tube worms, attributed to their effective, available and relatively inexpensive characters compared to other biocides. The most applied copper compounds are cuprous oxide, copper thiocyanate and copper flake, which are used in many different formulations of antifouling paint. Similarly, copper leachate from antifouling paints has been found to impact water quality. Consequently, California's department of pesticide regulation in the US has established a maximum allowable copper leach rate of 9.5 µg/cm²/day for copper-based antifouling products intended for use on recreational vessels, which became effective on July 1, 2018 (California Notice, 2018).

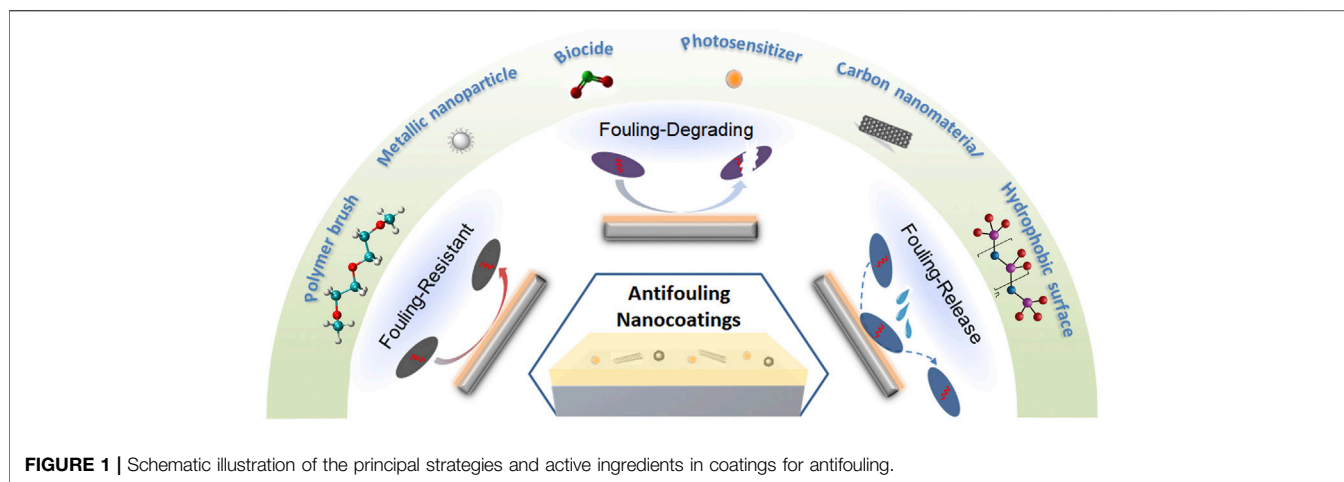


FIGURE 1 | Schematic illustration of the principal strategies and active ingredients in coatings for antifouling.

And the state of Washington (US) has regulated to cease the use of copper-based paint for recreational boats by 2021 due to its toxicity to juvenile salmon. European Commission urges the member states to find and invest in alternatives. Apart from copper, organic booster biocides, such as chlorothalonil, dichlofluanid, chlorine dioxide DCOIT, Diuron, Irgarol 1051, TCMS pyridine, zinc pyrithione and Zineb (Guardiola et al., 2012; Venkatnarayanan et al., 2017), are introduced as alternatives to the restricted organotin compounds in antifouling products. However, some of the booster biocides appear to be persistent and have a continuous leaching character from antifouling paints, potentially posing a significant threat to the aquatic environment with risks of accumulation in aquatic products and development of antibiotic resistance in bacteria. Therefore, eco-friendly and non-biocide-release coatings for marine biofouling prevention are urgently needed.

Polymer-Based Antifouling Coatings

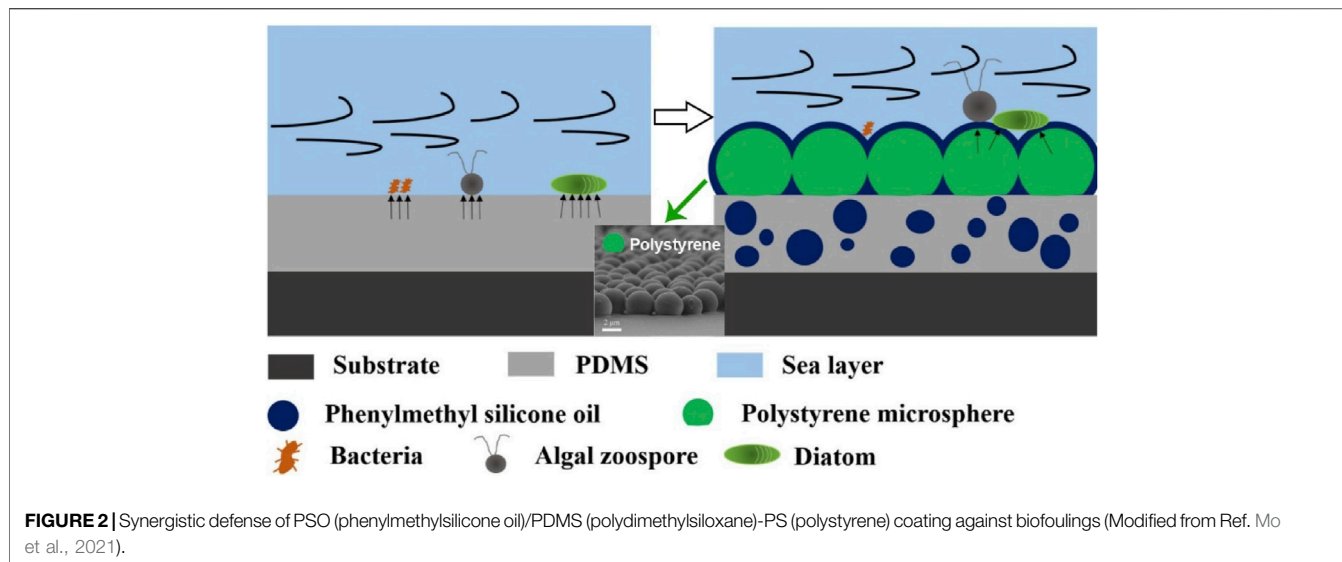
Surface PEGylation, i.e., grafting polyethylene glycol (PEG) to surfaces to develop linear PEG brushes, has long been a standard way to resist the adsorption of proteins (Li et al., 2019). The efficient repulsion of PEG brushes to foulants roots from the extensive hydration layer, the rapid conformational changes and steric repulsion (Hui et al., 2017). However, PEG has been found with short-term stability in biochemical environments due to readily subjected to oxidative degradation and enzymatic cleavage, that results in the formation of aldehyde-terminated chains and a subsequent reaction with amino-contained proteins (Chen et al., 2010). Moreover, PEG coatings used to swell in aqueous environments because of their highly hydrated nature, which deteriorates their mechanical strength, as well the chemically different substrates remain difficult for PEG to graft (Xie et al., 2019). Hence, studies on alternatives for PEG, such as polyoxazolines (Divandari et al., 2017), polyglycerol dendrons (Wyszogrodzka and Haag, 2009), polysaccharides (Rendueles et al., 2013), polypeptoids (Lin et al., 2011), polyacrylamide (Liu et al., 2012) and zwitterionic polymers (Knowles et al., 2017), has been conducted and these alternatives show similar or better fouling resistance. Besides

linear polymer brushes, cyclic- and loop-structured polymer brushes based on poly(2-alkyl-2-oxazoline)s, such as poly(2-methyl-2-oxazoline) (PMOXA) and poly(2-ethyl-2-oxazoline) (PEOXA), have been studied extensively they can generate denser brushes and thus show better protein-resistant properties (Wang et al., 2019). However, it is still costly and complex to obtain the highly dense cyclic polymers brushes in large quantities for large-scale application.

Poly(dimethylsiloxane) (PDMS) coatings represent another type of antifouling mechanism with fouling-release character, attributed to the chemical inertness and low surface energy of PDMS elastomer (Dafforn et al., 2011). In addition, additive of non-reactive silicone oils provides lubricity to the coating surface and therefore considerably reduces fouling by deceiving the mechanosensing ability of fouling organisms, especially for mussels, deterring secretion of adhesive threads, and decreasing the molecular work of adhesion (Amini et al., 2017). To further increase the hydrophobicity of coating surface, a layer of polystyrene (PS) microspheres could be assembled on top of PDMS coating (see **Figure 2**) (Mo et al., 2021). As shown in **Figure 2** (left part), fouling organisms can adhere to the surface of conventional silicone sample without PS microspheres, where it has more attachment points, and the fouling organisms usually show greater bonding strength. In contrast, on the surface of PS-sphere-coated PDMS (diffused with phenylmethylsilicone oil-PSO, right part in **Figure 2**), there are fewer attachment points and weakened bonding strength between fouling organism and coatings. Therefore, benthic diatoms are easily removed under the shearing force of seawater, so as to achieve the purpose of antifouling.

Hydrogel Coatings for Antifouling

Hydrogel is a polymer network which holds a high content of water (typically 80–90%). As aforementioned, many hydrophilic polymers like PEG and zwitterionic polymers can prevent attachment of various proteins, polysaccharides, and many microorganisms. However, hydrophilic polymers typically have difficulty in making surface coating due to the low adhesion strength to various substrates. Out of the approaches developed to



overcome this problem, hydrogel coatings are of especial interest because of their outstanding characters on both antifouling abilities due to superhydrophilicity and fouling-release properties due to the relatively low Young's modulus to destabilize the attachment of marine organisms (Brady and Singer, 2000). To render their practical application in marine antifouling, improvement of the adhesion strength of hydrogels has been investigated through surface covalent cross-linking (Gao et al., 2020) or by decoupling polymerization from crosslinking and interlinking, hydrogel paints were prepared and applied on various substrates by various operations (brush, cast, dip, spin, or spray) (Yao et al., 2019; Yang et al., 2021). The advantages of hydrogel paint lie in the ease of application, like a common paint, without involving mold, UV light, or oxygen-free environment. Secondly, the hydrogels are commonly made by free-radical polymerization and are readily copolymerized with silanes or hydroxyl-contained molecules. While, the substrate materials, such as glasses, metals, ceramics, or organic elastomers and plastics, have hydroxyl groups on their clean surfaces or can be easily acquired through surface plasma treatment, which results in high adhesion strength. Thirdly, dried hydrogel paint can be ground into powders and redissolved in water for application, and the extended shelf life greatly amplifies the advantage of hydrogel paint.

Inorganic Ingredient-Based Coatings for Prevention of Biofouling

Antifouling marine paints containing nanomaterials have been reported to offer super hydrophobicity, high durability, water repellent, anti-sticking, microbial resistance, elastomeric, and anti-corrosive properties (Yebra et al., 2006; Callow and Callow, 2011), which are novel solutions for the sustainable growth of maritime industries. Nanoparticles can be used efficiently in formulations of antifouling coatings resulting in improved properties (Lakhotia et al., 2018). Silver nanoparticles (AgNPs) (Kumar et al., 2017; Kumari et al., 2017), carbon

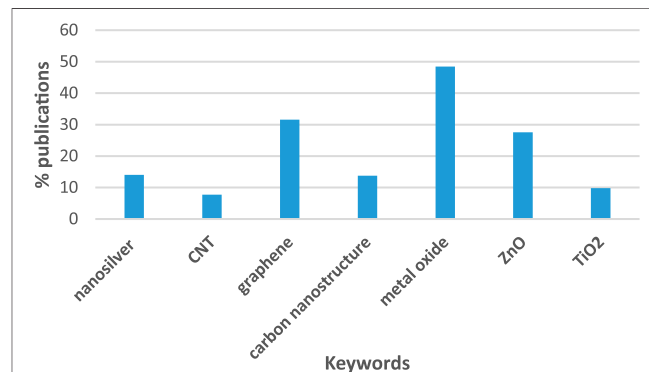
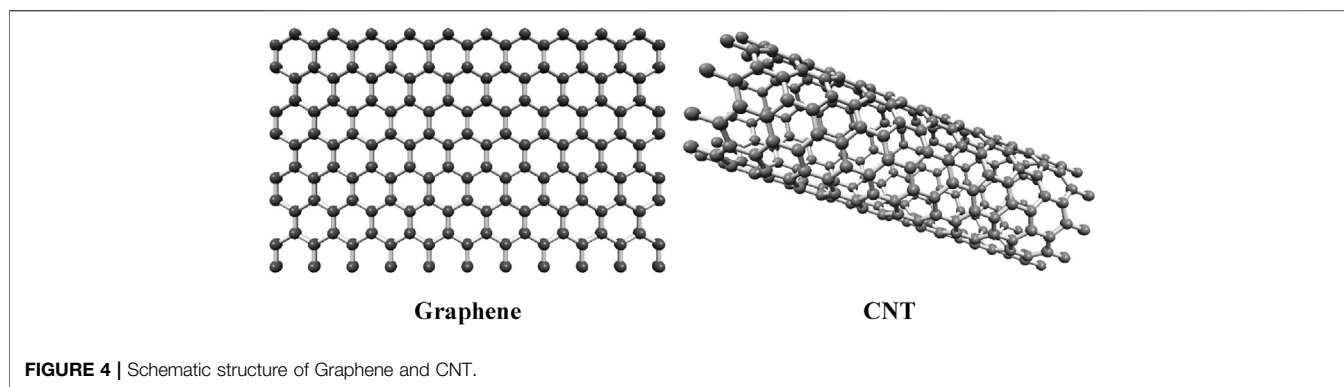


FIGURE 3 | Percent of publications dealing with nanostructure-based antifouling solutions. The results include publications from January 1, 2000 to July 19, 2020 performed by SCOPUS search. The search included key words "nanotechnology" and "biofouling" plus one of the key words.

nanostructures like carbon nanotube (CNT) (Dasgupta et al., 2017) and graphene (Bystrov et al., 2017) are commonly used for antifouling prevention. Additionally, several metal oxides semiconductors like titanium dioxide (TiO₂) (Kim et al., 2016), and zinc oxide (ZnO) (Al-Naamani et al., 2017; Kumar et al., 2019a) nanoparticles have been used for nanocomposite formulations showing antifouling activities. The analysis of literature suggests that most of antifouling studies were dealing with metal oxide nanostructures, such as ZnO and TiO₂ (Figure 3). Less attention is given to carbon nanostructures such as CNT. Below we reviewed the antifouling activity of nanosilver, carbon nanostructures and nanostructured metal oxides as well as composite nanocoatings.

Silver Nanoparticles

Silver-based coatings or silver deposited surfaces are well-known to provide bacteriostatic/bactericidal, fungistatic/fungicidal and



algistatic/algicidal properties to surfaces (Devasconcellos et al., 2012). Silver is known to have inhibitory effects against different kinds of microorganisms (Goswami et al., 2015; Kumari et al., 2016; Naskar et al., 2018). Therefore, it finds applications in medical and consumer products, and in environmental applications (Kumar et al., 2014; Rasheed et al., 2017). Nano-sized silver, i.e., silver nanoparticles, have found increasing application as an antimicrobial agent due to high surface to volume ratio and increased surface reactivity (Kumari et al., 2016). Several studies about the antimicrobial activity of silver nanoparticles and their action mechanism have been reported (Kumar et al., 2014; Durán et al., 2016). Silver formulations containing silver ions or silver nanoparticles are generally considered biologically benign and safer than any other heavy metal-based formulations (Rasheed et al., 2017). The bacteriostatic/bactericidal properties of nanosilver can also be useful in antifouling applications against microfouling (Devasconcellos et al., 2012; Li et al., 2013). Only a few studies investigated the effect of nanosilver on eukaryotes or macrofouling organisms. For example, it has been shown that a silver nanoparticle coating prevents the adhesion of marine and freshwater algae (Ren et al., 2014). In another study, nanosilver prevented mussel settlement through modification of the structure of biofilms (Yang et al., 2016b).

The mechanism of the antifouling properties of silver nanoparticles is not yet clear and debatable. One of the most accepted mechanisms is based on the ability of Ag^+ ions to anchor to cell walls. It is shown that Ag^+ ions penetrate into the bacterial cell and interacts with thiol groups of most of the vital enzymes, leading to deactivation of the enzyme, which ultimately stops bacterial growth leading to the death of the bacterial cell (Kim et al., 2007; Kumar et al., 2014; Pareek et al., 2018). The study with eukaryotes suggested that contact killing of the cells by Ag^+ ions is the main antifouling mechanism (Ren et al., 2014). The formation of free radicals by AgNPs is another proposed mechanism. Electron spin resonance (ESR) spectroscopy studies suggested that free radicals generated from AgNPs damage cell membranes when they come in contact with bacteria, resulting in membrane rupture and ultimately cell death (Danilczuk et al., 2006; Kailasa et al., 2019). However, antimicrobial activity of AgNPs depends on several factors including physico-chemical parameters such as size (Ginjunpalli et al., 2018; Kumar et al., 2019b), morphology

(El-Zahry et al., 2015), crystallinity (Kumar and Münstedt, 2005), surface coatings of nanoparticles (Brobbe et al., 2019), and microbial species in the environment (Pareek et al., 2018).

Graphene and Carbon Nanotube

Graphene is an allotrope of a single layer of carbon atoms arranged in a sp^2 -bonded hexagonal lattice (Figure 4). They are naturally found as the building blocks of graphite. Graphene materials show high specific surface area, electron conductivity and thermal stability that make it attractive for several environmental applications like photocatalysis, energy production and storage (Sun et al., 2015; Ozer et al., 2017; Xu et al., 2018). Carbon nanotubes (CNTs) represent a hollow, concentric cylindrical structure, typically with a diameter of a few nanometers and a length varying from a few nanometers to several microns ($100\ \mu\text{m}$) up to a few millimeters (4 mm) (Venkataraman et al., 2019). Nanotubes are primarily classified into three categories; single-walled carbon nanotubes (SWCNTs), double-wall carbon nanotubes (DWCNTs), and multi-walled carbon nanotubes (MWCNTs). The environmental application of CNTs mainly involves the fabrication of nanocomposite materials with biocidal properties and fouling release activities (Upadhyayula and Gadhamshetty, 2010; Dasgupta et al., 2017).

Carbon Nanotube Graphene

CNTs and graphene exhibit good antimicrobial activity towards both Gram-positive and Gram-negative bacteria as well as bacterial spores (Lukowiak et al., 2016; Al-Jumaili et al., 2017; Sun et al., 2020). CNTs, especially SWCNTs were shown to have significantly greater antibacterial activity than MWNTs, probably due to their smaller sizes, enabling membrane perturbation (Figure 5) (Kang et al., 2008). Additionally, CNTs can impact the recruitment of macro-fouling organisms (Beigbeder et al., 2008). Nano-sized carbon black more effectively prevented settlement of the barnacle *Amphibalanus amphitrite* larvae compare to single-layer graphene oxide (Mesarič et al., 2013). Just 0.5% weight percent of CNT affected the composition of biofilms by increasing the abundance of Proteobacteria and decreasing the abundance of Bacteroidetes, which in turn decreased the settlement of *Mytilus coruscus* (Yang et al., 2016a).

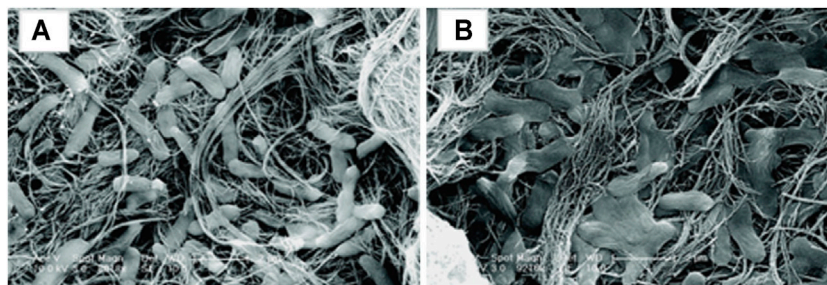


FIGURE 5 | SEM micrographs of *E. coli* exposed to CNTs for 60 min; **(A)** MWCNTs **(B)** SWCNTs. Reproduced with permission from (Kang et al., 2008).

One of the first CNT-based antifouling coatings was developed in 2008 (Beigbeder et al., 2008). It was based on the incorporation of synthetic multi-wall CNTs in silicone coatings. Nowadays, CNT-based coatings are actively used as antifouling and anti-corrosion marine coatings in the construction and oil and gas industries (Dustebek et al., 2016). CNTs are generally loaded into a polymeric matrix for application in protective coatings. Epoxy resins have been mostly used in reinforcement with different types of carbon nanotubes (single-wall, double-wall and multi-wall) (Jin F.-L. et al., 2015; Deng et al., 2015; Shen et al., 2016). CNTs act as an efficient adsorbent due to their large surface area, π -bond electrons on the surface, providing more chemically active sites on the nanotubes, and hollow and layered structures. CNTs have high tensile strength and Young's modulus, and in comparison to steel, CNTs have hundreds and tens of times the higher tensile strength and Young's modulus (Walters et al., 1999). The reinforcement of the paint matrix with CNTs improves mechanical properties in the coatings/paints. Dustebek et al. (Dustebek et al., 2016) investigated the effect of CNTs on the mechanical strength of a self-polishing antifouling resin-based paint and found that the increase in the amount of MWCNT leads to an improvement in the mechanical strength of self-polishing antifouling paints. Antifouling paints with 0.5% (w/w) and 0.7% (w/w) of MWCNTs showed a significant improvement in impact resistance (Dustebek et al., 2016). This study was further supported by another research that evaluated the effect of reinforcement of MWCNT and graphene oxide (GO) on mechanical properties of poly-dimethyl siloxane (PDMS) marine coatings (Cavas et al., 2017). Incorporation of MWCNTs in composites increased both tensile strength and percent of elongation of PDMS marine coatings. These studies indicate that CNT and GO beside of antifouling properties can improve the mechanical properties of nanocomposite coatings.

Numerous mechanisms of antifouling action of CNTs and GO have been proposed, but the actual mechanism is still not clear. A study suggested that different antifouling mechanisms can be involved (Mesarič et al., 2013). First, CNTs and GO are toxic to microbes and larvae of macrofouling organisms. The toxic effect of CNTs is widely known (Francis and Devasena, 2018). CNTs can increase cell apoptosis, oxidative stress, and inflammation. Second, CNTs and GO can inhibit larval

settlement by a non-toxic way simply interfering with the attachment and adhesion of cyprid larvae (Mesarič et al., 2013).

The physical size (in particular the length), diameter, surface area, concentration, treatment time, etc., play a role in the antifouling activity of CNTs. Yang et al. (2010) investigated the effect of length of SWCNTs' on antimicrobial activity to *Salmonella* cells using three different lengths of SWCNTs (<1 μm , 1–5 μm , and ca.5 μm) (Yang et al., 2010). Longer SWCNTs were reported to exhibit stronger antimicrobial activity by aggregating bacterial cells more effectively, whereas shorter SWCNTs were reported to aggregate between themselves without involving many bacterial cells (Figure 6). The tube diameter also plays an important task in the inactivation of microorganisms. Smaller diameters CNTs (<10 nm) can damage cell membranes by interaction with the cell-surface, while large diameter CNTs (15–30 nm) mostly interact by their sidewalls with the bacteria (Upadhyayula and Gadhamshetty, 2010).

Metal Oxides and Metal/Metal Oxide Hybrid Nanostructure

Metal oxide nanoparticles (NPs), such as ZnO, TiO₂, SnO₂, are highly photoactive and used for antimicrobial, self-cleaning, self-healing, anti-corrosion and anti-biofouling applications. Table 1 summarizes the antifouling effects of various photocatalytic metal oxide nanostructures on aquatic prokaryotes (bacteria) and eukaryotes (microalgae, barnacles, bryozoans, etc.). In general, investigators studied the antifouling effect of metal oxide nanoparticles, nanowires, nanorods, and hybrid nanostructures. ZnO, TiO₂ and hybrid metal-metal oxide, as well as hybrid carbon-metal oxides nanostructures were the most commonly used systems for the inhibition of biofouling (Table 1). ZnO nanoparticles have attracted more attention compared to other metal oxides due to their low cost and ready availability (Naskar et al., 2018; Wallenhorst et al., 2018). As per the FDA (Food and Drug Administration, United States) guidelines, ZnO is listed as a safe material for various uses. ZnO NPs are used in the food processing and packaging, as well as in the agriculture sector because of their biocompatibility, low toxicity and antimicrobial properties (Garcia et al., 2018). Photocatalysis of various toxic organic dyes and inorganic pollutants in industrial wastewater has been performed using ZnO and TiO₂ semiconductor oxides under light irradiation. TiO₂ is widely used in self-cleaning

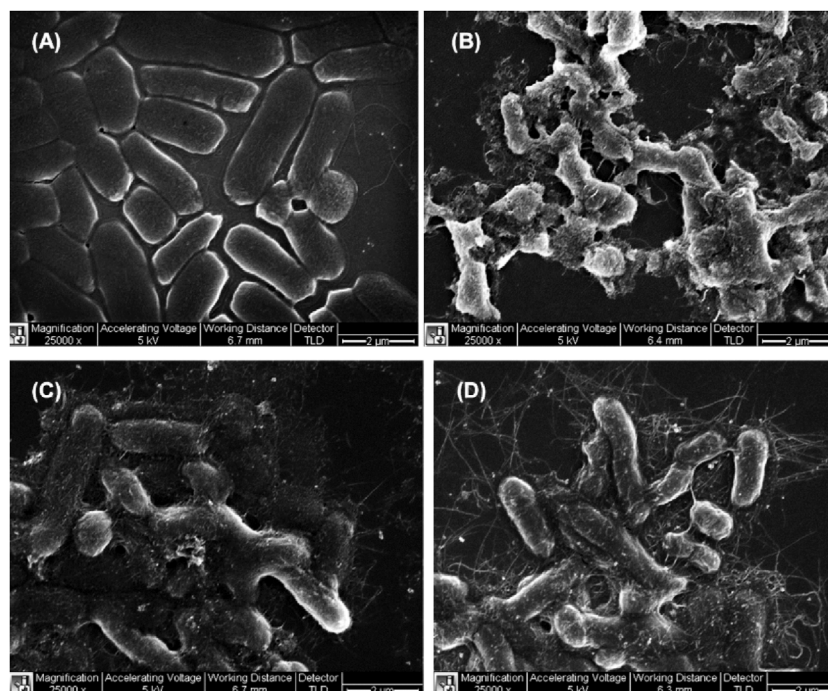


FIGURE 6 | SEM images of *Salmonella* (A) without SWCNTs, and treated with SWCNTs of (B) $< 1 \mu\text{m}$ (C) $1-5 \mu\text{m}$, and (D) $> 5 \mu\text{m}$. Reproduced with permission from (Yang et al., 2010).

coatings and paints but their application is limited to outdoors, as they require activation by UV light (Xu et al., 2017; Adachi et al., 2018). The most common bacteria used for testing include Gram-positive (*B. subtilis*, *Micrococcus*) and Gram-negative (*E. coli*, *P. aeruginosa*) pathogens (Table 1). In opposite, the most common eukaryotes used in metal oxide nanostructures studies are environmental species of microalgae and invertebrate larvae.

Silver nanoparticles is a mostly used antimicrobial agent that synthesized with environment-friendly methods and can be non-toxic for mammals. Titanium oxide nanotubes have been used to improve the functional properties of silver nanoparticles because of their superior specific surface area both on inner and outer surfaces of the tubular structure. Yee et al., 2017 proposed an innovative 2-steps hydrothermal synthesis of a silver–titania nanotube (Ag/TNT) composite that showed nanotubular TiO_2 structures with Ag nanoparticles uniformly dispersed throughout the nanomaterial (Yee et al., 2017). The inhibitory properties of the composite material against biofilm were studied and the result demonstrated the Ag/TNT reduced the biofilm formation of marine bacterium *Halomonas pacifica* by 98% compared to bare titania nanotubes. Additionally, Ag/TNT also showed growth inhibition of marine microalgae *Dunaliella tertiolecta* and *Isochrysis* sp (Yee et al., 2017). A ternary nanocomposite (PDMS/GO- Al_2O_3 NRs) superhydrophobic coatings were also developed from PDMS, and graphene oxide anchored with alumina nanorods (GO- Al_2O_3 NRs) hybrid sheet via solution-casting method (Selim et al., 2018; He et al., 2021). The results showed that the well-dispersed GO- γ - Al_2O_3 NRs hybrid sheets increased the contact angle (151°), decreased the surface free

energy (13.25), and improve micro-nano roughness on the surface, and thus the developed nanocomposites may have promising antifouling applications in the shipping industry (Selim et al., 2018). In a recent study, silicone/graphene-based two novel superhydrophobic nanocomposite were developed, and the results shows PDMS/GO- γ - AlOOH nanorod composite had better antibacterial activity than PDMS/RGO nanocomposite against different bacterial strains might be due high surface area and stabilizing effects of the GO- γ - AlOOH hybrid nanofillers. The GO- γ - AlOOH (3 wt%) nanostructured coating had profound superhydrophobic antifouling properties due to their homogeneity, and high WCA of 151° and a rough surface (Selim et al., 2022).

Aluminum oxides are inexpensive, non-toxic, and one of the most stable inorganic materials. Boehmite is an aluminum oxide hydroxide (γ - AlOOH) particle that contain hydroxyl groups on its surface. Boehmite nanoparticle has the orthorhombic structure, in which the boehmite nanoparticles' surface are covered with OH groups. Boehmite nanoparticles can improve hydrophilicity and surface properties of the membrane due to presence of extra hydroxyl groups on their surface, and thus reduce fouling of the resulted membranes (Vatanpour et al., 2012; Farjami et al., 2021). Among the alumina compounds, boehmite has highest hydrated surface and hydrophilicity. The effects of two different solvents, dimethylacetamide (DMAc) and N-methyl-2-pyrrolidone (NMP) were evaluated on performance of the pristine and EPVC/nano-boehmite nanocomposite membranes, and the results showed that the membranes prepared in NMP solvent using boehmite

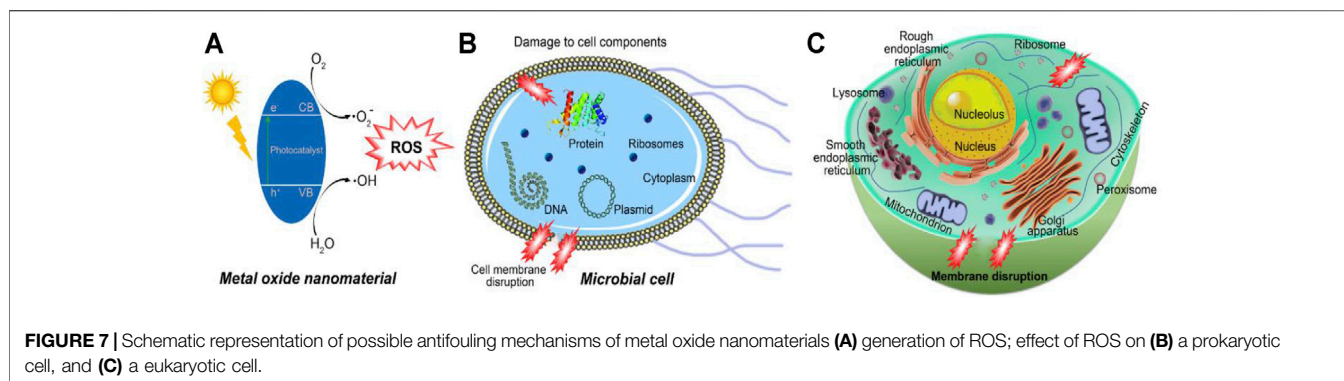
TABLE 1 | Antifouling properties of common metal oxide nanostructures.

Nanostructure	Organisms	Mechanisms	Applications	References
Prokaryotes				
TiO ₂ nanoparticles	<i>Stenotrophomonas maltophilia</i>	Photocatalytic effects	Marine underwater archaeological sites	Ruffolo et al. (2013)
TiO ₂ nanofibers	<i>Micrococcus</i> sp. Gram-positive bacterium, <i>Bacillus</i> sp.	Photo-degradation and inhibition of microbial growth properties of polyacrylonitrile -TiO ₂	Antifouling effects under visible light	Ademola Bode-Aluko et al. (2021)
Ag-TiO ₂ hybrid Ag@TA-SiO ₂ nanoparticles	<i>Halomonas pacifica</i> <i>Escherichia coli</i>	Reduced biofilm attachment by 98% Coating suppressed 98.6% of protein adsorption, and antibacterial efficiency by 99.1 and 82.7% for <i>E. coli</i> and <i>S. aureus</i>	Antimicrofouling effect Antimicrofouling effect	Yee et al. (2017) Deng et al. (2021)
ZnO nanoparticles	<i>S. aureus</i> <i>S. aureus</i> (bacterium)	Photocatalytic and toxic effects	Prevention of microfouling on glass surfaces	Valenzuela et al. (2019)
ZnO nanowires	<i>Bacillus subtilis</i>	Cell membrane modification, and ROS production	Photocatalysis, Antimicrobial agents	Bojarska et al. (2017)
ZnO nanorods	<i>Escherichia coli</i>	ROS production leads to oxidation of membrane lipids causing damage of membrane and cell lysis	Anti-biofouling activity	Al-Hinai et al. (2017)
ZnO nanostructures	<i>Pseudomonas aeruginosa</i>	Generation of ROS increases with increased oxygen vacancies resulting in enhanced antibacterial properties	Enhanced biofouling resistance	Rasmi et al. (2018)
MgO nanoparticles	<i>Escherichia coli</i> <i>Salmonella enterica</i>	Damage cell membrane leading to intracellular contents leakage	Antifouling food surfaces	Jin and He, (2011)
Al ₂ O ₃ nanoparticles	<i>Escherichia coli</i>	Cell wall disruption because of tiny particle size and high surface energy	Antifouling coating having anti-adhesion ability	Dong et al. (2015)
PDMS/GO-Al ₂ O ₃ NR	<i>Pseudomonas aeruginosa</i> <i>Micrococcus</i> sp., <i>Pseudomonas putida</i> , and <i>Aspergillus niger</i>	Formation of micro-nano roughness, lower surface free energy	Superhydrophobic self-cleaning antifouling surface	Selim et al. (2018)
CeO ₂ nanoparticles	<i>Pseudomonas aeruginosa</i> <i>Staphylococcus epidermidis</i>	Cell stress leading to cell death	Anti-infection applications	Alpaslan et al. (2017)
Graphene oxide (GO) nanoparticles	<i>Bacillus</i> sp. <i>Pseudomonas</i> sp.	Significant reduction of biofilm and biomass thickness	Anti-biofouling and anti-corrosion properties	Balakrishnan et al. (2021)
Eukaryotes				
TiO ₂ solution	<i>Chlorella mirabilis</i> <i>Chroococcidiopsis fissurarum</i> (microalga)	Photocatalytic degradation in presence of UV light	Clay bricks	Graziani et al. (2014)
TiO ₂ nanoparticles	<i>Marine biofouling</i>	Riblet surface structure and hydrophobic wettability	Anti-biofouling surface	Li et al. (2020)
ZnO nanorods	<i>Dunaliella salina</i> (microalga)	Formation of ROS	Prevent membrane fouling	Sathe et al. (2016a)
ZnO nanorods	<i>Bugula neritina</i> (bryozoan)	Formation of ROS	Prevent fouling on glass substrata	Al-Fori et al. (2014)
ZnO-SnOx core-shell nanoparticles	<i>Tetraselmis</i> sp. (microalga) <i>Bacillariophyceae</i> spp.	Formation of ROS	Prevent fouling on glass substrata	Kumar et al. (2021)
TiO ₂ and Nb ₂ O ₅ nanoparticles	<i>Navicula</i> spp. (Diatoms) Barnacles, hydroids, oysters	Formation of ROS	Prevent fouling on glass substrata	Dineshram et al. (2009)
Ag-TiO ₂ hybrid	<i>Dunaliella tertiolecta</i> and <i>Isochrysis</i> sp.	Antifouling activity	On fouling microalgae	Yee et al. (2017)
Ag@TA-SiO ₂ nanoparticles	<i>Nitzschia closterium</i> <i>Dicrateria zhanjiangensis</i>	Coating reduced attachment of microalgae <i>N. closterium</i> and <i>D. zhanjiangensis</i> by 93.5 and 97.6%, respectively	Prevent fouling	Deng et al. (2021)

nanoparticles had higher anti-fouling and fouling resistance properties (Farjami et al., 2021).

Antifouling properties and toxicity of metal oxide nanomaterials depend on the biological species, with some species being more sensitive than others (Dobretsov et al., 2020). Additionally, physico-chemical properties of

nanomaterials such as particle size, shape, surface charges, and surface groups affect their toxicity. The smaller size of nanomaterials can easily enter through cell membranes and other barriers of the living organisms. The amount of uptake of nanomaterials decreases with the increased particle size (Hoshyar et al., 2016). The basic principle of metal oxide



based photocatalytic disinfection however involves the generation of highly reactive intermediate species, and these species lead to the photocatalytic destruction of adsorbed entities (organics, micro-organisms, and macro-organisms).

Different surface charges and groups on nanostructures exhibit different surface oxidation reactivity resulting in the generation of ROS at varying levels. The use of TiO_2 or ZnO is limited to UV light irradiation due to their wide bandgap and the high recombination rate of photo-generated charges. Thus, efforts have been focused on achieving high photocatalytic efficiency with these materials especially under visible light. In order to render ZnO , TiO_2 , SnO_2 and other semiconductor materials suitable for visible light photocatalysis, several techniques have been developed, such as surface modification of semiconductor materials, creation of oxygen vacancies, doping of metal/non-metal atoms, depositing noble metals, and coupling with other semiconductors or carbon materials (Rehman et al., 2009; Youssef et al., 2018). Doping with metal or nonmetal makes TiO_2 and ZnO to absorb visible light. Doping metal or non-metal atoms can generate impurity levels in the bandgap of semiconductors altering the photoelectric properties to cover its spectral absorbance in the visible region (Bora et al., 2017; Raizada et al., 2019).

The antifouling mechanisms of nanostructured metal oxides are based on the release of metal ions, such as Ag^+ and Zn^{2+} ions from respective nanomaterials as well as the formation of ROS (Figure 7). Exposure of metal oxides with UV/visible light irradiation leads to the transfer of electrons from the valence band to the conduction band generating electron-hole pairs. The photo-generated exciton if separated can efficiently reduce and/or oxidize a compound adsorbed on their surface by generating $\bullet\text{OH}$ radicals, and/or by the generation of O_2^- anions (Figure 7). Both the radicals and anions can react with pollutants transform them to lesser harmful byproducts or degrade them (Allahverdiyev et al., 2011; Carré et al., 2014; Khan et al., 2015). Similarly, ROS and metal anions prevent fouling by micro- and macro-organisms (Al-Fori et al., 2014). Production of ROS at low dosage levels resulting in harsh oxidative stress that causes the damage of genetic material such as DNA and RNA, oxidation of lipid, alteration of protein, inhibition of enzymes, etc. (Figure 7), while prokaryotic and eukaryotic cell death occurs at higher concentrations of ROS (Pan et al., 2010; Allahverdiyev et al., 2011; Carré et al., 2014). The generated ROS are short-lived and

only affect organisms that come in direct contact with them (Bora et al., 2017).

Metal oxide nanorods and nanowires based photocatalytic coatings have attracted recent attention owing to its ease of controlled fabrication processes (Wei et al., 2005; Inderan et al., 2015; Jin et al., 2015a; Jin et al., 2015b; Wang et al., 2018). ZnO nanorod-based photocatalytic coatings have been found effective against micro- and macro-fouling on a glass substrate and fishing net substrates in the laboratory (Al-Fori et al., 2014; Spireescu et al., 2021) and field experiments, respectively (Sathe et al., 2017) (Figure 8). ZnO nanorods have increased stability, reduced loss of nanoparticles compared to ZnO nanoparticles and can be fabricated into various types of support materials (Al-Fori et al., 2014). Additionally, ZnO nanorods have lower toxicity compared to ZnO nanoparticles, which was confirmed by experiments with bacteria, microalgae, larvae of invertebrates and adult mussels (Falfushynska et al., 2019; Dobretsov et al., 2020).

Inorganic-Polymer Nanocomposite Coatings for Prevention of Marine Biofouling

Apart from purely polymer and organic molecule mixed coatings, organic/inorganic hybrid composites prepared by blending nano-sized inorganic fillers in polymer matrix have been investigated on the fouling-release and fouling-degrading performance. And the nanomaterial-based antifouling coatings have been proved as one of the most promising coatings for biofouling prevention, amongst others (Tobaldi et al., 2017). While metal oxide nanostructures are capable of preventing of micro- and macro-fouling, their effectiveness is low due to their solubility. Additionally, nanoparticles can be lost and cause harmful effects on the environment. In order to enhance stability, reduce environmental impact, and enhance the antifouling effect, metal oxide nanostructures are usually attached to support substrata or incorporated into a stable matrix of coating. For example, ZnO nanorods attached to glass substrata was effective against the marine bacterium *Acinetobacter* sp., the marine microalga *Tetraselmis* sp. and the bryozoan *Bugula neritina* under the artificial sunlight (Al-Fori et al., 2014).

Various nanostructure-based nanocomposite antifouling coatings reported in the literature are summarized in Table 2. ZnO and TiO_2 nanoparticles are most commonly used, while

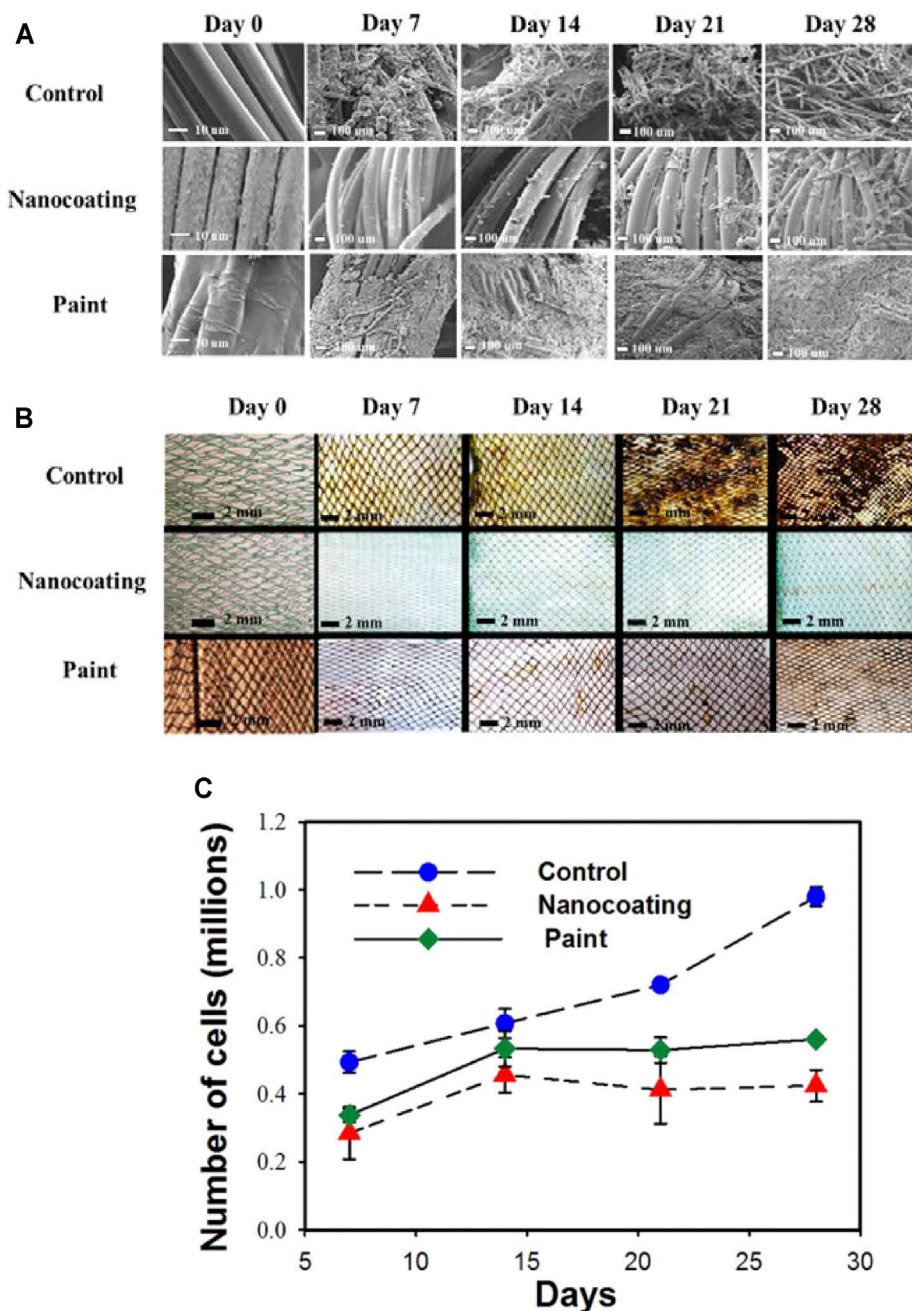


FIGURE 8 | Total microbial profusion and surface coverage of the control, ZnO nanocoating and copper-based antifouling paint coated net substrates **(A)** scanning electron micrographs **(B)** optical micrographs, and **(C)** population count. Reproduced with permission from (Sathe et al., 2017).

Cu_2O , CuO and Al_2O_3 are also incorporated into nanocomposite coatings (Table 2). PDMS has certain advantages as a coating. It has a hydrophobic surface and, thus, used for fouling release antifouling applications (Zhang and Chiao, 2015). Biopolymers, such as chitosan, have been used to develop environmentally friendly nanocomposite antifouling coatings (Carteau et al., 2014; Krishnan et al., 2015; Pounraj et al., 2018). Chitosan and ZnO or TiO_2 based nanocomposite coatings have been reported as effective against several marine microorganisms including

biofilm-forming bacterium *Vibrio fischeri* (Baniamerian et al., 2018), *Pseudoalteromonas nigrifaciens*, and diatoms *Navicula* sp. (Al-Naamani et al., 2017). While metal oxides have photocatalytic properties that prevent biofouling at the light, the addition of chitosan enhances antifouling properties in dark conditions. Most of the studies with ZnO nanoparticles or nanorods nanocomposite antifouling coatings were conducted in the laboratory conditions (Table 2). Since in the field there are many different species of biofouling organisms, as well as

TABLE 2 | Metal oxide nanocomposite coatings for marine antifouling applications.

Nanostructures	Polymer matrix /substrate	Properties	Target organisms	Applications	References
Anti-microbial					
Cu ₂ O nanoparticles	Epoxy coating	Anti-microbial	<i>S. aureus</i> , <i>E. coli</i> , <i>Bacillus</i> sp., <i>K. pneumonia</i> (bacteria)	Cu ₂ O offered reduction of biofilm formation and shielding against damage of surface (i.e., scratch and abrasion)	(M.El Saeed et al., 2016)
ZnO nanoparticles (Polyaniline /zinc oxide nanocomposites)	Polyurethane film	Anti-microbial	<i>Vibrio harveyi</i> (bacterium)	Nanocomposites for the prevention of biofouling	Mooss et al. (2019)
ZnO nanoparticles	Chitosan	Anti-microbial	<i>Bacillus licheniformis</i> (bacteria) <i>Pseudoalteromonas nigrifaciens</i> (bacterium) <i>Navicula</i> sp. (diatom)	Hybrid coatings for control of marine biofouling organisms	Al-Naamani et al. (2017)
ZnO nanoparticles	PDMS	Anti-microbial	<i>Bacillus flexus</i> (bacterium) <i>Navicula</i> sp. (diatom)	Antimicrobial fouling release coating	Gomathi Sankar et al. (2015)
ZnO nanorods	Chitosan	Anti-fouling	<i>Bacillus subtilis</i> , <i>Escherichia coli</i> (bacteria)	Anti-biofilm coating	Al-Belushi et al. (2020)
Ag nanoparticles	PDMS	Anti-microbial	Marine bacteria	Antimicrobial fouling release coating	Yang et al. (2016b)
TiO ₂ nanoparticles	PDMS	Anti-microbial	Marine bacteria	Antimicrobial fouling release coating	Yang et al. (2016a)
TiO ₂ nanoparticles	Chitosan	Anti-microbial	<i>Dunaliella salina</i> (microalga)	Hybrid coatings for control of marine biofouling organisms	Natarajan et al. (2018)
TiO ₂ nanoparticles	Fluorinated acrylic paint	Anti-microbial	Bacteria and diatom	Marine antifouling paint	Zhang et al. (2016)
Al ₂ O ₃ (Alumina) and zeolite nanoparticles	Polysulfone	Anti-microbial	<i>E. coli</i> (bacterium) <i>P. aeruginosa</i> (bacteria)	Anti-adhesion anti-microbial coatings	Dong et al. (2015)
Prevention of micro- and macro-fouling					
ZnO nanoparticles	Alkyd resin	Anti-macrofouling	Marine organisms	Confirmed <i>in situ</i> anti-fouling properties	Yong et al. (2015)
ZnO nanorods	Epoxy/polyaniline	Anti-microbial and anti-fouling	Marine organisms and microbes	Confirmed <i>in situ</i> and <i>ex-situ</i> anti-fouling properties	Mostafaei and Nasirpour, (2013)
ZnO nanorods	Silicone	Anti-microbial and anti-fouling	Marine organisms and microbes	Super hydrophobic fouling release coatings	Selim et al. (2019)
CuO nanoparticles	Poly methyl methacrylate	Anti-microbial and anti-fouling	Marine organisms and microbes	Marine antifouling paint	Sathya et al. (2016)
CuO nanoparticles	Polydimethylsiloxane	Anti-microbial and anti-fouling	Microalgal community	Antifouling to inhibit algal fouling and larval settlement	Ramanujam Padmavathi et al. (2021)
ZnO-SnO _x core-shell nanoparticles	Chitosan	Anti-microbial and anti-fouling	Marine organisms	Chemically resistant antifouling coating	Kumar et al. (2021)

coatings are exposed to different physical and biological factors, it is important to test coatings the field conditions in order to prove their antifouling activity.

CONCLUDING REMARKS AND PERSPECTIVES

Biofouling is a major problem for maritime industries constantly affecting marine installations. Traditional antifouling methods are mainly based on the applications of biocidal agents that detract or kill organisms close to vicinity, which is apparently not environment friendly. This review summarized polymeric and polymer-inorganic composite alternatives to toxic antifouling paints, that show marine antifouling effects via fouling resistant, release, or degrading mechanisms. For polymer and hydrogel antifouling coatings, surface chemistry and coating techniques often determine the

antifouling performance. For inorganic-polymer nanocomposite, the intrinsic characteristics of inorganic nanoparticles is responsible for fouling degradation. In photocatalytic nanostructure-based antifouling applications, ROS generated by photocatalytic nanostructures in presence of light can kill /inactivate micro- and macro-organisms. Compared to toxic biocides, the generated ROS are short-lived and only affect organisms that are in close proximity of the coatings. Thus, photocatalytic nanocoatings are less toxic to aquatic organisms compared to traditional antifouling coatings. Metal oxide nanostructure-based coatings are being considered as one of the promising environment-friendly antifouling technologies strategies that is suitable for marine applications. In contrast, only a few studies that reported inhibition of macro-fouling in the field conditions have been conducted. This could be due to the deteriorated efficiency of nanocomposites antifouling coatings in the field or the difficulties in scaling up such coatings.

An ideal antifouling coating is expected to be durable, reliable, easily applicable, cost-effective, eco-friendly, and substrate-independent. It remains very challenging to realize all the desirable requirements simultaneously. No single chemistry/strategy has yet been identified as the universal antifouling method. Instead, one multifunctional coating with synergetic strengths by combining several antifouling strategies is probably the correct way reaching the goal of a successful antifouling coating. With the large amount of high-quality and sophisticated research that is being performed, zwitterionic polymers attract much attention in the development of next-generation antifouling materials, due to the simplicity of synthesis, ease of applicability, abundance of raw materials and availability of functional groups. At the same time, inorganic nanomaterials possess great advantage in preparation and application for antifouling. Therefore, the inorganic-polymer composite coatings represent another potentially commercial solution, especially considering their effectiveness and low cost. However, it is still necessary to develop new polymers or composite to improve the antifouling performance, especially focusing on long-term durability when used in both static and dynamic environments. Apart from the efficiency enhancement, coating techniques also need to be developed in order to successfully translate these coatings toward large-scale applications to find their way outside the lab. Hence, the frequently used chemical grafting approaches need to be

simplified, in order to cope with the physisorption techniques for coating, often by layer-by-layer or spray-painting. Combining simple coating techniques with a self-replenishing antifouling character to enhance antifouling durability and crosslinkers or catch-bonds to enhance mechanical stability, could result in technologically mature antifouling coatings with large-scale applicability in real maritime environmental conditions and on different materials such as vessels, fishing nets, and pipes. Finally, the impacts of environmental toxicity of such coatings should be more thoroughly investigated.

AUTHOR CONTRIBUTIONS

SK, and JD contributed to conception and design of the study. SD and FY organized the database. SK and FY wrote the first draft of the manuscript. All authors contributed to manuscript revision, read, and approved the submitted version.

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