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Antibacterial Applications of Green Ni-P-W Nanocomposite Coatings

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ABSTRACT

In modern era as increasing demand of healthy and safe consumer products, various green nanoparticles are being created for these through more eco-friendly and cost effective methods by several producers and researchers. In current experimental work, azadirachta indica (neem) (nanoparticle size: 100-140 nm, amount: 1.5 gpl) along with sodium tungstate (Na_2WO_4) (size: 50-130 nm, amount: 0.5 gpl) nanopowders were dispersed randomly into an acidic (pH 5.6) NiP electroless bath and successfully deposited on mild steel (AISI 1040) substrate. The well developed as-coated and heated NiP-W/ green NiP-W nanocoatings were subsequently examined for surface morphology by SEM instrument along with antibacterial activity against gram positive bacillus cereus bacterial strain. The result of these studies revealed that uniform scab of white globular tungsten (W) nanoparticle are found in electroless NiP matrix and that in unheated coatings primarily amorphous structures whereas in heated coatings crystalline structures are exhibited. It is too observed that sole green NiP-W nanocomposite coating demonstrated good resistance against gram positive bacillus cereus bacterial strain.

Keywords: Electroless, coating, characterization, green NiP-W, antibacterial activity.

1. INTRODUCTION

Worldwide, significant efforts are being made to invent and apply biodegradable technologies for manufacture of the consumer goods based on several herbs in order to improve and enhance healthcare solutions [1]. The basic ideas in nanoscience and green nanotechnology are expanding quickly as first the synthesis and characterization and after it employment of green nanoparticles fully or partially into consumer goods [2,3]. The term "green nanotechnology" refers to a branch of green technology that combines green chemistry and green engineering principles moreover full growth of nanoparticles in plants [4]. The green-nanoparticles/nanoparticles have shown a wide range of potential uses in surface coatings due to their high surface area to volume ratio and better reactivity as well as antibacterial activity when compared to their bulk equivalent [5]. The physical/chemical properties along with various experimental results of synthesized nanoparticles are most significantly influenced by biological species properties, as well as by reactions variables like temperature, pH and reactant concentrations [6]. These days, nanoparticles have been applied to some base materials (MS, Al, Mg, *etc*.) either as coatings or as reinforcements to create nanostructures *in situ* conditions and have a variety of uses, including coatings that are self- cleaning, tribologically resistant, anticorrosive, and UV-protective, *etc*. [7-9]. Moreover, the green nanomaterials in recent years are prepared by variety of methods, including plants (such as tulsi, neem, turmeric, *etc*.), vegetables (such as onion: allium cepa, lemon grass, *etc*.), and even microorganisms like bacteria, algae, fungi, as well as even viruses and have had successes and industrial applications [10-18]. Additionally, various natural green ingredients that are selectively added to conventional coatings can be regarded as green nanomaterials [19]. The co-deposition of second phase hard and soft nanoparticles (ZrO_2, SiC, SiO_2, TiO_2, ZnO, Al_2O_3, Si_3N_4, MoS_2, WS_2, PTFE, BN (h), CNF, *etc*.) into EL Ni-P matrix has been explored for its potential in triobology and corrosion resistance properties [20-36]. Considering the green nanomaterials, in our prior research work electroless alkaline green NiP-ZrO_2 nanocomposite coatings (NiP-ZrO_2 + turmeric nanopowder) have been successfully coated on mild steel substrate and further tested for antibacterial activity. As among above soft and hard nanoparticles, tungsten is a very hard and corrosion resistant material moreover each component of the neem (*a. indica*) plant, including leaves, roots, stems, flowers, seeds, bark, fruits, has prevailing pharmacological effects

including anti-fungal, antiviral, antibacterial applications. Therefore in yet another our study, the electroless alkaline green NiP-W nanocomposite coatings (electroless NiP-W + *Azadirachta indica* nanopowder) have been coated on mild steel substrate and further tested for antibacterial activity against a bacterium named *Bacillus cereus* (Figure 1). Further the as-coated coupons were heated at 400 ºC in a pure argon environment for one hour duration to know the heating effects on coated coupons.

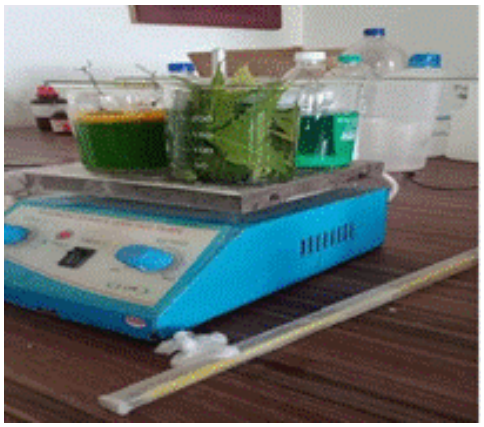

Fig. 1: Photograph of neem (*Azadirachta indica*) leaves and green NiP-W coating process

2. EXPERIMENT

The 20 mm × 20 mm × 04 mm mild steel coupons (AISI rating 1040 rating; iron balance, carbon 0.18%, silicone 0.041% and manganese 1.66%) were polished using SiC emery paper with a grit range of 50 to 1200 to eliminate corrosion products and foreign objects. After polishing, coupons were degreased with acetone and then sensitized as well as activated with $SnCl_2$ and $PdCl_2$ solutions respectively [20-24]. During the deposition process, sodium tungstate nanoparticles (amount 0.5 gpl, 50-130 nm size) and neem nanopowder (amount 1.5 gpl, 100-140 nm size) were separately added to electroless Ni-P matrix. A tiny Teflon-coated magnet was used to stir the mixture continuously. The all electroless bath parameters are depicted into Table-1. Once the deposition process was completed; as-coated coupons were wash down through deionized water and then dried at room temperature.

Table-1: Chemical compositions and established electroless bath parameters

S.No.	Chemicals (Source)	Amount/conditions	Function
1	$NiSO_4 \cdot 6H_2O$ (Sigma-Aldrich)	04.19 g	As a source of Ni^{2+} ions
2	$C_6H_5Na_3O_7 \cdot 2H_2O$ (Merck)	04.70 g	Complexing agent to prevent the releaseof Ni^{2+} ions without control
3	Sodium acetate (Merck)	02.56 g each	buffer that is acidic tomaintain a pH of 5.6
4	$NaOH/CH_3COOH$ 5% solution	10 mL added drop wise	To maintain the solution pH at 5.6
5	$NaH_2PO_2 \cdot H_2O$ (Loba Chemicals)	02.50 g	Accelerate to provide more Ni^{2+} ions
6	Na_2WO_4 nanoparticles (50-130 nm)	00.05 g	reinforcement in the electroless NiP matrix
7	Neem nanopowder (100-140nm range)	00.15 g	Acted as antibacterial agent
8	Operating parameters for bath	83-85 ºC temperature range with continuous stirring	–
9	Annealing temperatures	400 ºC for 1 h in 99% argon environment	To understand impact of heat performance

The as-coated coupons were too heated for 1 h duration at 400 ºC in a tube heating system with a 99% pure Argon gas environment before being annealed to room temperature. It was too observed that the plating thickness (micrometer) was comparable to the weight increase in gram [13,20,24,28-32]. The morphology of heated and unheated coated coupons (NiP-W and green NiP-W) was further examined using the Zeiss Evo18-SEM instrument (figure 2). For a bacteria culture named as *Bacillus cereus*, the antibacterial properties of acidic electroless NiP-W/green NiP-W coatings are too examined. For this, all necessary tools were required, including nutrient broth, Mueller-Hinton agar (MHA), sterile swabs, weighing scales, tetracycline antibiotic discs, sterile discs, 15% DMSO, and Erlenmeyer flask the analytical-grade chemicals utilized and were all obtained from Hi-media, Merck, and Sigma.

3. RESULTS & DISCUSSION

In figure 2, scanning electron microscope photograph of well-developed as-coated and heated NiP/NiP-W/green NiP-W coatings are shown. The SEM microstructures of NiP-W/green NiP-W nanocomposite coatings differ significantly from that of NiP coating. The W nanoparticles in the NiP-W nanocomposite coating are finely and uniformly disseminated throughout the NiP matrix, in contrast to the NiP coating, which has a cauliflower-like structure. The constructed composite layer has a usual structure and small changes in the topography of unheated and heated coatings are seen for green NiP-W coatings. The bulges into heated coatings are developed and give the impression of coming together at 400 °C. Moreover for application point of view of developed coatings, four stages of growth for bacteria in batch cultures are lag, exponential, stationary, and death. The lag phase is the amount of time it takes for bacteria to develop the physiological conditions that allow them to multiply and divide rapidly. When DNA replication, RNA transcription, and protein synthesis all happen at once, a cell grows and divides exponentially. Bacterial growth slows down during the stationary phase, and a severe nutritional deficiency during the dying phase results in cell lysis. Depending on the antibacterial potency of the chemical, the addition of an antibacterial agent to a culture can interrupt any stages, leading to either a complete growth inhibition or a decrease in the cellular density of the culture by passing a photodiode sensor through a one millimeter thick layer of bacteria. The number of bacterial cells that can be counted using a spectrophotometer is readily determined using the optical density. It is possible to estimate the bacterial growth hindrance of a batch culture with growth-halting substances like antibiotics or other antibacterial compounds by constructing a standard curve of a bacterial culture with known cell concentration in it at various points over the course of a 24-hour period. The optical density (OD) at a wavelength of 600 nm is measured to accomplish this. A calibration curve can be made by comparing the measured OD_{600} to the predicted OD_{600} for a range of concentrations. A different method is used to calculate expected OD_{600}, which is then converted to OD_{600} using the rule-of-thumb one OD_{600} = 5108 cells/ml for *E. coli* bacteria. In the current investigation, we examined the bacterial toxicity of NiP-W/green NiP-W nanocomposite coatings for bacterial strain *Bacillus cereus* (Gram-positive bacteria). In a flask, 3.32g of nutrient broth (250 ml), a multipurpose growing medium, was dissolved in 250 ml of distilled water and after it media was autoclaved at 120°C for 35 minutes to sanities it. This (15.03 ml) medium was

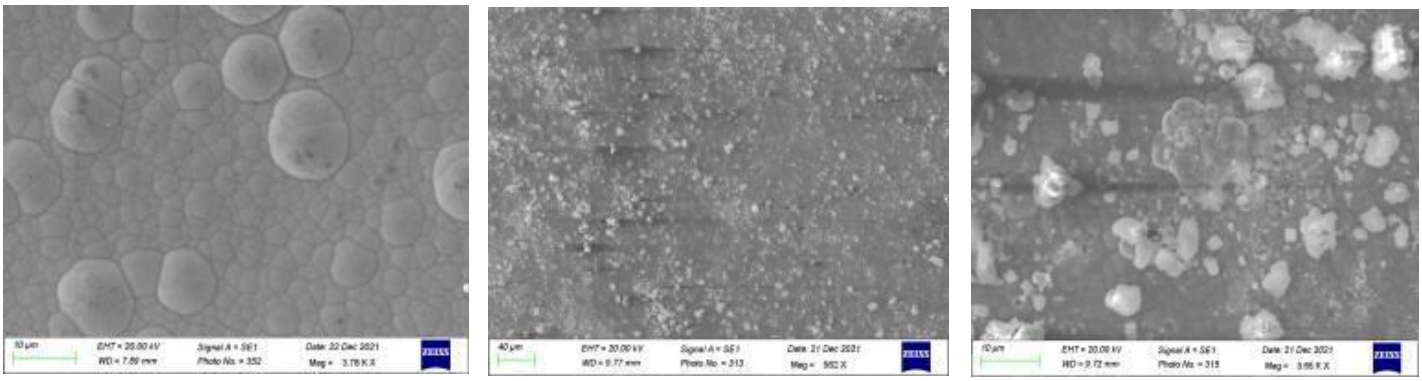

Fig. 2: SEM images of (a) Ni-P coating (b) heated NiP-W coating (c) heated green NiP-W coating

medium was then sterilized in test tubes and kept at 40 °C until needed using aseptic procedures. In a conical flask, 9.04 g of M-H agar was dissolved in 250ml of distilled water, brought to a boil to completely dissolve it, then autoclaved for 30 min at 120 °C to form an antibiotic susceptibility testing medium. It was autoclaved, let to cool to ambient temperature (40°C) and then packaged. Prior to usage, 25 ml of agar material was added to 100 × 15 mm circular petri plates, which were kept at one test tube containing 15 ml of nutrient broth underwent aseptic inoculation with frozen stock (1ml) of culture after being brought to room temperature. After inoculation, a test tube was held at 35 °C for 24 hours. After 1 ml of bacterial culture was removed and put into a glass test tube containing 5 ml of PBS, the number of colony-forming units (CFU) per milliliter (CFU/ml) was calculated using serial dilutions. The number of bacterial colonies on each plate was counted after incubation, after visible colonies emerged, and these data were recorded together with their corresponding dilution factor for all plates at each time point. All freshly coated mild steel coupons were evaluated for their antibacterial activity using the disc diffusion experiment [33–36]. Pipetting out 0.5 ml of each bacterial culture, Muller-Hinton agar plates were used to aseptically swab culture the bacteria. Tetracycline, an antibiotic, served as a positive control. On the inoculated agar plate, coated mild steel sample, positive and negative controls, and were fixed. After a 24-hour incubation period at 350 ºC, the MHA plates were examined for zones of inhibition. *B. cereus* bacterial strains were cultivated overnight, and CFU/ml was used to compute their CFU/ml. By adding 1 ml of culture to a cuvette, the OD_{600} of the culture was measured and compared to a blank culture. The *B. cereus* have CFU/ml 1.1×10^4 and OD after 24 hours (against blank media 0.0393± 0.00316) 0.3914± 0.0042. All the tubes were incubated at 35 ºC and at regular intervals of time their optical density *via* absorbance measurement was recorded. The OD of nutrient agar tubes *viz.* uninoculated (A), inoculated without sample (B), inoculated with sample as-coated NiP-W (C), inoculated with as coated green NiP-W (D), inoculated with sample heated green NiP-W (E) was taken at the intervals of 5, 10, 15, 24 hours. A culture's optical density was assessed in comparison to a blank medium (A) and values of OD rises as bacteria grows and density rises. All results are graphically expressed as inoculated without sample (B) > inoculated with sample as-coated NiP-W (C) > inoculated with sample heated green NiP-W (E) > inoculated with as coated green NiP-W (D) > uninoculated (A). To justify the above results of optical density variation, of sample D showing maximum toxicity was further tested via disc diffusion assay. Freshly coated mild steel sample was placed with the positive controls on *B. cereus* swab cultured Muller-Hinton agar, after 24 hours zone of inhibition was again looked and for inoculated with sample as coated green NiP-W (D) showed significant zone of inhibition, thus justifying the above results of toxicity against *Bacillus cereus* (Fig. 3 and 4).

(a) (b) (c) (d)

Fig 3. : Toxicity of heated NiP-W and green NiP-W coating against *B. cereus* on MH agar (a) sampling (b) results (c) NiP-W inhibition zone (d) green NiP-W inhibition zone

4. Conclusions

A brief discussion about an eco-friendly coating namely green NiP-W nanocomposite coating along with its antibacterial functionality against a bacterial strain is discussed. The study suggests that green coatings may be a cost-effective and eco-friendly as compared to chemical and physical methods that require toxic chemicals and high energy inputs for environmental sustainability. The results by surface instrumental study explicated that

surface morphology of green NiP-W nanocomposite coating is totally different from that of NiP/W nanocomposite coating. Overall the as-coated depositions mostly have mostly amorphous structures while that heated coating revealed crystalline structure. Further in all prepared coatings, it is found that only green NiP-W nanocomposite coating revealed good antibacterial resistance against a Gram-positive culture namely *B. cereus*.

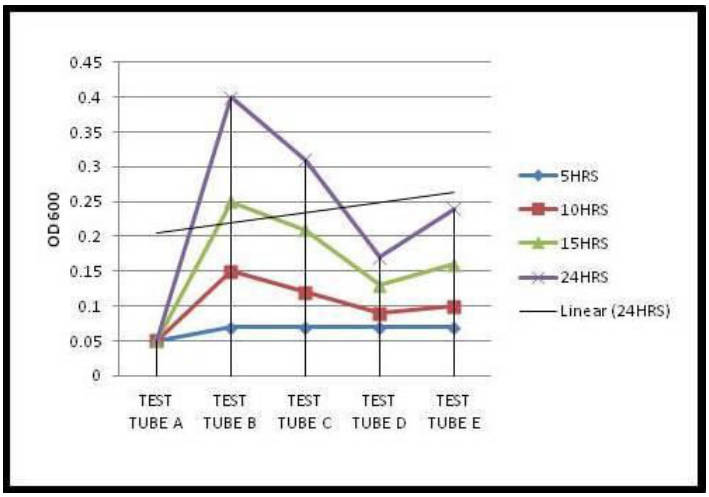

Fig.4: Variation in optical density of *Bacillus cereus* culture with and without samples

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