



Enhanced electrochemical and mechanical performance of BN reinforced Ni-P based nanocomposite coatings

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ARTICLE INFO

Keywords:

Nanocomposite coatings
Electrodeposition
Reinforcement
Hardness
Corrosion

ABSTRACT

Adequate corrosion protection and improved mechanical properties are necessary requirements to overcome operational costs in the industries. Ni-P based coatings are known to possess better superior corrosion resistance than its counterparts. Boron Nitride nanoparticles (BNNPs) are well known refractory material with higher hardness and chemically inert nature suitable for reinforcement. In the current investigation, the effect of incorporation and increasing concentration of BNNPs in Ni-P matrix is thoroughly investigated in terms of mechanical and electrochemical performances. Successful incorporation of BNNPs within the Ni-P matrix was achieved by employing tailored Watts bath and optimized deposition parameters. The addition and increment of BNNPs reveal a significant impact on the characteristics of pure Ni-P coatings. Improvement in the structural, morphological, mechanical and corrosion behavior of BNNPs reinforced nanocomposite coatings can be ascribed to uniform incorporation of BNNPs in the deposit leading to the dispersion hardening effect that enhances strength to the coating improving surface hardness up to 58 % in comparison to pure Ni-P coating. Moreover, reduction in the active area caused by inert BNNPs leads to the improvement in corrosion resistance properties with protection efficiency (PE%) reaching up to 95 % for Ni-P-1.5 g/L BN nanocomposite coating in comparison to the bare mild steel substrate. BNNPs reinforced Ni-P based nanocomposite coatings provide a possible choice for their application in many industries like in the aerospace, automotive, marine, oil and gas industry.

1. Introduction

Metallic components are extensively utilized in various industries. Gradually, these assets degrade or depreciate affecting the efficiency of plant and increasing the operational cost. Corrosion is the gradual depreciation of metallic material which becomes more lethal with heavy operation and corrosive environment. Corroded surfaces and part are also prone to sudden failure which can cause loss of human life and breakdown of entire facility. As the corrosion is spontaneous process where the material traces back to its raw state in the form of complex oxides and carbides, hence, it is not possible to completely eliminate corrosion. Among many corrosion mitigation techniques adopted across the globe, surface modification seems to be most efficient as it safeguards the bulk material without compromising its inherent and useful properties. Metallic coatings are among the most suitable technique utilized for surface modification for steel structures as it improves the

corrosion resistance of the component along with the improved mechanical characteristics. Several of its advantages include tailored properties depending upon the application, more economical and easier synthesis route. Coatings can increase the value of products up to ten times the cost of the coating [1].

Inorganic coatings are popular these days, due to their excellent characteristics. They offer good mechanical strength, high corrosion protection, simpler process and are cost effective. Nickel based coatings started to gain attention because of the significant properties of Ni such as good mechanical properties and non-hazardous nature to replace the carcinogenic chromium coating [2]. Presently, nickel-based coatings such as the Ni-B have been reported to show good wear resistance [3]. Ni-Co are known to have adequate magnetic properties [4], Ni-W are found to be advantageous in optical applications [5], whereas Ni-P exhibit high corrosion resistance but inferior wear resistance [6]. These types of coatings have gained importance and are employed

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extensively. Alloy coatings are preferred over the pure metal, to obtain different valuable properties which are superior to the ones for pure metal [7]. The nickel-phosphorus alloy produces enhancement in mechanical properties, wear and corrosion resistance, magnetic behavior, fatigue limit and lowers the macroscopic deformation. To bring a further improvement, hard or soft nanoparticles, or polymers are included in the alloy matrix. In the past, many types of ceramic materials have been employed, that include, B₄C [8], WC [9], TiC [10], TiO₂ [11], TiN [12], Y₂O₃ [13], SiC [14], CeO₂ [15], SiO₂ [16] and Al₂O₃ [17], for example. These nanoparticles are reinforced into the metallic coating to enhance, corrosion protection, wear resistance, and the structure. The benefit by the dispersed nanoparticles in the metal matrix is dependent on the degree of dispersion, their content, material and shape [18].

Various fabrication techniques have been explored by the research community for the preparation of composite coating such as electroless deposition, electrodeposition, pulse electrodeposition, plasma spraying, chemical vapor deposition (CVD) etc. Direct Current electrodeposition is emerging as one of the most easier route for coating owing to its electrodeposition is considered an effective and promising procedure as it is reliable for large scale, cost efficient, demands less space, low temperatures and very less machinery. It is vastly applied for metal matrix composites as it is fast, reproducible, less expensive and generates dense structures.

This work is based on the fabrication of Ni-P metallic coatings reinforced with different concentrations of Boron Nitride through electrodeposition process. Formerly, Ni-P-BN nanocomposite coating fabricated through electroless process have been studied which included the effect of heat treatment on electrolessly deposited coatings [19]. Li et al. [20] electrodeposited BN incorporated Ni-W alloy coatings and concluded an improvement in corrosion resistance, hardness and wear resistance with increased concentration of BN. Similarly, Sangeetha & Kalaigan [21] carried out research on inclusion of hexagonal BN to fabricate Ni-W/BN nanocomposite coatings through direct current and pulse current electrodeposition. The results revealed that the incorporation of BN did not have any effect on the Ni-W morphology, rather enhanced the mechanical and corrosion resistant properties of coatings. Additionally, Awasthi et al. [22] deposited boron nitride in Ni based coating by electrophoretic deposition process, on copper plate, which were further modified using graphene, diamond and carbon nanotubes. Employing Boron Nitride with a particle size of 0.5 μm, Ni-BN composite coatings were developed by Pompei et al. [23] using electrodeposition on brass foils and reported an improvement in microhardness and wear resistance. Furthermore, Paydar et al. [24] aimed to improve the Ni matrix by incorporating boron nitride (0.5 μm) combined with boron carbide (1 μm), developing electrodeposited coatings on copper substrate, in order to study the morphology and hardness of the coating, while considering some of the major factors, such as current density, temperature, pH and agitation speed. Farrokhzad [25] reported an improvement in high temperature oxidation resistance of Ni-P-BN nanocomposite coatings that were electrodeposited on carbon steel. Moreover, using High Velocity Air Fuel (HVOF) spraying process, Mathiyalagan et al. [26] deposited Ni-P-(cubic)BN on mild steel substrate and reported promising wear properties. Zhao et al. [27] developed Ni60 + Cu/BN + MoO₃ composite coatings with two different forms of BN, cubic and hexagonal structured where plain carbon structural steel was the substrate through laser cladding methodology and reported improvement in mechanical properties. Secondly, applying intense procedures, such as laser cladding and HVOF is very costly, as they require intricate equipment for setup and operation [28]. In addition to that, electroless process is lengthy and time consuming. Even with no application of electricity, its operating cost is higher than that of electrodeposition [29]. Instead, electrodeposition was applied as it is a quick process, carried out at low temperatures [30]. According to our best literature review, this is the first study on the incorporation of Boron Nitride NPs in Ni-P metallic coatings, by the electrodeposition process, and there has not been a thorough study carried out on the

corrosion resistance and mechanical properties of this particular type of coating. Furthermore, in this research, Boron Nitride nanoparticles were chosen as a reinforcement owing to its promising inert nature. The BNNP possesses high hydrophobicity and impermeability, as well as good thermal conductivity and electrical insulation, with great mechanical properties [31]. It is chemically inert and thermally stable with high melting point of (2973 °C) [25]. Boron Nitride is considered a soft material due to its low coefficient of friction. Thus, in research it has grown popular as a solid lubricant and also an applicant for dry machining purpose [32]. This study primarily focused on the incorporation of BN to Ni-P, that resulted to be very effective. The aim was to analyze the effect of increasing concentration of BN, as 0.5 g/L, 1.0 g/L and 1.5 g/L, in the Ni-P matrix on the morphology, corrosion resistance and mechanical properties examined by the help of different characterization procedures.

2. Experimental

2.1. Materials

The Ni-P-BN coatings were developed using electrodeposition on carbon steel substrates. The electrolyte bath comprised of nickel sulfate hexahydrate, nickel chloride hexahydrate, boric acid, phosphoric acid, sodium hypophosphite and sodium chloride, and all these chemicals were bought from Sigma Aldrich. The composition of the chemical bath is listed in Table 1.

Boron Nitride nanoparticles with an average particle size of 70–80 nm were introduced as nanoparticle reinforcement. The nanopowder was provided by US Research Nanomaterials, Inc., with composition of 99.8 % BN. The steel substrate used carbon steel and the substrate had dimensions as 30 × 30 × 2 mm³.

2.2. Deposition procedure

The carbon steel substrate was polished sequentially on SiC grit papers increasing in size from 80 to 1200 (80, 120, 220, 320, 500, 800, 1000 and 1200). The polished samples were then cleansed by sonicating in acetone for 15 min to get rid of any loose sand particles that may be attached to the substrate surface. Then the sample was washed with warm water and the back side (the side that is not supposed to be coated) was covered with masking tape. The sample was then dipped in 20 % H₂SO₄ solution for 30s for acid prickling in order to activate the surface of the sample to be coated. The substrate was then again washed with warm water and then dipped directly into the electrolyte bath. Table 1 mentions the conditions or parameters used for the electrolyte bath. Fig. 1 shows the schematic of the electrodeposition process. The bath was agitated continuously at 300–400 rpm. The temperature of the bath was kept at 70 °C and the current density applied was 50 mA/cm². The

Table 1

Optimized composition of bath and the applied parameters for electrodeposition of coatings.

Bath components	Composition (per 1 L)
Nickel sulfate hexahydrate	250 g
Nickel chloride hexahydrate	15 g
Boric acid	30 g
Phosphoric acid	6 g
Sodium hypophosphite	20 g
Sodium chloride	15 g
Operating conditions	
Temperature	70 °C
pH	2.5
Current density	50 mA/cm ²
Bath agitation	300–400 rpm
Deposition time	30 min

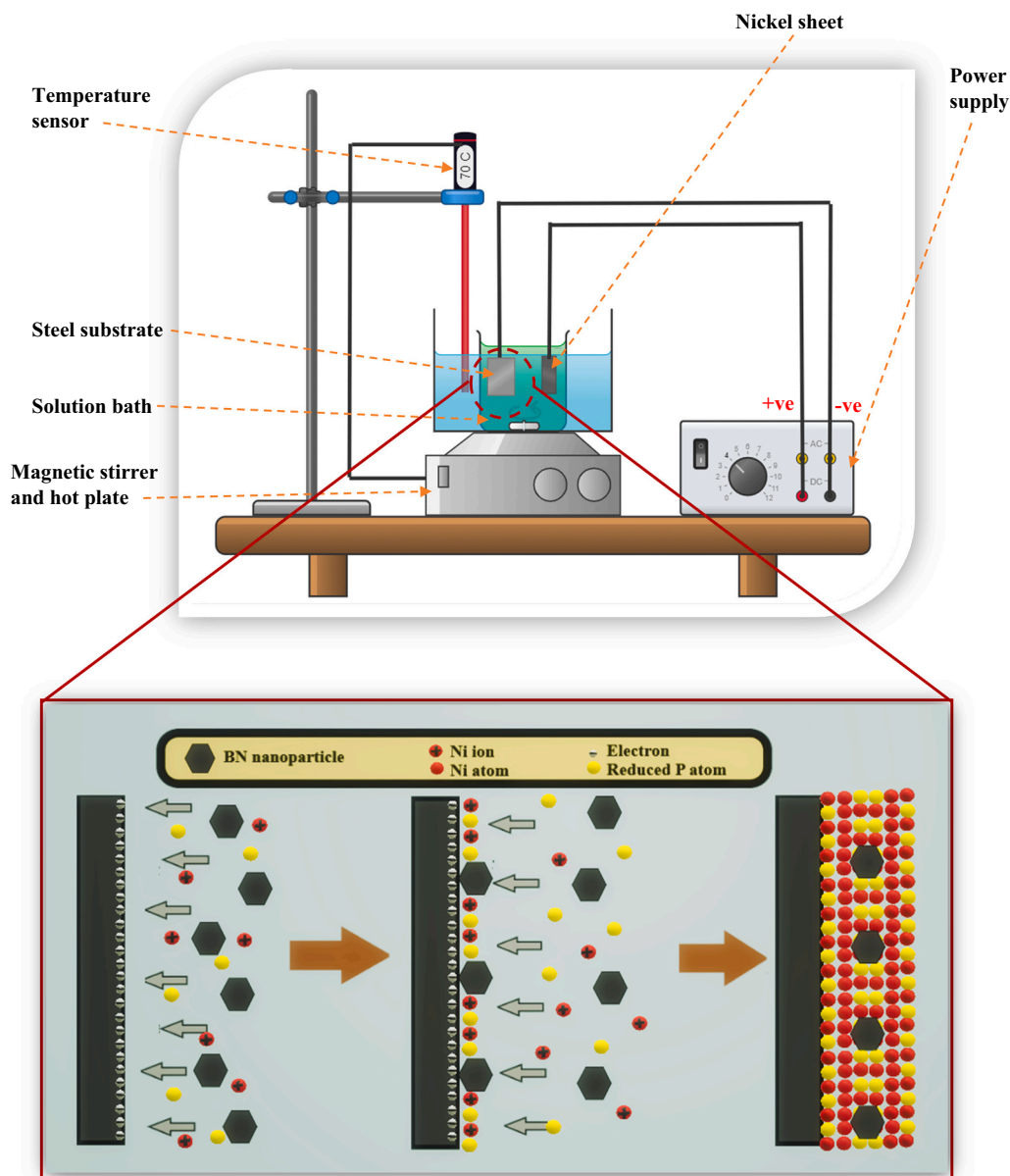


Fig. 1. Schematic diagram of the electrodeposition process for fabricating Ni-P-BN nanocomposite coatings.

pH recorded was 2. The sample was electrodeposited for 30 mins and then removed, washed with warm water, and let to air dry. The process was repeated for each BN concentration, and then the resulting samples were labelled accordingly.

2.3. Characterization

The structural analysis was performed using the X-ray diffractometer, from PAN Analytical-Empyrean, Netherlands. Using Cu $K\alpha$ radiations, it recorded X-rays diffractions at a scan rate of $0.02^\circ/\text{second}$ in the 2θ range from 20° to 100° . The chemical composition of pure Ni-P and Ni-P-BN electrodeposited coatings was determined using X-ray photoelectron spectroscopy from AXIS Ultra DLD, Kratos, UK. The monochromatic X-Ray with Al $K\alpha$ source. The binding energy of the reference was C 1s (284.6 eV). The process was carried out at (160 eV) passing power, and the high-resolution spectrum was evaluated at (20 eV) passing energy. Field Emission Scanning Electron Microscope, FESEM from Nova, Nano-450, Netherland, was employed to explore the morphological characteristics of the electrodeposited Ni-P-BN

nanocomposite coatings, along with that the EDX (energy-dispersive X-ray spectroscopy) results were delivered to identify the surface attributes of the prepared coatings. To measure the thickness of the fabricated coatings, a gauge meter, PosiTector 6000 from DeFelsko, Proctor Avenue Ogdensburg, NY, USA, was used. Atomic Force Microscopy, AFM using the Asylum Research, Oxford Instruments, USA, was employed to examine the surface profile of the electrodeposited pure Ni-P and Ni-P-BN nanocomposite coatings. Moreover, the mechanical properties included microhardness, which was measured using the Vickers hardness testing machine (FM-ARS9000, USA) from FUTURE-TECH CORP. Kawasaki, Japan. A 25gf of testing load was applied and the indenter was in contact for 10s. The resulting hardness values obtained are an average of 7 readings from different locations. The nano-indentation values were measured at a load of 1mN by the Nano indenter (MFP-3D) attached to the AFM. The result obtained in form of load versus displacement curves were used to find the electrochemical parameters. Erosion analysis was carried out by utilizing an air-jet erosion rig set up. Erodent particles employed for testing were alumina of 53–84 μm particle size with the methodology adopted as per

ASTM G76 [33]. Erodent feed rate of 0.94 g min^{-1} impinged the coated samples mounted on a sample holder facing the nozzle with varying incident angle from 30 to 90 with fixed erosion time of 120 s and constant erodent velocity of 2 kg/cm^2 . The working distance between the nozzle outlet and the test specimen is 10 mm. The material removal depth and crater dimensions were measured by 3D optical surface metrology system Leica DCM8 profilometer. Corrosion resistance testing was carried out in a brine solution of 3.5 wt% NaCl at 25°C using Gamry 600 potentiostat/galvanostat/ZRA, from Warminster, PA, USA. A three-electrode system was applied, that included a working electrode (electrodeposited coating), reference electrode (Ag/AgCl), and counter electrode (graphite). The coated substrate was monitored in open circuit potential, OCP, for 30 min before beginning the electrochemical measurements. An AC perturbation of 10 mV amplitude was employed within the frequency range of 10^5 to 10^{-2} Hz to record the response. Additionally, the potentiodynamic polarization testings were executed within -250 mV to $+250 \text{ mV}$ from OCP at a scan rate of 0.167 mV s^{-1} .

3. Results and discussion

3.1. Structural and compositional analysis

X-ray Diffraction technique explores the phases and structures of the developed coatings. The Fig. 2 shows the XRD pattern obtained for pure Ni-P and all the BN concentrations applied coatings, that are Ni-P-0.5 g/L BN, Ni-P-1.0 g/L BN and Ni-P-1.5 g/L BN. A broadened peak can be clearly seen at around 2θ equal to 44.5° that depicts the presence of FCC structured Ni(111) (ref. code 98-007-6667), implying that the coatings have an amorphous nature. This is supported with the idea that, the addition of BNNPs in the Ni-P matrix generates a heterogenous nucleation process while coating is being developed in the electrodeposition process, which results in disfigurement in the structure [34,35]. Additionally, another justification for this occurrence is due to the changes happening by the presence of phosphorus atoms present in the nickel lattice. The P atom in Ni lattice causes a distortion and obstructs the position of Ni atoms in the lattice, leading to an overall amorphous structure. It has been reported that increasing the P content in the Ni-P deposited coatings, makes their structure go from nanocrystalline and amorphous to completely amorphous [36] [37]. Another diffraction peak is seen at 2θ equal to 82.3° , resembling iron, Fe(112) (ref. code 98-063-1729), presence, which is most likely interference from the substrate. Moreover, it is worth to note the presence of peak at 2θ equal to 26.7° , in case of 0.5 g/L, 1.0 g/L and 1.5 g/L BN coatings. This peak

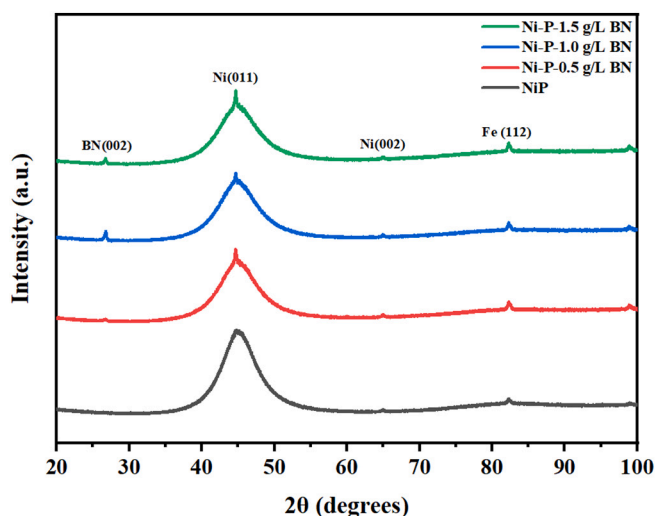


Fig. 2. XRD spectra of Ni-P, and Ni-P-BN coatings with increasing BN concentrations of 0.5, 1.0 and 1.5 g/L.

represents the existence of BN particles, BN(002) (ref. code 98-016-8892). The BN(010) at 2θ 41.7° and BN(012) at 2θ 50.2° may not be identified due to the amorphous Ni, which overshadows the existence of other peaks of BN. Additionally, the BN content is low in the matrix, comparing to the Ni content present. Therefore, the Nickel peaks show high intensity dominating over the BN, and the intensity was found to be very low [38,39].

3.2. XPS analysis

XPS survey spectra of Ni-P-1.5 g/L BN nanocomposite coating is shown in Fig. 3 and considered elements of the coatings are indicated based upon their position over the axis of binding energy. Peaks of Ni2p, P2p, B1s, and N1s can be clearly observed, indicating their presence in the nanocomposite coating. Peaks representing oxygen and carbon could be due to the formation of superficial oxides or impurities prior to the installation of sample for analysis.

Highly magnified spectra of each element with their respective chemical state are displayed in Fig. 4. Tougaard background correction was employed along with Lorentzian-Gaussian fit on the experimental data to deconvolute the peaks of various elements for identifying their actual chemical state. Binding energy range of 845 eV to 887 eV in Fig. 4 (a) reveals the photoionization spectral region of Ni2p which is deconvoluted into peaks positioned at 852.3 eV and 869.6 eV indicating Ni2p_{3/2} and Ni2p_{1/2} of metallic nickel state with their satellite peaks positioned around 858 eV and 876 eV. Furthermore, peaks indicated by 853.6 eV and 872 eV can be ascribed to the oxides of nickel for the Ni2+ chemical state respectively [13,40,41]. High magnified region of phosphorus (P2p) with its deconvoluted peaks are shown in Fig. 4(b). Peaks at 129.1 eV and 130.2 eV can be attributed to P2p_{3/2} and P2p_{1/2} indicating the phosphorus in its elemental state. More to this, the broad peak observed around 132.5 eV can be regarded to the hypophosphites and/or intermediate chemical states of phosphorus that are remained in these forms as the solid solution in the as developed coatings [13,40–42]. High magnification region of B1s is displayed in Fig. 4(c) with its deconvoluted peaks indicating the presence of boron with the peak identified at 186.9 eV attributing to the boron in its metallic form [43,44]. Moreover, peak of B–N band can be easily observed at 189.4 eV confirms the successful incorporation of boron nitride in the solid solution matrix of Ni-P in Ni-P-1.5 g/L BN nanocomposite coating [45,46]. The non-existence of any other chemical state of B1s indicates its inert and non-reactive nature toward the reactions and activities during the formation of nanocomposite coating in the chemical bath of

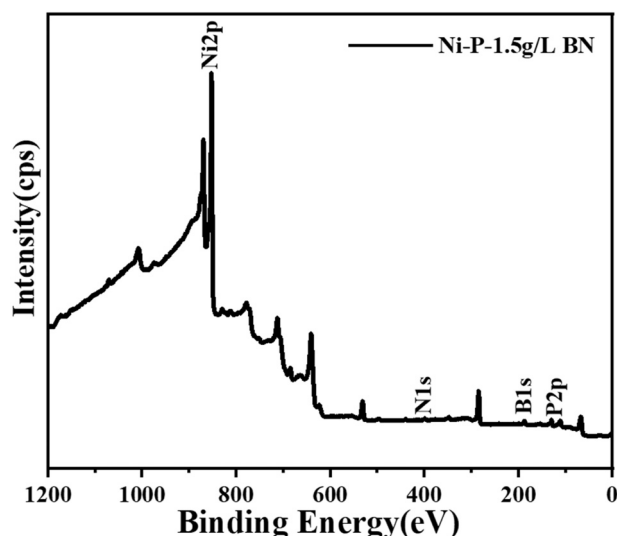


Fig. 3. Survey spectra of Ni-P-1.5 g/L BN nanocomposite coating.

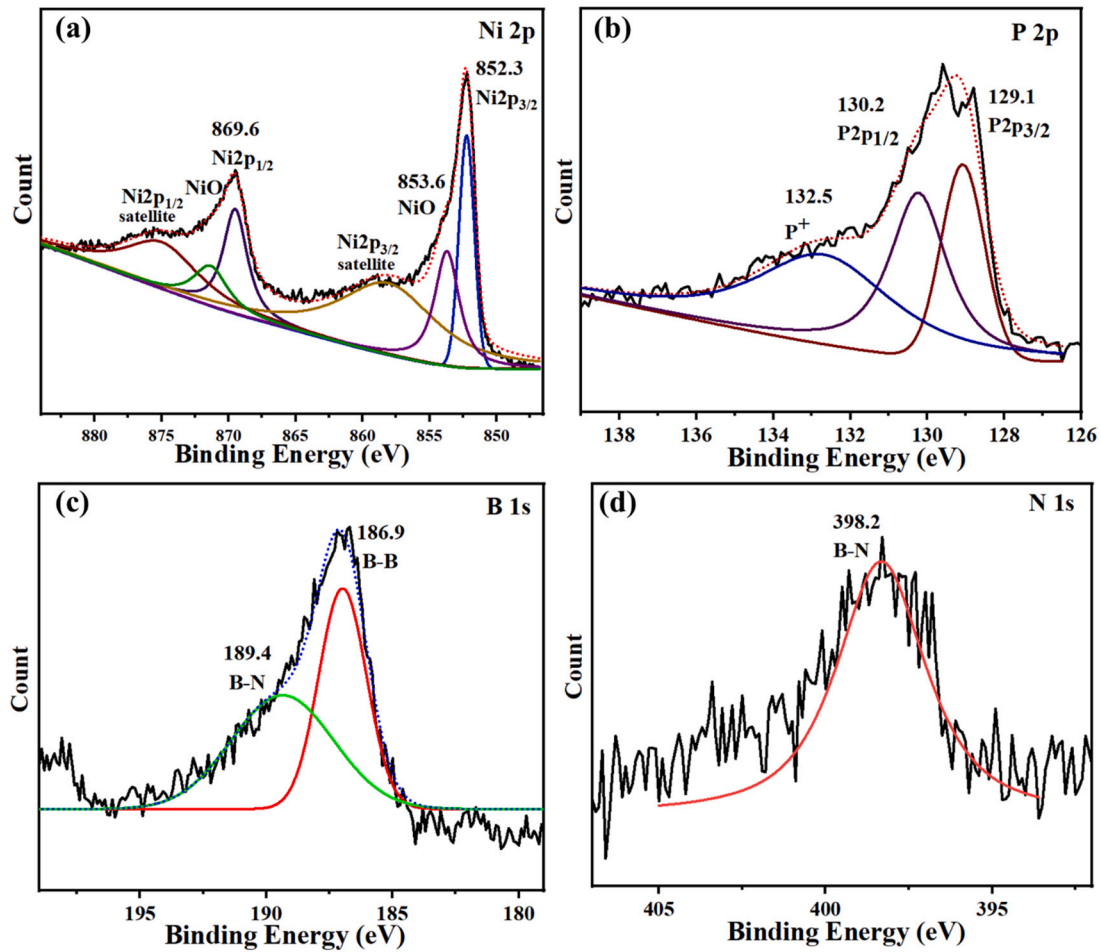


Fig. 4. High magnification XPS spectra of (a) Ni2p, (b) P2p, (c) B1s and (d) N1s in their respective chemical states.

electrodeposition. Furthermore, Fig. 4(d) shows the spectra of N1s, the peak revealed around 398.2 indicates the B–N bond of boron nitride of the reinforcement in its inert state that are also reported in the literature [45,46].

3.3. Surface morphology

Fig. 5 reveals the morphological details of pure Ni-P coatings along with increased concentrations of BNNPs in the chemical bath. Fig. 5(a), (a'), (b), (b'), (c), (c'), (d) and (d') shows the micrographs at 2500× and 5000× of magnification. It can be inferred from Fig. 5(a) and (a') that pure Ni-P coatings possess near to planar geometry with irregularly

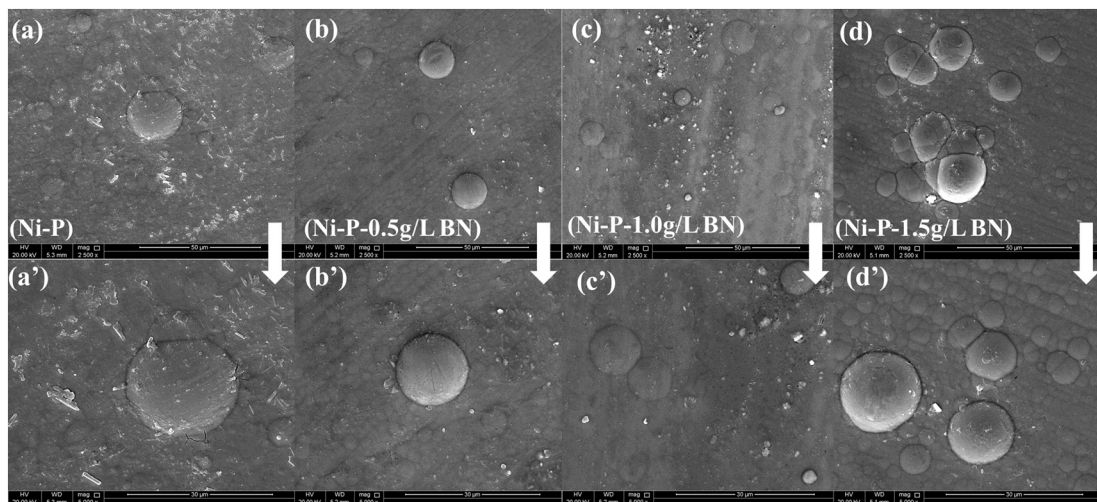


Fig. 5. SEM micrographs at 2500× magnification (a) Ni-P, (b) Ni-P-0.5 g/L BN, (c) Ni-P-1.0 g/L BN, (d) Ni-P-1.5 g/L BN and at 5000× magnification (a') Ni-P, (b') Ni-P-0.5 g/L BN, (c') Ni-P-1.0 g/L BN, (d') Ni-P-1.5 g/L BN nanocomposite coatings.

originated cellular structure without any observable defects as reported in the literature [47,48]. Introduction of BNNPs modifies the planar microstructure to colony like columnar structure with increased cellular growth sites with increasing the concentration of BNNPs in the chemical bath as seen in Fig. 5(b), (b'), (c), (c'), (d) and (d'). This can be ascribed to the heterogeneous nucleation sites due to the BNNPs in the chemical bath. Moreover, at higher concentration of 1.5 g/L of BNNPs, the excessive nucleation leads to the successive cellular growth and morphology tries to shift toward nodular structure. Furthermore, size of the nodule seems to be reduced due to the introduction and increment of BNNPs in the chemical bath [35,39,49,50].

The thickness for electrodeposited Ni-P-BN reinforced coatings was measured using the gauge meter PosiTector 6000. Five measurements were taken, and an average was calculated. Table 2 presents the average thickness of all the coating compositions which is found to be around 23 μm .

Energy Dispersive X-ray spectroscopy was applied on Ni-P alloy matrix coating, as well the coatings with the incorporation of BNNPs. For clear comparison, the compositional analysis is shown for pure Ni-P and Ni-P-BN coatings in tabulated form. Fig. 6 shows the EDX analysis plot for pure Ni-P, Ni-P-0.5 g/L BN, Ni-P-1.0 g/L BN and Ni-P-1.5 g/L BN. The peak at 2 keV indicates the presence of P element. The peaks at about 0.8 keV and 7.5 keV is for Ni. Fig. 6(b–d) confirmed the presence of BN in the developed coatings, from the exhibited peaks in the EDX spectra. The atomic wt% of B is noticed to be increasing from 3.85 wt% to 4.69 wt% to 7.87 wt%, as the incorporation of BN concentration increases from 0.5 g/L to 1 g/L to 1.5 g/L. However, for N, the weight% is observed to be 0.34 wt%, 0.39 wt% and 0.35 wt%, for Ni-P-0.5 g/L BN, Ni-P-1.0 g/L BN and Ni-P-1.5 g/L BN respectively. Taking into consideration the fact that EDX technique is a single point analysis, therefore it is likely that lower N was detected at that specific location, demonstrating a mean value of 0.36 wt%, rather than a significant increment with the increase in BN concentration. Furthermore, it can be noticed also that the deposition of BNNPs causes a reduction in the Ni content. From pure Ni-P, containing 88.58 wt% of Ni, lowers to 85.60 wt% in Ni-P-0.5 g/L BN, which further decreases to 84.17 wt% in Ni-P-1.0 g/L BN, that is seen to lower down to 82.49 wt% in Ni-P-1.5 g/L BN. Additionally, it can be observed that the P content for pure Ni-P and Ni-P-BN coatings is 9 or 10 wt%. Coatings with phosphorous contents >9.0 wt% are considered as high phosphorous Ni-P coatings and show superior corrosion resistance [51]. Therefore, in this case, it can be concluded that the BN reinforced coatings contain a significant amount of P, which has not been replaced by B or N, are high phosphorous Ni-P coatings. This causes them to have amorphous microstructure and thus great corrosion resistance [52].

The Fig. 7(a–d) shows the topographical details of pure Ni-P and BNNPs reinforced nanocomposite coating. A comparison is carried out for the quantitative result, Root Mean Squared Roughness (R_{rms}), to have better idea of the effect of increasing BN concentration on the composite coatings fabricated. It is observed with the increase in incorporation of nanoparticle BN content, the R_{rms} values are rising gradually, implying that the surface roughness is increasing, which is due to the incorporation of insoluble and hard BNNPs in the Ni-P matrix. These results are in agreement with other literatures as similar results have been shown in other studies [53,54]. Furthermore, the AFM proved that the coating does not contain any defects, such as cracks or pores, with good homogeneity [13,20].

Table 2

Thickness of coatings Ni-P, Ni-P-0.5 g/L BN, Ni-P-1.0 g/L BN and Ni-P-1.5 g/L BN samples recorded.

Coating composition sample	Ni-P	Ni-P-0.5 g/L BN	Ni-P-1.0 g/L BN	Ni-P-1.5 g/L BN
Thickness (microns)	22.60	23.33	22.08	22.27

3.4. Mechanical properties

3.4.1. Microhardness and nanoindentation

The Fig. 8(a) illustrates the results of microhardness measured for the pure Ni-P coatings and the reinforced coatings. It can be clearly observed that the reinforcement improves the hardness of the metallic coating. The pure Ni-P coating without any reinforcement has hardness ~ 440 HV₂₅. The addition of 0.5 g/L BN to the Ni-P raises the hardness to about 615 HV, and further increase is seen with addition of 1.0 g/L BN and 1.5 g/L BN. The maximum hardness reached is in terminal Ni-P-1.5 g/L BN equal to 697 HV₂₅, which is about 58 % more than for pure Ni-P coatings. This increment in hardness can be explained by this idea that BN nanoparticles behave as a protection against plastic deformation and inhibit the movement of dislocations. Furthermore, this observation can also be justified by the dispersion hardening. When hard ceramic materials are in the Ni-P matrix, the nanoparticles form composite structures and boost the load-endurance features [35]. The NPs do not deform when a crack propagates, and it therefore changes the deformation path of the crack. The nanoparticles provide an obstacle to the cracks. Overall, if the incorporation of NPs is increased, the structure of pure Ni-P matrix is improved with more compactness and becomes denser, and obstructs the flaws, pits or cracks present [7,55].

Nanoindentation test was performed to further analyze the mechanical properties of prepared coatings. Fig. 8(b) illustrates the nano-indentation loading and unloading profiles of the pure Ni-P and Ni-P-BN reinforced coatings, with the increase in BN concentration. More importantly, it was observed that with the increase of BN concentration in the coatings, the indentation depth decreased under constant loading. The terminal depth of indentation, for pure Ni-P about 18.3 nm is reduced to about 5.6 nm, with the addition of 1.5 g/L BN NPs to the Ni-P. The maximum indentation depth was observed to decrease from 260 nm for pure Ni-P to 232 nm for Ni-P-1.5 g/L BN. This leads to the fact that incorporation of higher BN nanoparticles improves the resistance to indentation and results in an enhancement in mechanical properties of the coatings, as the BNNPs in the Ni-P matrix act as an obstacle in response to plastic flow [10,13,56]. The load displacement curves for all samples depict no irregularity or nonuniformity, implying that the coatings were synthesized with no cracks or pores [10,53]. Using the load displacement curves, mechanical properties such as hardness and elastic modulus were determined by Oliver-Pharr method as tabulated in Table 3 for a clear comparison. Incorporation of BNNPs has resulted in an increase in hardness from 4.18 GPa to an ultimate value of 7.15 GPa which are in harmony with the microhardness results. With the successive incorporation of BN, the Elastic modulus was observed to be increased. The elastic modulus for pure Ni-P coating is 8.01 GPa and with the addition of Boron Nitride nanoparticles, the E_c improved to 10.88 GPa.

3.4.2. Erosion analysis

Fig. 9(a) presents rms depth of the erosion impact versus angle variation at four distinct namely 30°, 45°, 60° and 90°. A general incremental trend can be observed up to 60° of impact angle for both types of the coating before a slight decrease in depth can be observed at 90°. Moreover, the crater depth for pure Ni-P coatings is significantly larger than the crater depth for 1.5 g/L BN reinforced Ni-P-BN nanocomposite coatings. The depth at 30° impact angle for Ni-P coating is observed to be around 15 μm which is decreased to nearly 7 μm in case of nanocomposite coatings indicating the enhanced strength imparted to the Ni-P matrix by hard BNNPs. Moreover, similar increment is observed at 45° and 60° of impact angle indicating the erosive behavior of brittle. Furthermore, at 90° of impact angle the depth reaches to the termination of coating thickness after which carbon steel substrate imparts the ductile behavior to the erosion mechanism. Such erosive behavior of brittle and ductile materials is as reported in the literature [57–60]. Fig. 9(b) reveals the comparison of crater length showing a decent decrease of crater length of nanocomposite coating when the angle of

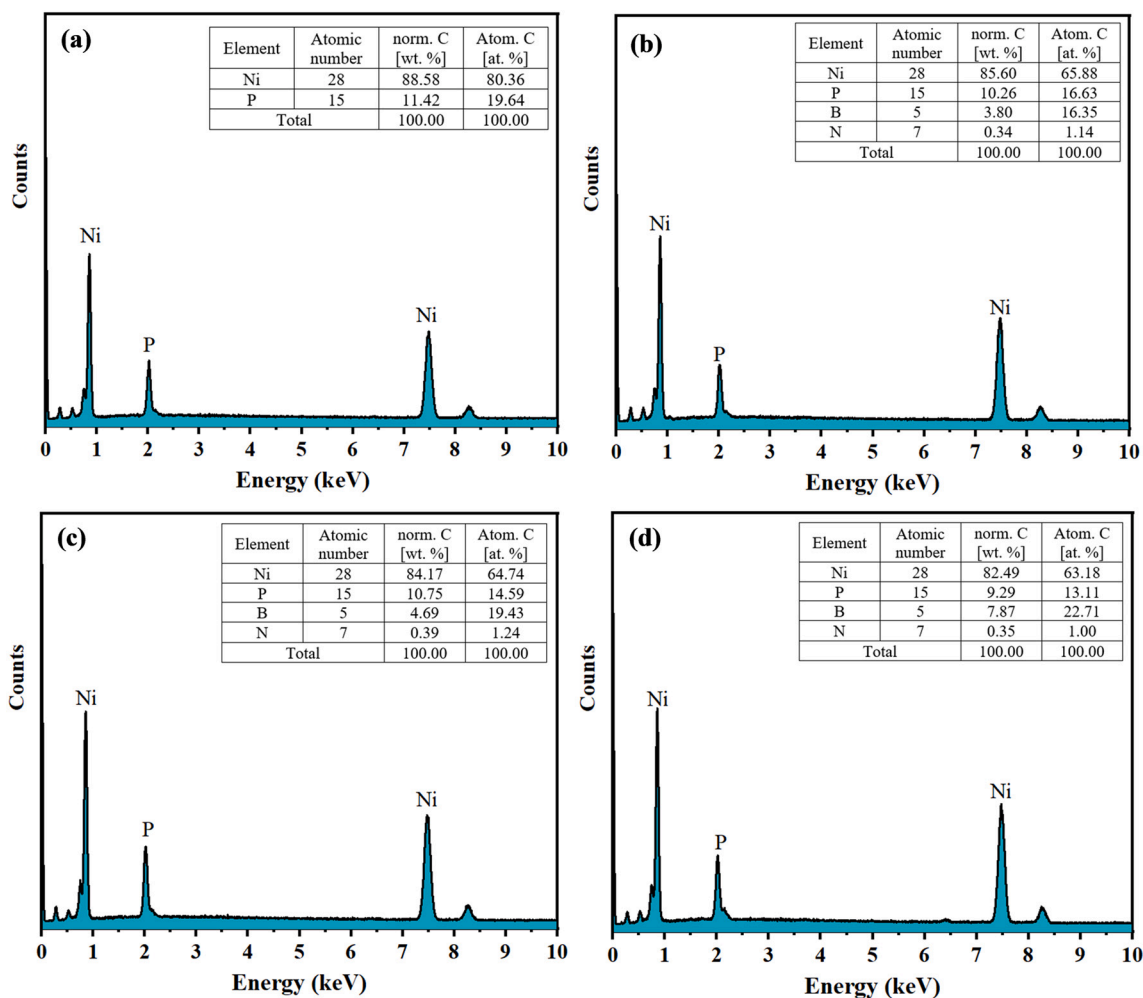


Fig. 6. EDX composition analysis of (a) Ni-P (b) Ni-P-0.5 g/L BN (c) Ni-P-1.0 g/L BN (d) Ni-P-1.5 g/L BN.

impact is increased from 30° to 90° for both the types of coating. It further indicates the improved strength of the BNNPs reinforced nanocomposite coating in comparison to the pure Ni-P coatings. Fig. 10(a), (c), (d) and (f) shows the 3 dimensional contours of the erosive surfaces revealing the crater geometry of pure Ni-P and Ni-P-1.5 g/L BN nanocomposite coating. It can be clearly observed that a shift of crater geometry from elliptical to spherical shape when the angle is varied from 30° to 90°. This can be ascribed to the surficial material removal at lower angle (30°) of impact which transforms into deeper penetration at higher angles (90°) as reported earlier [33,61,62]. Moreover, Fig. 10(b) and (e) presents the profilometric path of the coatings at varied angles showing the improvement of erosive strength of Ni-P-1.5 g/L BN nanocomposite coating in comparison to pure Ni-P coatings. It can further be noted that at 90° of impact angle, the penetration reaches to the maximum of almost 40 μm for pure Ni-P coating and 34 μm for BNNPs reinforced nanocomposite coating such that the ductile behavior of carbon steel substrate can be accounted for erosive mechanism of coatings.

3.5. Corrosion studies

3.5.1. Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy is widely utilized to understand the corrosion resistance mechanism of coatings. Fig. 11(a) presents the fitted bode plot of the carbon steel, Ni-P coatings and BNNPs reinforced nanocomposite coatings. Fig. 11(b) and (c) displays the equivalent electrical circuits adopted for fitting the experimental

data such that improvised Randle cell was used for carbon steel and cascaded two-time constant circuit for as deposited coating. Electrical elements used for fitting include R_s accounting for solution resistance, R_{ct} representing the charge transfer resistance, R_{po} corresponds to pore resistance of the coatings, CPE1 and CPE2 represents constant phase elements which accounts for the capacitor owing to any surface inhomogeneity and discrepancy obtained using the equation below,

$$\frac{1}{Z_{CPE}} = Q(j\omega)^n \quad (1)$$

where Q is admittance, j is an imaginary number, ω is angular frequency of the AC signal, and n is the CPE exponent.

- $n = 1$: element is pure capacitor.
- $n = 0$: element is pure resistor.
- $0 < n < 1$: element deviated from ideal behavior.

Phase angle response of the carbon steel and as prepared coatings are shown in Fig. 11(a). It can be observed that the phase angle of carbon steel in the mid-frequency region is narrowed, presenting a single-time constant. An appreciable shift in the phase angle diagram toward mid-high frequency region along with increased broadness reveal the presence of cascaded two time constant for the Ni-P and successive nanocomposite coatings. Impedance response of the substrate and as prepared coatings are presented in Fig. 11(a), increased impedance of pure Ni-P coatings can be attributed to formations of protective hypophosphite layer which impedes corrosion as reported in the literature

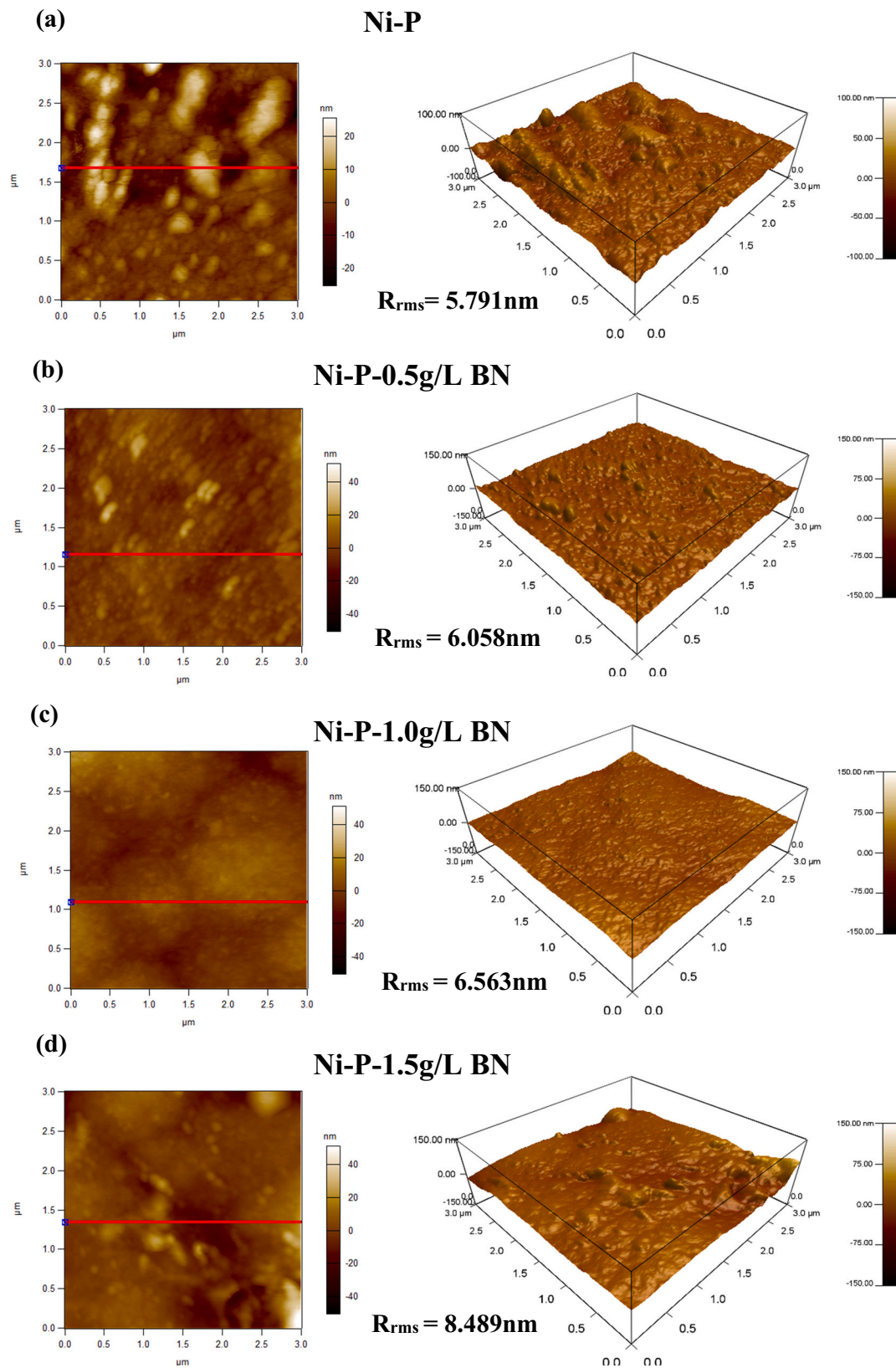


Fig. 7. AFM 2D and 3D images of (a) Ni-P (b) Ni-P-0.5 g/L BN, (c) Ni-P-1.0 g/L BN, (d) Ni-P-1.5 g/L BN nanocomposite coatings.

[13,39]. Moreover, when the BNNPs are introduced in the chemical bath it fills the voids and micropores of the coatings and hence enhances its corrosion resistance. Increasing the concentration of BNNPs in chemical bath leads to excessive deposition of inert nanospecies which further

occupies the microvoids and results in increasing the overall impedance. Moreover, charge transfer resistance (R_{ct}) also increases on increasing the BNNPs concentration which can be attributed to the resistance provided by the inert BNNPs by covering the active area and restricting

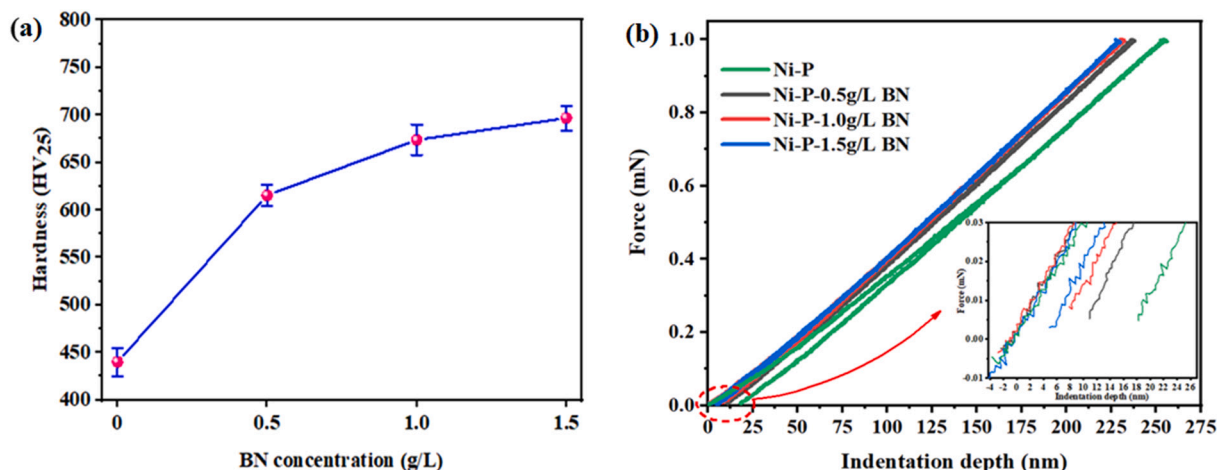


Fig. 8. (a) Hardness (b) nanoindentation load and displacement profiles of Ni-P, and Ni-P-BN coatings with increasing BN concentrations of 0.5, 1.0 and 1.5 g/L.

Table 3
Mechanical parameters obtained from nanoindentation test.

Sample	Modulus, E _c (GPa)	Hardness (GPa)	Maximum Indentation Depth, H _{max} (nm)
Ni-P	8.01	4.18	259.66
Ni-P-0.5 g/L BN	10.17	6.41	237.47
Ni-P-1.0 g/L BN	10.69	6.75	231.64
Ni-P-1.5 g/L BN	10.88	7.15	231.58

the movement of electron from the solution to the carbon steel substrate as explained in the literature [13,39].

The Nyquist plots shown in Fig. 12(a) reveal the improved impedance trend as described by Bode plot, increased diameter of the semi-circular projectile loop shows a sequential rise in impedance due to the incorporation of BNNPs, with the highest value of impedance exhibited at Ni-P-1.5 g/L BN approximately equal to 5 kΩ·cm². Furthermore, Fig. 12(b) presents a comparison for R_{p0} and R_{ct} with the introduction and increment of BNNPs in the chemical bath. It can be clearly observed that the pore resistance and charge transfer resistances are improved as a result of incorporating and incrementing BNNPs in the Ni-P matrix. This enhancement in the corrosion resistance can be primarily accredited to (i) pores occupancy of BNNPs in the Ni-P matrix, (ii) decrease of dynamic area of Ni-P matrix by due to inert BNNPs.

3.5.2. Potentiodynamic polarization

Potentiodynamic polarization plot is presented in Fig. 13, it is observed that the polarization curves shift toward the less negative potential and lower current densities, with the increase in incorporated BNNPs. For a better comparison, using these polarization curves, some electrochemical parameters such as, corrosion current density (i_{corr}), corrosion potential (E_{corr}), cathodic and anodic Tafel extrapolation (β_a and β_c), and corrosion protection efficiency (PE%) are determined and tabulated in Table 4.

The corrosion protection efficiency was calculated by the following equation:

$$PE\% = \left(1 - \frac{i_{sample}}{i_{steel}}\right) * 100\% \quad (2)$$

where, i_{sample} is the corrosion current density for the coating and the i_{steel} is for carbon steel.

Table 4 results show that the highest current density, about 58 μAcm⁻² is of carbon steel, a considerable decrease is seen in the current densities of 20.4 μAcm⁻² is can be observed for pure Ni-P coatings suggesting an improvement in the corrosion behavior which further decreases to 17.0, 10.2 and 3.2 μAcm⁻² for the incorporation of 0.5, 1.0 and 1.5 g/L BNNPs in Ni-P matrix, leading to an improvement toward corrosion. The corrosion protection efficiency is seen to be increasing, from about 65 % to 95 %, from pure Ni-P to Ni-P-1.5 g/L BN. Therefore, after analyzing the corrosion parameters, the sample Ni-P-1.5 g/L BN is

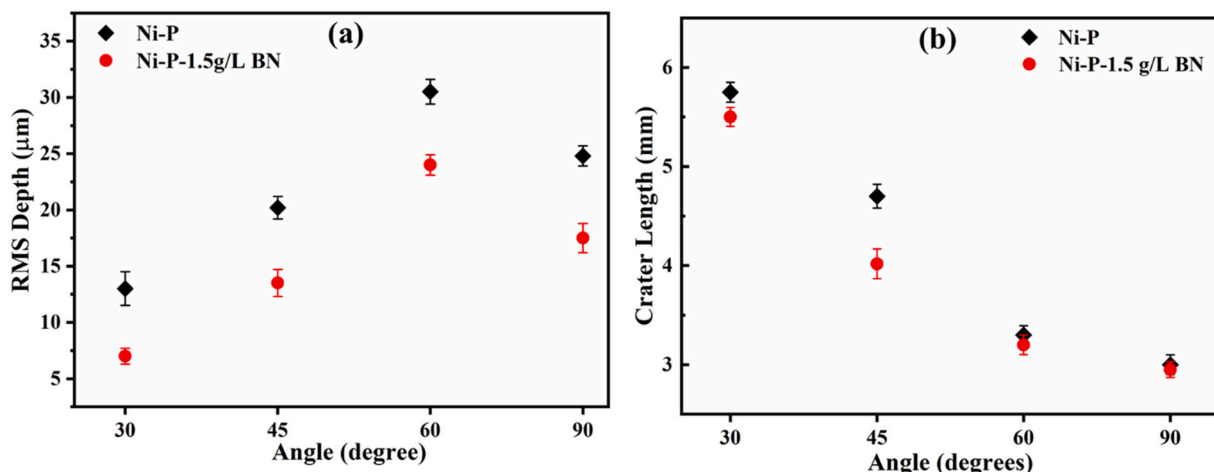


Fig. 9. Comparison of (a) RMS impact depth and (b) maximum crater length of Ni-P and Ni-P-1.5 g/L BN nanocomposite coating.

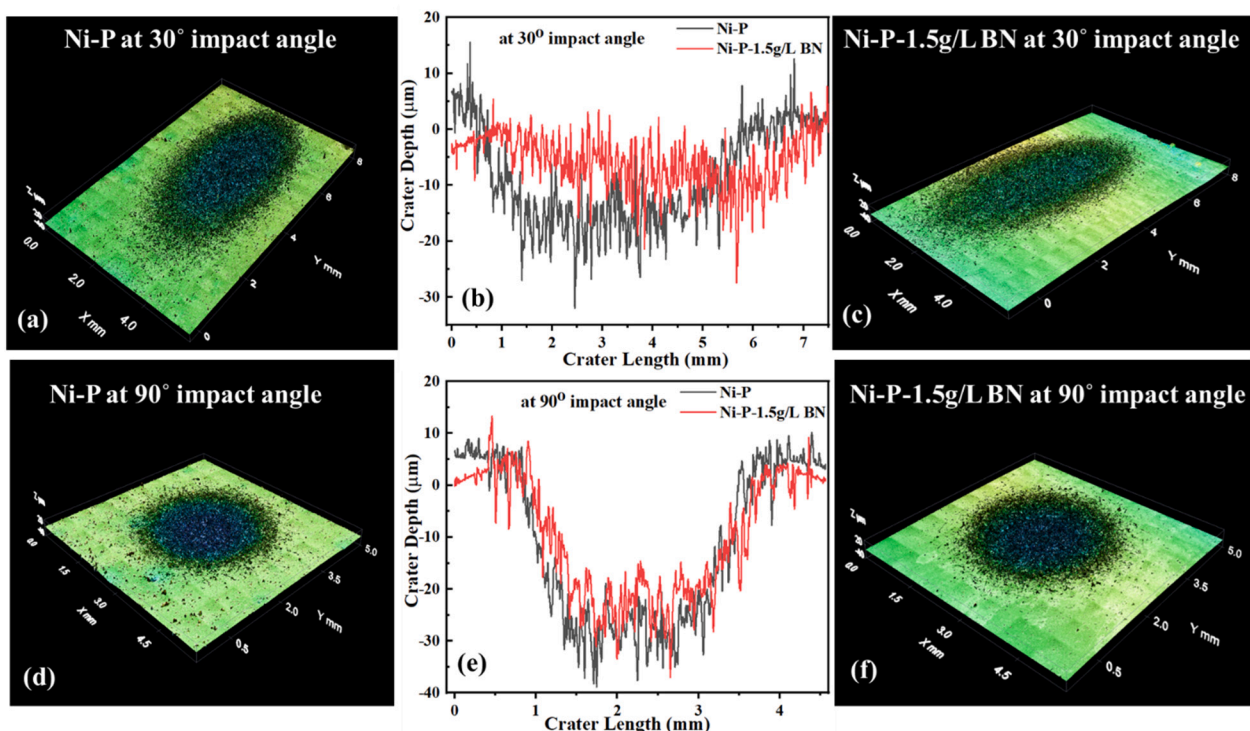


Fig. 10. Topographical visualization of erosion behavior of (a) Ni-P, (c) Ni-P-1.5 g/L BN and (b) their crater profile at 30°. Its comparison, (d) Ni-P, (f) Ni-P-1.5 g/L BN and (e) their crater profile at 90° impact angle.

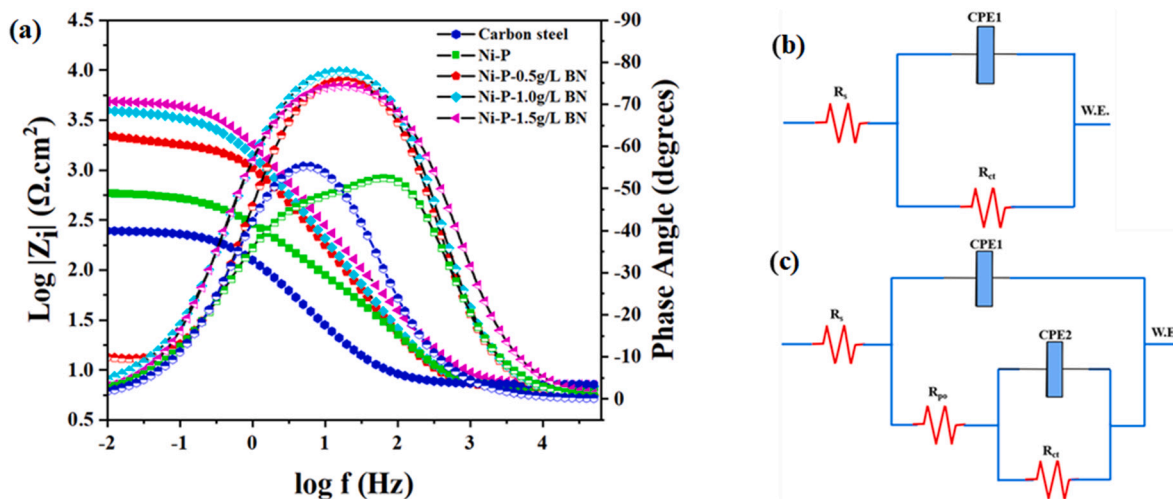


Fig. 11. (a) Bode plots: phase angle and Impedance diagram of carbon steel, Ni-P and Ni-P-BN NC coatings and circuit diagrams for EIS fitting of (b) carbon steel and (c) coatings.

the most effective for corrosion inhibition, with its lowest i_{corr} of $3.2 \mu\text{Acm}^{-2}$ and highest corrosion protection efficiency of 95 %. This major improvement with the addition and increasing of inert and insoluble BNNPs can be attributed to the reduction of the active area by these nanospecies which also impedes the movement of active corrosion species by blocking its pathway to reach the substrate and initiate corrosion as described in the previous section [13,39].

4. Conclusion

Ni-P-BN nanocomposite coatings are successfully synthesized and effect of addition and increment of BNNPs are studied for structural, compositional, morphological, mechanical and electrochemical

properties. XRD and XPS confirmed the presence of elements Ni, P, B and N. SEM and AFM reveal the transformation of plane to nodule like morphology with increased roughness. Microhardness and nano-indentation results indicate a gradual enhancement in mechanical properties in the Ni-P coatings as the BN concentration is increased, reaching to the ultimate value (of microhardness~697 HV₂₅, hardness~7.15 GPa and elastic modulus ~10.88 GPa) for Ni-P1.5g/L BN. Erosion analysis reveal the strength of Ni-P1.5g/L BN nanocomposite coating in comparison to pure Ni-P coatings. EIS and Potentiodynamic polarization assessment proved the improvement in corrosion protection efficiency with the increment in BN concentration in Ni-P matrix with Ni-P1.5g/L BN exhibiting the highest PE% ~95 %. The Ni-P-BN nanocomposite coatings confirm superior mechanical and corrosion

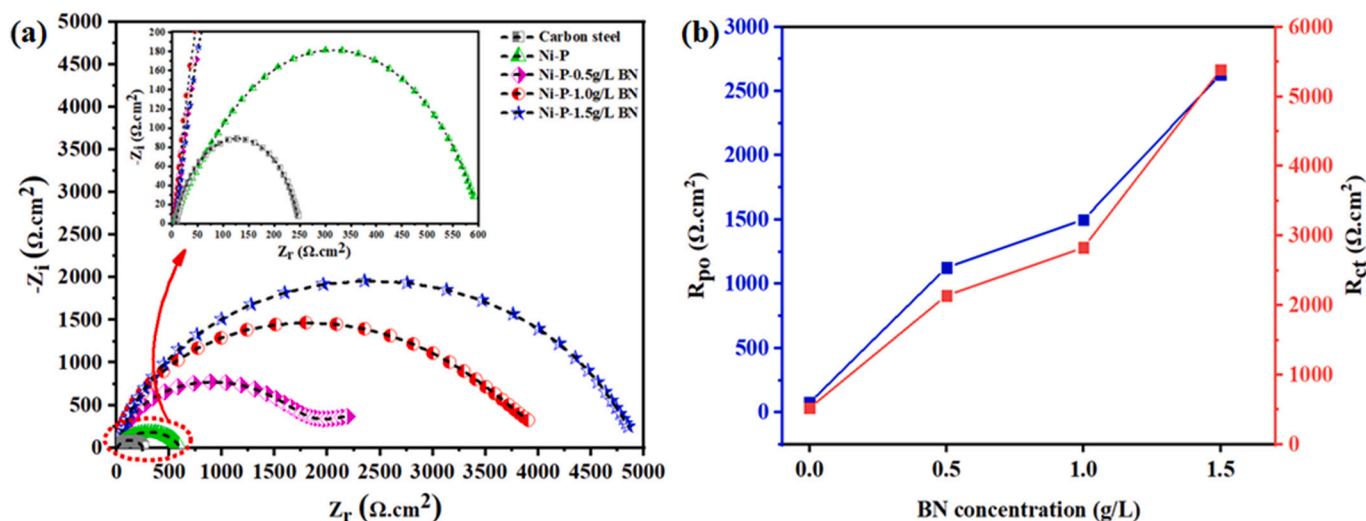


Fig. 12. (a) Nyquist plots of carbon steel, Ni-P and Ni-P-BN NC coatings and (b) Impedance vs BN concentration in the Ni-P-BN coatings.

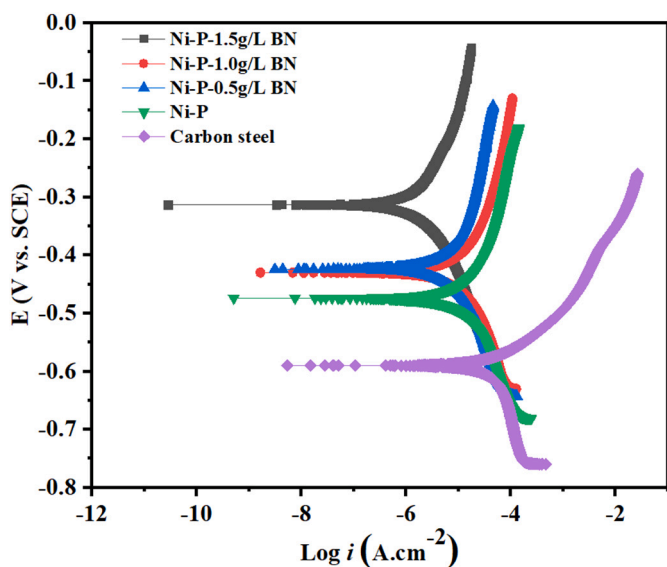


Fig. 13. Tafel plots of the carbon steel, Ni-P coatings before and after adding different concentrations of BNNPs (0.5, 1.0 and 1.5 g/L).

Table 4

Electrochemical corrosion parameters determined from potentiodynamic polarization curves of Ni-P and Ni-P-BN (with increasing concentration) immersed in 3.5 wt% NaCl medium.

Sample	E_{corr} (V vs SCE)	i_{corr} (μAcm^{-2})	β_a (V dec. ⁻¹)	β_c (V dec. ⁻¹)	PE%
Carbon steel	-0.59	57.5	0.075	-0.304	-
Ni-P	-0.47	20.4	0.298	-0.236	64.5
Ni-P-0.5 g/L BN	-0.43	17.0	0.321	-0.271	70.4
Ni-P-1.0 g/L BN	-0.42	10.2	0.426	-0.260	82.3
Ni-P-1.5 g/L BN	-0.31	3.2	0.310	-0.220	95.2

protection features than pure Ni-P, implying their effective use in automobile, aerospace and oil and gas industries.

CRedit authorship contribution statement

Izza Fatima: Investigation, Validation, Data curation, Writing – original draft, Visualization. **Osama Fayyaz:** Conceptualization,

Methodology, Formal analysis, Writing – review & editing. **Moinuddin M. Yusuf:** Data curation, Formal analysis. **Abdullah Al Ashraf:** Data curation, Formal analysis. **R.A. Shakoor:** Resources, Supervision, Project administration, Funding acquisition, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

Acknowledgment

The present work is supported by Qatar University Grant-IRCC-2020-006 and IRCC-2022-491. The opinions expressed in this article are solely the responsibility of the authors. The authors acknowledge the services of Central Laboratory Unit (CLU), Qatar University for Microstructural analysis (FE-SEM/EDS). XPS facility of Gas Processing Center (GPC), Qatar University, was utilized to study compositional analysis. Open Access funding is provided by the Qatar National Library.

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