RESEARCH ARTICLE

Development and Characterization of Bio-based PLA-PU-Garnet Coatings towards Anti-corrosion

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Although synthetic polymers offer excellent versatility in coating applications, due to intrinsic permeability to gases and vapours, including, but not limited to, oxygen, carbon dioxide, and organic vapours, their application is highly limiting. On the other hand, some natural polymers render great alternative to synthetic polymers in such areas of research. In this work, flexible polyurethane is used to make the PLA as flexible coating. Hence, our present study is focussed on the development of PLA-PU hybrid organic polymer with the incorporation of garnet waste powder (in-organic) as coatings deposited by brush-coating method on mild steel. To overcome the brittle nature of the PLA, small amount of polyurethane having a rubbery nature is added along with garnet. The mechanical and barrier properties significantly be improved with the addition of a garnet (5%, 10%, and 15%) in PLA-PU blends. The various characterization techniques like Fourier Transform Infrared Spectroscopy (FTIR), Crosshatch test, contact angle, and morphological analysis by Scanning Electron Microscopy (SEM) and Electrochemical Impedance and Salt-spray and Cross-hatch adhesion tests are checked.

Introduction

High-performance coatings with enhanced properties such as stiffness, and chemical and hygroscopic resistance tend to offer a wide variety of applications in areas such as aerospace, marine, automotive, electronics, industrial components, pollution control equipment, rehabilitation products, etc. Accordingly, hybrid polymer composite coatings have been developed to provide the emerging demands in the field of high-performance engineering including wear and corrosion-resistant applications. Incorporating appropriate inorganic fillers in biopolymers has been endowed as a promising route to enrich the barrier properties of biopolymers for specific end-use applications. The use of organic (PLA-PU) and inorganic (Garnet) hybrid coatings can enhance their adhesion to metal substrates and improve corrosion resistance.

A coating is a barrier applied to the surface of an object. The purpose of applying the coating may be decorative, functional, or both. A major consideration for most coating processes is that the coating is to be applied at a controlled thickness, and a number of different processes are used to achieve this control, ranging from a simple brush for painting a wall to some very expensive machinery applying coatings in the electronics industry. There is a wide range of coating processes for many different types of material at thicknesses ranging from just a few microns, up to several millimetres.

This paper stands unique in addressing novelty in creating and utilizing biomaterials for the high-performance coatings manufacturing technique. The development of bio-based coatings based on PLA modified with PU and loaded with garnet waste has never been tried and reported so far. This bio-based PLA-PU-Garnet coating is an environment-friendly and recyclable product at an affordable cost compared to conventional synthetic polymer-based products that pose a severe environmental threat. This organic-inorganic hybrid bio-composite coating shall challenge the currently existing synthetic coating materials in terms of its enhanced degradability, barrier properties, and performance as well. Apparently, this research work will also be a paradigm in contributing to the development of bio-polymers that are useful in reducing waste disposal and replacing diminishing resources.

Experimental

Methodology

The obtained Garnet dispersion solution was mixed with two component PU-PLA coatings and stirred magnetically for 10-15 min. Prior to coating, the specimens were rubbed with emery paper from 150 to 1000 grit, respectively, degreased in acetone, and then dried at room temperature. The neat PU-PLA coating was coated on Q235 steel at the thickness of 100-120 μm, and dried at room temperature. **Fig. 1** exhibits the preparation process of Garnet-based PU-PLA anti-corrosion coatings. When the Garnet content was 5%, 10%, and 15%, a three-dimensional random distribution of Garnet existed in the PU-PLA coating, which gave a tortuous path for electrolytes to penetrate through the coating. When the Garnet content was 5%, 10%, 15%, uniformly distributed in the coatings, and an aggregate existed in the coatings, which may increase the anticorrosion performance in PU-PLA coatings.

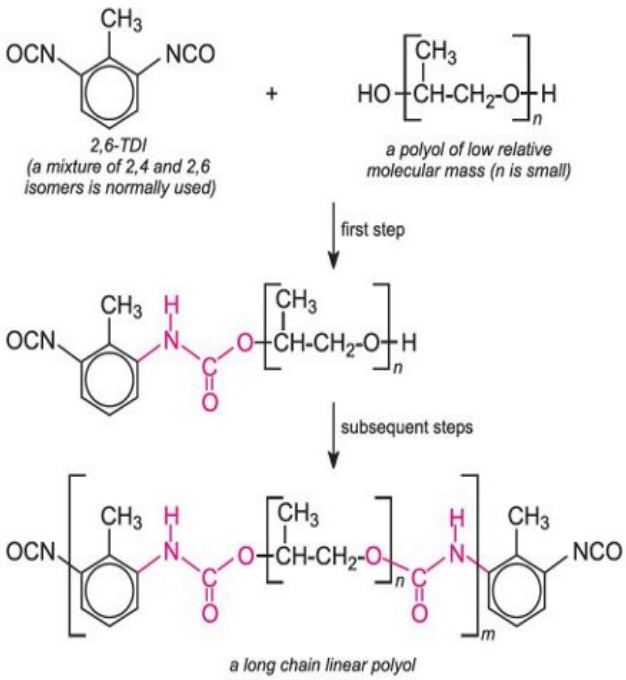

Fig. 1. Structure of Polyol (Polyurethane).

Materials

Polylactic Acid (PLA)

The advantage of PLA as a coating material is a great benefit due to its environmental impact and ability to environmentally degrade resulting in better coating performance than all the other coating products used today. Compared to petroleum-based plastics, bioplastics such as PLA are carbon neutral as their renewable sources absorb carbon. It does not release toxic fumes when oxygenated. PLA is biodegradable within three months having specific and controlled composting conditions. The mechanical and barrier properties of PLA significantly improved with the addition of a garnet (5%, 10%, and 15%) in PLA-PU blends.

Polyurethane (PU)

Polyols can be polyether polyols (Fig.1), which are made by the reaction of epoxides with active hydrogen-containing compounds. Polyester polyols are made by the polycondensation of multifunctional carboxylic acids and polyhydroxy compounds. They can be further classified according to their end-use.

Vegetable oil-based synthesis of polyurethane

Vegetable oils represent the promising route to renewable chemicals and polymers because of their inherent biodegradability, ready availability, and low toxicity. These Vegetable oils have been used in coatings and paints for centuries because their unsaturated oils can polymerize when exposed to oxygen in the air. Unsaturated fatty acids in vegetable oil are linoleic acid, and oleic acid containing one, two, and three double bonds between two carbon atoms respectively.

Preparation of PU Based on EKO-MA (EKO-MA PU)

2g of EKO-MA polyol was mixed with 1% w/v DBTDL catalyst in a 250ml Round bottom flask for 20 min in a nitrogen atmosphere was heated to 50°C at 400 rpm and then IPDI was added dropwise to the Rb so that the molar ratio of NCO/OH was between 0.8 and 1.6 and mixed for a further one hour at 50°C at 300 rpm and finally the reaction continued for 4 hours and the semi viscous fluid polyurethane was obtained (Saha et al.,2019).

Garnet

Garnet (G) is a gemstone that has been known and used by humans for many thousands of years. The type used in water jet machining is red garnet. Once the work is finished the used garnet is considered as waste. Nearly 70,000 pounds of waste sand is generated annually by the cutting operation. So, we used waste as a useful material. The usability of garnets from different suppliers and/or mining sites can differ due to the variability in their mechanical properties, namely hardness and stiffness.

Preparation of Coating Solutions

PLA pellets were dried in a vacuum oven at 50ºC at 100 mbar for 4 h. Solutions containing 5%, 10 %, 15%, 20 %, and 25 % of PLA were prepared by dissolving the pellets in suitable solvents with constant stirring at room temperature.

Coating of Mild Steel Samples Mild steel materials were cut into 25 mm X 25 mm square plates and 60mm X 150mm rectangular plates and were placed on a leveled surface table. A brush was used to apply the PLA solution over the surface of the MS plate and allowed to dry at room temperature for 24 hours.

Preparation of PLA-PU Blend Coating Solution

Castor oil (PU) resin and hardener were mixed in the prepared PLA solution. The polymer blend of PLA and castor oil (PU) is shown in **Fig. 2**. PU resin and hardener were mixed into PLA solution in a similar procedure as mentioned above, with varying percentages (5%, 10%, 15%, 20%, and 25%) using a homogenizer until they mixed completely and formed a uniform solution.

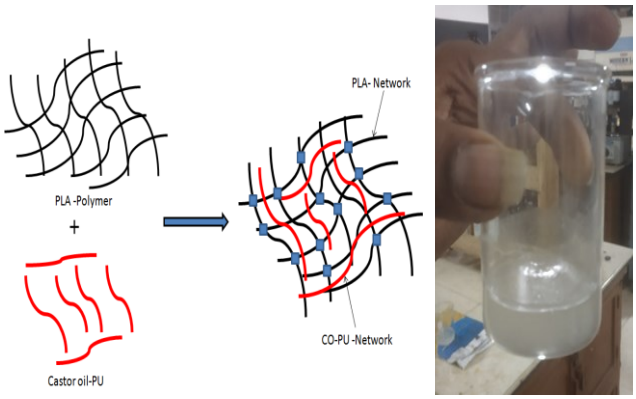

Fig. 2. PLA-PU polymer SIPN (Semi–Inter-Penetrating Network).

Preparation of PLA-PU-G Coating Solution

Then garnet powder with three different percentages 5%, 10%, and 15% is added into the blends of PLA-PU solution (70-30 ratio) under constant stirring conditions. The resultant coating formulation was applied on mild steel panels as a coating. In general brushing and spraying coating are the two types of techniques widely used.

Hence, we tried both techniques to find out which gives a good finish to the surface. Using a spray, the PLA-PU-G solution is poured into the spray gun using a 10-micron sieve cloth so that the larger particles are filtered and the coating obtained will be the same size and uniform. The sample coated (Figure.5) with the spray technique is shown which clearly shows that this technique is not suitable for coating.

Coating of PLA-PU

Surface-prepared mild steel is taken, and a blend of PLA-PU is coated over one side of the surface, which was then allowed to dry at room temperature for 24 hours. Later the second side of the surface was coated in a similar way and dried accordingly to get the coated samples.

Coating of PLA-PU-G for SEM

Surface-prepared mild steel is taken, and the blend of PLA-PU-G is coated over one side of the surface and then allowed to dry at room temperature for 24 hours. These samples were subjected to SEM analysis.

Contact Angle Test

Contact angle measurement has been used in the study of surface energy, wettability, and adhesion of low surface energy materials. The surface energy of a solid can be determined from the measurement of the contact angle of a pure liquid drop on that solid.

A right-angle lattice pattern is cut into the coating penetrating through to the substrate. The resistance of the coating to separation of the substrate is classified using the ISO table.

Results and discussion

Fourier Transform Infrared Spectroscopy

Fig. 3 describes the FT-IR spectra with several changes in chemical structure due to the interactions that occurred between PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% blends. **Table 1** enlisted above provides us with the band assignment of PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% blends. The regions of interest for PLA-PU are 1582.64, 1569.22, 1577.28, and 1584.96 cm-1 for the C=O stretch, and 3554.82, 3559.64, 3553.47, and 3558.15cm-1 for the O–H stretch. The peaks at about 1582.64, 1569.22, 1577.28, and 1584.96 cm-1, which belong to the C=O stretching, and peaks at about 1079.70, 1082.07, 1076.70, and 1080.15 cm-1 the C–O–C stretching of PLA-PU, are clearly visible in all the PLA-PU-G spectra.

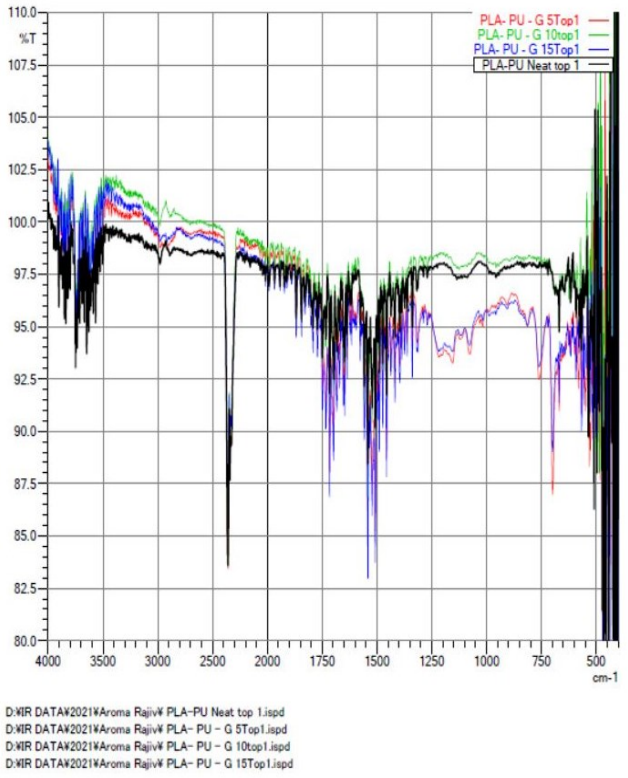

Fig. 3. FTIR graph of PLA-PU NEAT, PLA-PU-G 5%, PLA-PU-G 10%, PLA-PU-G 15%.

Table 1 enlisted above provides us with the band assignment of PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% blends.

Table 1. FT-IR: band assignment of PLA and its blend with PU and Garnet 5%, 10%, and 15%.

Type of vibrations	Chemical group	PLA-PU	PLA-PU-G (5%)	PLA-PU-G (10%)	PLA-PU-G (15%)
		Band position cm^{-1}			
O-H stretching	O-H	3554.82	3559.64	3553.47	3558.15
N-H stretching	N-H	3314.53	3318.52	3314.89	3318.52
N-C-O stretching	N-C-O	2284.53	2280.69	2285.11	2281.32
C=O stretching	C=O	1749.42	1751.36	1759.45	1761.58
C=O stretching	C=O	1582.64	1569.22	1577.28	1584.96
CH_3-asymmetric stretching	CH_3	1452.36	1452.40	1452.2	1453.36
COC- asymmetric stretching	C-O-C	1181.37	1180.44	1182.68	1181.37
CH_3- rocking	CH_3	1128.38	1130.29	1124.38	1131.73
COC- symmetric stretching	C-O-C	1079.70	1082.07	1076.70	1080.15
C-CH_3 – stretching	C-CH_3	1041.90	1041.56	1042.90	1043.36
An amorphous phase of PLA	Amorphous phase	867.85	869.90	869.85	870.55
The crystalline phase of PLA	Crystalline phase	753.58	754.17	752.38	759.58

Clearly, a distinct band at 1756 cm^{-1}, which corresponds to the C=O stretching vibration in the PLA, appears at the spectrum curve of the SiO_2. The peaks at about 2284.53, 2280.69, 2285.11, and 2281.32 cm-1, belong to the N-C-O stretching of PU. The band at 2276 cm−1 is the most specific band for isocyanate-terminated products and represents the asymmetrical stretching vibrations of an isocyanate group. The appearance of this band confirmed the presence of the reactive isocyanate group in the polymer blends PLA-PU. The peak at 3314.53, 3318.52, 3314.89, and 3318.52 cm-1 belongs to the N-H stretching of PLA-PU. Based on this inference from FT-IR, we understood that PLA-PU-G blend is more suitable as a coating material and PLA-PU-G of optimum concentration could be taken forward for industrial coating applications to replace the existing epoxy and solvent-based toxic coatings.

Contact Angle Test

Contact angle data is important to determine the surface behaviour of the polymers. The hydrophilic surface will offer a good contact angle along with biological species and improve the antimicrobial efficacy of the robust ingredients reported by Jaroslaw Drelich *et. al.* 2011. **Fig. 4** shows interesting data on water contact angle values. The importance of coating with numerous polymeric resins is performed to remove moisture and to improve water impermeability to coated metal surfaces.

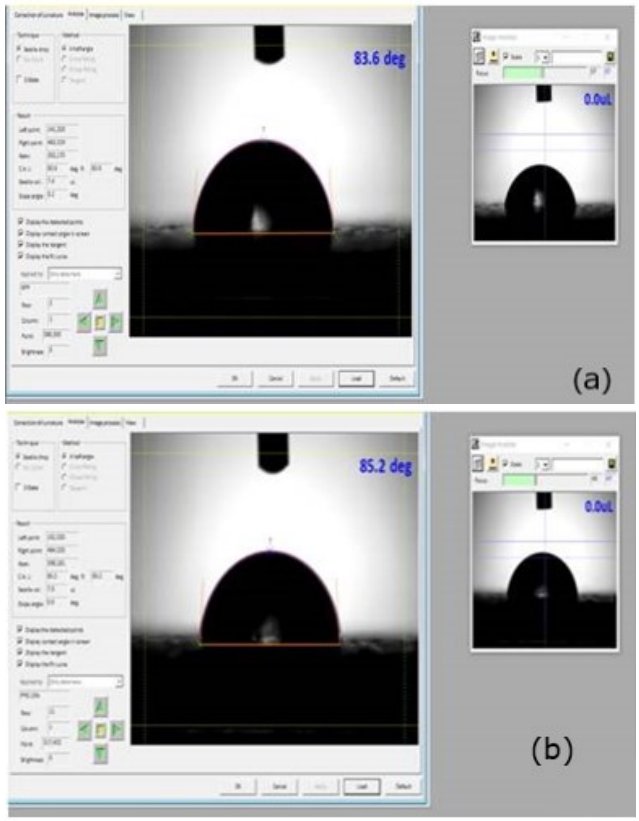

Fig. 4. Contact angle (a) NEAT PLA-PU (b) PLA-PU-G 15%.

Hence, after the surface coating, the standard contacting liquid on the coating surface must increase the contact angle values in comparison to neat PLA-PU (a). As the values of contact angle increase, the hydrophobic nature also increases proportionally. Concerning the Cobb test values, as PLA-PU-G (b), (c), (d) is more or less hydrophobic; the contact angle values complement its nature with an increasing trend. In the case of PLA-PU-G (b), (c), and (d) blend of optimum composition, the contact angle value was maximum. The coating capacity of this PLA-PU-G (b), (c), and (d) polymer homogeneously fills the micro pores of the metal surface. It also increases the surface evenness and reduces its water and air permeability.

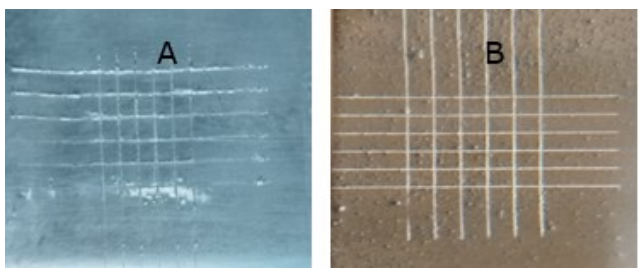

Fig. 5. Surface of crosscut area (A) Neat PU-PLA and (B) PU-PLA/Garnet 15%.

Cross-Cut Adhesion Test

Cross Hatch adhesion test was performed according to the standard ASTM D3359-17. The PLA-PU-G 15% was selected in this case as it exhibited the best results. Adhesion was measured according to ASTM D3359-17 standard in which the scale 5B is the quality of adhesion found in the substrate. To investigate the possible cause of adhesion failure or success of each substrate, the contact angle measurement was performed on all substrates was calculated. Additionally, the surface morphology of each substrate was also studied with a Scanning Electron Microscope.

Scanning Electron Microscope (SEM)

The SEM images of NEAT PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% coated mild steel samples are illustrated in **Fig. 6**. SEM results clearly prove and indicate their excellent coating and adherence with the mild steel surface.

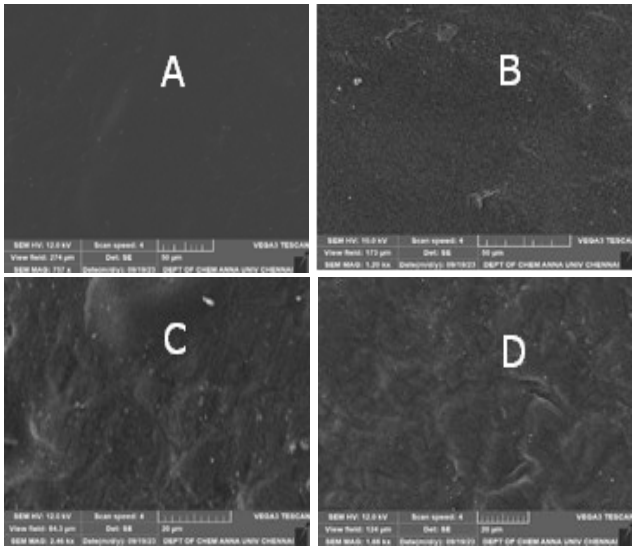

Fig. 6. SEM images of (A) PLA-PU NEAT, (B) PLA-PU-G 5%, (C)PLA-PU-G 10%, (D)PLA-PU-G 15%.

From the images, it is clearly understood that the blend of NEAT PLA-PU(a), PLA-PU-G 5%(b), PLA-PU-G 10%(c), PLA-PU-G 15%(d) filled the pores of the mild steel surface by forming a continuous layer to demonstrate their barrier properties towards air, water, and water vapor, etc. The barrier layer of PLA blends formed over the mild steel surface resists the entry of corrosive species within the environmental conditions and indicates they're useful for industrial corrosion coating applications.

Energy Dispersive X-Ray Analysis (EDS OR EDX)

From the SEM images, the EDX results were obtained. The results revealed that PLA-PU and PLA-PU-G have a rough structure. Besides, they show that PLA was successfully blended with PU and Garnet.

Garnet can be confirmed by the presence of silicon content. **Table 2** shows the values of the elemental content of the PLA-PU-G as measured by the EDAX technique (At. %) and the theoretical calculations from the molecular formula (wt. %).

Table 2. Element weight percentage.

PLA-PU-G Elements (wt%)				
C	O	Al	Si	Fe
84	10	3	3	1

Electrochemical impedance studies

Then EIS data obtained after immersing the samples in 3.5% NaCl solution gives the corrosion performance of coated samples. It is to be noted here that all the samples (5% to 15%) contain one time constant with a big arc in the Nyquist plot. The experimental values of different EIS parameters like Warburg Impedance, Charge transfer resistance (Rct), Pore resistance (pRo), Solution resistance (Rs), and Coating capacitance (Cc) were determined by fitting the data to an equivalent circuit.

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Moreover, impedance values in the low-frequency region remain on the order of 2 million after 28 days of immersion indicating that coating has immense barrier properties. Rct values also remain in the order of 2 million after 28 days of immersion indicating that the coating has excellent non-conduction properties against corrosion current. It is important to propose an ideal electrical circuit model (**Fig. 7**) that interprets the impedance data of the coating being investigated without causing a loss of information pertaining to it.

Based on the observations obtained from all the coating systems of our study, an electrical circuit diagram was accordingly proposed and shown in Fig. 13 to explain their corrosion-resistant behaviour.

R_s - The solution resistance,

R_{cot}-The coating resistance,

R_{ct} -The charge transfer in the coating/substrate interface paralleled with Cdl for the coating/mild steel interface.

Charge transfer resistance (R_{ct}) is the resistance against the process of electron transfer from the electrode to the electrolyte.

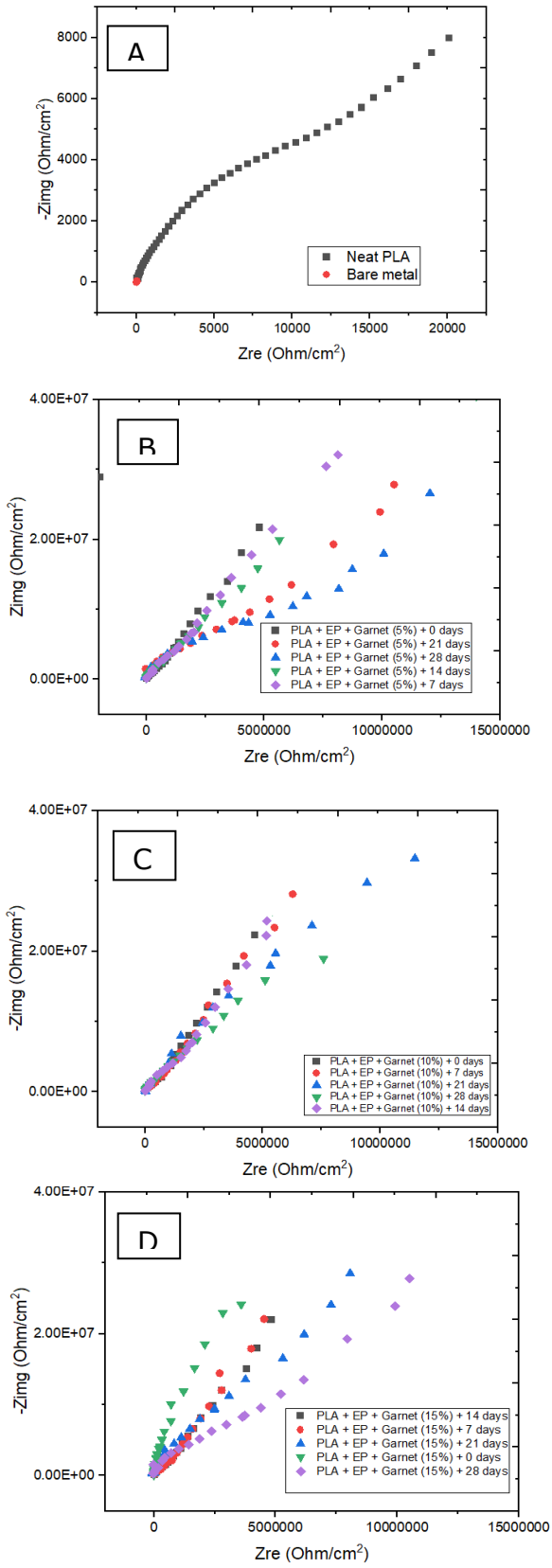

Fig.. 7. Electrochemical Impedance data of Nyquist (A) Neat PLA-PU, Bare Metal, (B) PLA-PU 5% Garnet. (C) PLA-PU 10% Garnet, (D) PLA-PU 15% Garnet.

Potentiodynamic polarisation

The potentiodynamic polarisation curve gives information on corrosion current and corrosion rate. The PE shown in the graph hints the corrosion potential lies more towards the cathodic side with the samples of 28th day. Hence it is clear that the corrosion performance of the specimen is well and good. Plus, the corrosion current lies in nA range again indicating that the sample got better barrier performance.

Salt spray test

A salt spray test was conducted on the Neat, 5%, 10%, and 15% PLA-PU-G coated specimens using a 5-weight percent NaCl solution. Images of the coatings collected at 0 and 1000 hours throughout the approximately 250 hours that the process took place are displayed in **Fig. 8**. The coatings are inspected for blisters and corrosion at these intervals. Upon observation, the tidy PU exhibited susceptibility to corrosion even after 1000 hours of exposure to salt spray. When 5%, 10%, and 15% PLA-PU-G blends were compared, the 10% blend experienced minor blistering on the cut region, while the 5% and 15% PLA-PU-G blends remained intact and showed no signs of corrosion. This shows that the corrosion properties of the PLAPU blend improve as garnet content increases.

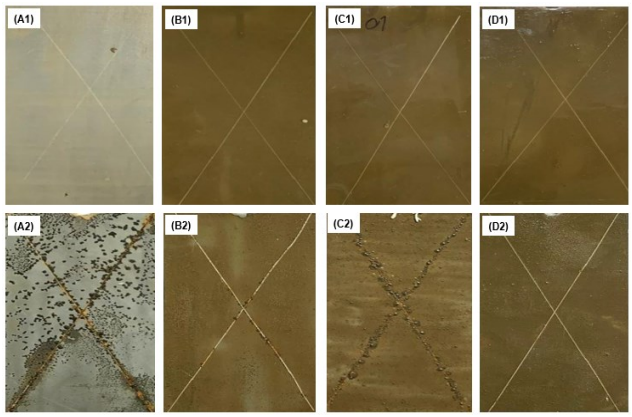

Fig. 8. (A1) (A2), (B1) (B2), (C1) (C2) and (D1) (D2) are the samples of PLA-PU NEAT, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15%, kept in Salt Spray for 0 and 1000 hours, respectively.

Atomic force microscopy (AFM)

In the AFM image of the PLA-PU-G 15% coated sample in **Fig. 9(a)**, the majority of the area is occupied by continuous, flexible soft segments, shown in brown. This occurs due to the smooth coating of PLA-PU. The rare hard segments observed might be due to the presence of garnet particles in the blend. After 1000 hours of salt spray, as seen in **Fig. 9(b)**, osmotic blisters formed on the coated sample. There is a significant increase in hard domains (white) and a decrease in soft segments. The salt spray test accelerated the weathering and aging of the coated sample, which can be assessed by the changes in the sample's gloss.

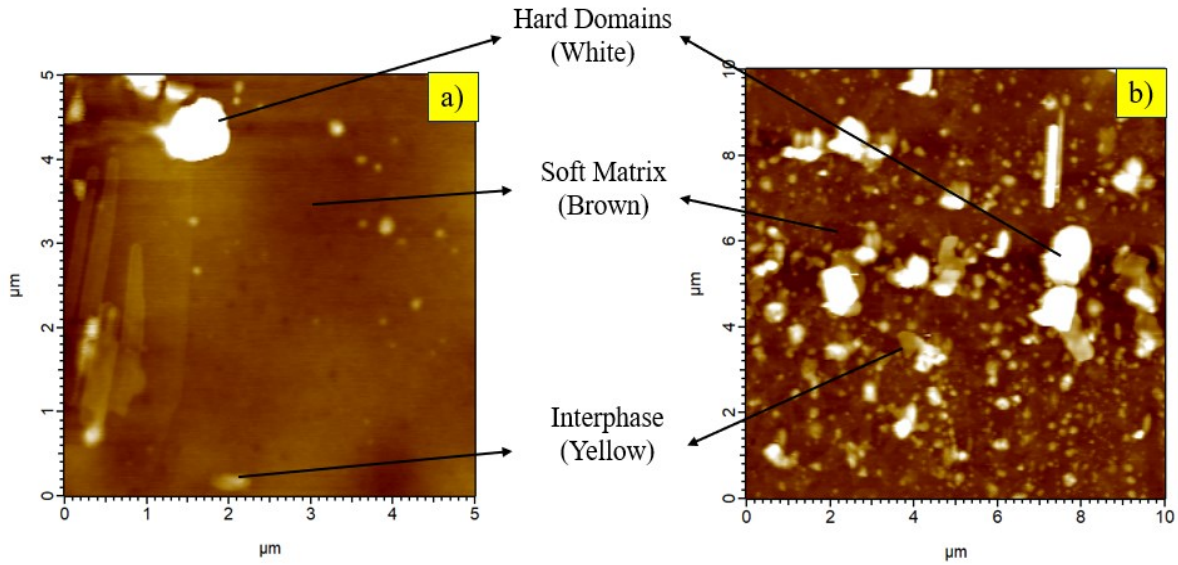

Fig. 9. AFM images for the interphase (yellow), soft segments (brown/yellow), and hard segments (white) for the PLA-PU-G 15%: a) Before salt spray, b) After salt spray for 1000 hours.

Conclusion

The present work functionally attempts to develop a PLA-PU-G bio-based coating material on mild steel for the prevention of corrosion and wear occurring on plates. Bio-based coatings have a greater benefit to the environment as well as for industrial applications. The mechanical blending of 70% PLA with 30% (optimized) was done and it was applied as a coating by brush over mild steel. Furthermore, the PLA-PU coating was loaded with an inorganic garnet waste material to offer improved mechanical, thermal, and barrier properties to the coating and exhibit a good surface finish over the mild steel. FT-IR analysis has ascertained a minimum interaction that occurred between the PLA and PU. The contact angle images of the PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% show a hydrophobic nature against air, water, and other foreign material entering the substrate. From the scratch test, the adhesion strength of polymer blends PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% towards the mild steel substrate was found very good compared to the neat PLA-PU. The SEM images of the PLA-PU, PLA-PU-G 5%, PLA-PU-G 10%, and PLA-PU-G 15% coating showed rough and white spots indicating proper dispersion of garnet in the PU-PLA matrix. The garnet-reinforced PLA-PU matrix having optimum loading of garnet has provided enhanced barrier properties ideally suitable for corrosion and wear resistance than neat PLA. Accordingly, an ideal formulation with respect to this blend will be at 15% garnet as most of the tests show maximum values at this concentration. Hence, we conclude that this study conducted can be used as proof conforming the need and importance of bio-based coating material, which can act as both corrosion and wear resistance material to replace the pollution-causing synthetic coating materials.

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Conflicts of interest

"There are no conflicts to declare".

Keywords: Coating; corrosion; biomaterials; polylactic; polyurethane.

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Rajiv G. completed his M.Tech (Polymer Science and Engineering) from Anna University. Currently proceeding PhD from 2019 in the title of Anticorrosive coatings for marine application using epoxy resin. Worked in Nippon Paint India Pvt ltd. as an R&D – Technical office from 2010 – 2013. In 2011 awarded the best technical presentation on the topic of Matex Gold cost-effective high-performance wall coatings. In 2012 worked on an Indo-Japan project in the title of insect paint.

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Graphical Abstract