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Development of Electroless Ni-P Coatings on Sisal Fiber

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Abstract
Improving the properties of natural fibers can be interesting from a technological point of view. Modifications can result in a high specific strength, electromagnetic interference (EMI) shielding, antistatic brush etc. Plant fiber composites would make them an option for use in components such as rifle stocks and knife handles etc. The greatest challenge would be to ensure that the components are all weather capable, but today several materials exist that have a high moisture tolerance. Moreover with proper surface coating techniques, most natural fibers materials can be toughened up to use as reinforcement. An attempt was made using a proprietary experimental set-up to coat natural fiber with electroless nickel phosphorus (ENi-P), since sisal fiber is a non-conducting material. In this article the surface morphology, ingredients and cross-section images of the modified natural fibers were characterised by a scanning electron microscope equipped with EDAX.

Key words: *composite, mechanical properties, electroless, nickel, sisal, SEM, EDAX.*

Introduction

Electroless coating finds applications in the chemical, nuclear, telecommunications, consumer electronics and computer industries. The first inventors of Ni-P electroless coating were Brenner and Riddel in the year 1946. Electroless plating involves a number of steps carried out in order. The first step is sensitisation, which is followed by activation, and then controlled electron deposition using a chemical reduction reaction with suitable additives for acceleration or retardation of the deposition rate [1, 2].

Sisal fiber is a natural fiber extracted from the leaves of the sisal plant. Sisal is the most commonly produced fiber globally and accounts for the world's largest plant fiber production. The sisal plant produces 200-250 commercially usable leaves during its life- cycle. The major producers of sisal fiber are Brazil, Mexico, China, Tanzania, Kenya and Madagascar. The areas of application of sisal fiber are the automotive industry, in electrical applications, railways, geo-textiles, defense and the packaging industry.

Most of the research on sisal-polymer composites were carried out in the mid-1990s, and sisal fiber was proved successful in composite applications. With this in mind, Kuruvilla et al (1999) made a review of sisal fiber reinforced composites. Sisal-polymer composites were also fabricated and their mechanical properties were studied by most of the researchers concerned [4,5]. The various coating procedures adopted for synthetic fibers created an interest in the coating of sisal fibers in order to improve the mechanical properties of sisal-polymer composites [6,7]. The oxidation of carbon and metal coated carbon fibers was introduced in 1970 [8]. In this continuation, electrophoretic deposition (EPD), followed by pressureless sintering, to produce dense, defect-minimised, carbon-fiber-reinforced borosilicate-glass-matrix composites with a nickel interface were proposed as a platform for coating carbon fibers for composite applications. One researcher reported the effect of silver coating and the size of fibre on the electrical properties of sisal fibre-reinforced epoxy composites [9].

To shield and limit against electromagnetic interference (EMI) and electrostatic discharge (ESD), conductive polymer composites started replacing coated materials for various shielding applications in the electrical and electronics industries, especially for electronic household appliances. This trend has been driven mainly because of the better characteristics of these polymers in terms of electrostatic discharge (ESD), shielding from EMI, thermal expansion, density, corrosion and oxidation resistance properties [10,11].

In order to explore the possibility of using modified sisal fibers as reinforcements in composites, an attempt was made to develop and characterise electroless nickel phosphorus (ENi-P) coatings on sisal fiber in the present investigation.

Materials & methods

The interface between the matrix and reinforcement is critical and can affect the properties of composites manufactured significantly. If the interface is not modified properly, it can lead to the degradation of the properties of composites. To develop better adhesion and interfacial bond between sisal fiber and epoxy matrix, the electroless coating process was used to deposit Ni-P onto sisal fiber in the present investigation. The electroless coating of the fiber, which is a simple, low-cost and easy- to- use process has

been successfully applied to prevent undesirable interfacial reactions and promote wettability through the increased overall surface energy of the reinforcement [12]. Due to the lighter weight of Ni and relatively stable coating quality, plating Ni on the surface of sisal fibers has been widely used [1]. Sisal fiber (Sri Balaji Coir Industries, Dharmapuri, Tamilnadu, India) in the chopped form was used in this study. The electroless Ni-P deposition process was undertaken to deposit Ni-P onto the sisal fiber. The process constitutes the following sequence of steps: cleaning, rinsing, drying, sensitising, activation and metallisation.

Pre-cleaning is done for the fibers before electroless nickel deposition so that the properties required are obtained on the surface of the fiber. The fibers are precleaned in acetone followed by a methanol wash to remove the contaminants present in it. The alkali treatment process in hot sodium hydroxide (NaOH) solution removed lignin, hemi cellulose and other soluble compounds on the surface of the fiber. The alkali treatment imparts improved mechanical properties by the removal of impurities on the fibers. Generally the level of interfacial adhesion increases by use of treated fibers in the processing of fiber reinforced composites.

Sensitization is a process of catalysing the nonconductive substrate, which enables the fiber surface to act as a catalyst for the deposition of nickel. In the case of sensitization, the fibers are subjected to a surface sensitizer (Tin II chloride 0.1g/l) for 15 min. During the process the fibers absorbs the Tin II chloride ions, which acts as catalyst for Ni deposition. Also activation is carried out for activating the fiber surface for electroless nickel deposition. In activation, the nucleation effect is performed on the sensitized surface in acidic palladium chloride solution (0.01 g/l).

The pH of the bath was adjusted to 8.0±1.0 by the addition of the required amount of ammonia solution as and when required. The bath temperature was maintained at 80±1°C. The electrolytic bath was heated by an electrically heated water bath whose temperature was maintained by a proportional integral derivative (PID) controller. The bath composition is given in the ***Table 1***. Based on the literature, the composition of the bath is selected. The other compositions are also tried and the coating obtained by these compositions only. The activated sisal fibers are immersed in the bath and electroless coating is performed for various coating durations, such as 5, 20, 25 and 60 minutes. As the coating duration increased, the Ni-P coating on the fibers increased in thickness. The samples prepared were then characterised by an SEM, JSM 6390A, JEOL, Japan, fitted with an EDS analyser.

Results and discussion

SEM images of coated and uncoated sisal fibers

The cross section of the uncoated single sisal fiber is shown in ***Figure 1***. The estimated diameter of the fiber from the SEM is ~100 μm. An SEM image was taken at

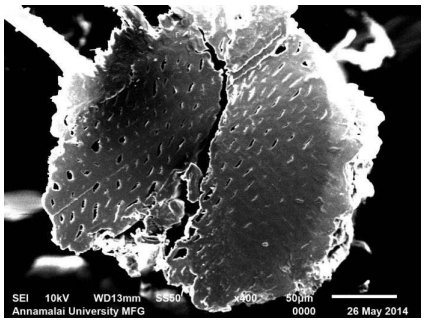

Figure 1. *Cross section of the uncoated sisal fiber.*

Table 1. *Bath composition.*

Element	Composition, g/l
Nickel sulfate	30
Sodium hypophosphate	32
Tri-sodium citrate	40
Ammonium chloride	50

400 X magnification and the layer of sisal fiber was observed. The main content of natural fiber is cellulose micro-fibrils which are bonded by amorphous materials called hemicelluloses. Lignin acts as a coupling agent, which helps to improve the stiffness of the cellulose/hemicellulose composite. SEM images of cross-sectional and surface views of sisal fibers coated with ENi-P for 5, 20, 25 and 60 minutes under the same plating conditions are shown in ***Figures 2-5***. With an increase in the coating duration, the thickness of the coating was found to increase marginally from 1.42-8.23 μm. There is no appreciable change in the coating thickness beyond 60 minutes duration.

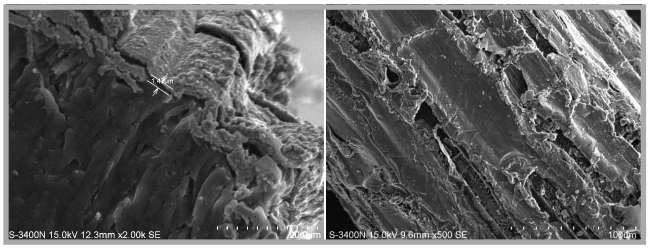

Figure 2. *Sisal fiber coated with Ni-P (5 mins).*

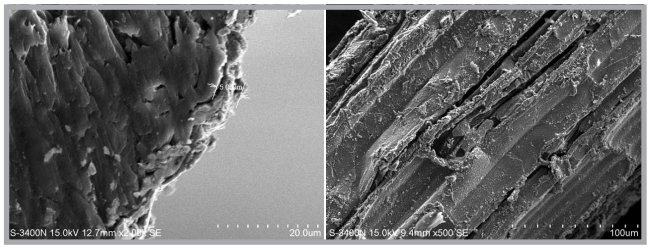

Figure 3. *Sisal fiber coated with Ni-P (20 mins).*

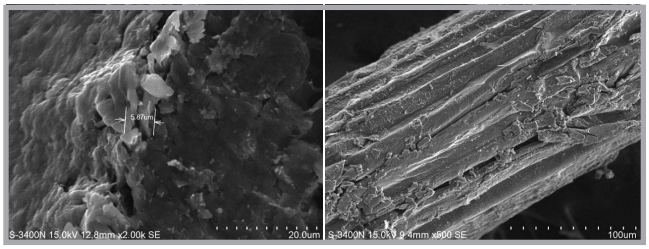

Figure 4. *Sisal fiber coated with Ni-P (25 mins).*

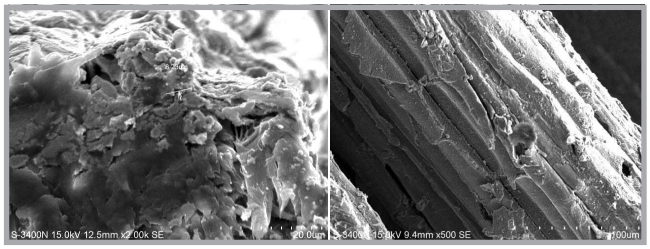

Figure 5. *Sisal fiber coated with Ni-P (60 mins).*

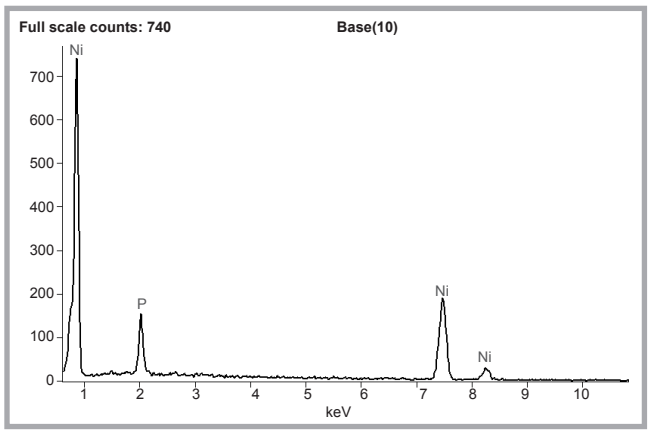

Figure 6. *EDAX spectrum of Ni-P coating on sisal fiber for 5 mins.*

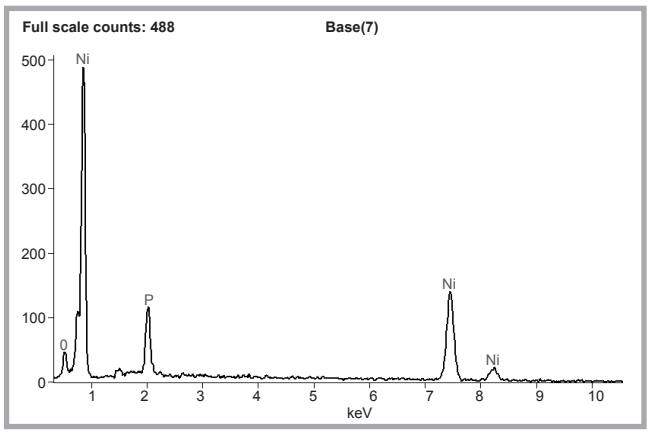

Figure 7. *EDAX spectrum of Ni-P coating on sisal fiber for 20 mins.*

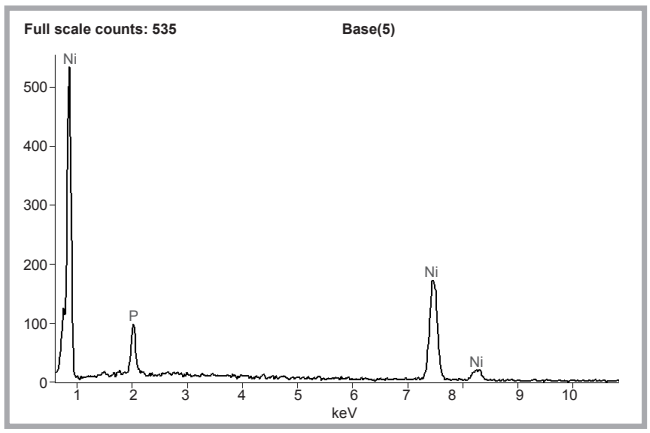

Figure 8. *EDAX spectrum of Ni-P coating on sisal fiber for 25 mins.*

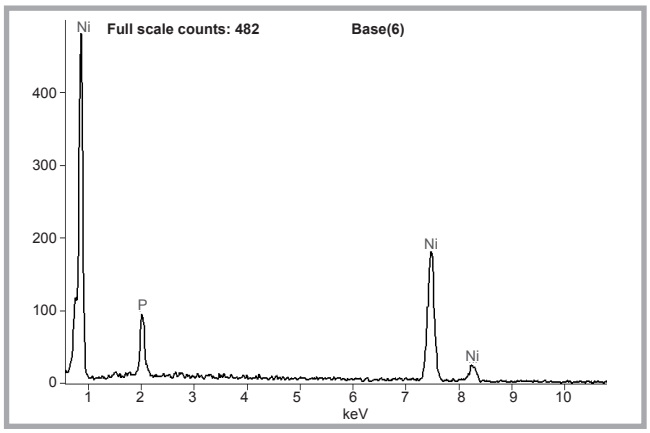

Figure 9. *EDAX spectrum of Ni-P coating on sisal fiber for 60 mins.*

Sisal fibers are used to fabricate polymer composites using epoxy, polyester, vinyl ester and other types of resins. A low value of mechanical properties is obtained as compared with synthetic fiber reinforced polymer composites due to the poor interfacial bonding between the fiber and matrix. In order to improve the adhesion and interlaminar bond between the sisal fiber and matrix, fiber coating was suggested in the present investigation.

From ***Figures 2-5***, it was observed that the surface seems to be rough and shows strong adherence of the coating onto the fiber. The surface roughness of the coated fibers indicates that if these fibers are used as reinforcements in polymer matrix composites, that would develop strong interfacial bonding between the matrix and reinforcement.

EDAX spectrum of coated sisal fibers

Energy dispersive X-ray analysis identifies the presence of various materials in the fiber coating. ***Table 2*** lists the various materials and their oxides present in the coating. The spectrum processing option of oxygen by stoichiometry was used for EDAX analysis.

EDAX analysis (***Figures 6-9***) of the coated fiber reveals the presence of Ni and P in the coatings. It is observed that as the coating duration increases, preferential deposition of Ni takes place with respect to P. The presence of nickel with maximum weight percentages of 91.90, 93.13, 93.76 and 93.78 were observed for 5, 10, 25 and 60 minutes of coating, respectively. The presence of phosphorous with maximum weight percentages of 8.10, 6.87, 6.24 and 6.22 were observed for 5, 10, 25 and 60 minutes of coating, respectively.

Table 2. *Composition of coating as per EDAX.*

Coating time, min	P K		Ni K	
	Weight, %	Atom, %	Weight, %	Atom, %
5	8.10	14.31	91.90	85.69
10	6.87	12.26	93.13	87.74
25	6.24	11.21	93.76	88.79
60	6.22	11.17	93.78	88.83

Fabrication and testing of sisal-epoxy composites

Liquid epoxy resin and hardener (HY951) at a 10:1 ratio was placed in a plastic container and mixed thoroughly for 20 min. Uncoated or coated sisal fiber was added, mixed thoroughly, and poured into a plastic mould. A stainless steel mould of a size of 300 × 300 × 3 mm was used for composite plate fabrication using the compression molding process. The samples were cut from the composite plate according to ASTM D 638-10. The tensile behavior of the sisal-epoxy composites was measured using a Dual

Table 3. *Properties of uncoated and coated sisal fiber reinforced epoxy composites.*

Si. No	Fiber length, mm	Fiber loading, %	Uncoated sisal-epoxy composites			Coated sisal-epoxy composites		
			Tensile strength, MPa	Flexural strength, MPa	Impact strength, kJ/m^2	Tensile strength, MPa	Flexural strength, MPa	Impact strength, kJ/m^2
1	10	30	20.4	30.2	28.5	28.2	39.0	38.8
2	25	30	24.4	33.5	33.2	34.6	44.2	44.8
3	40	30	31	45.2	41.3	43.3	59.2	55.8
4	55	30	30.9	51.5	43.1	43.9	68.9	58.5
5	70	30	24.2	40.3	34.7	33.4	53.7	46.2

Column Table Top Universal Testing Machine (Tinius Olsen H10K). The length, width and thickness of each sample in tensile testing were 165, 25 and 3 mm, respectively. A three point flexural test was conducted for specimen dimensions of 125 mm × 12.5 mm × 3 mm according to ASTM D 790-10. The izod impact test was carried out using a Tinius Olsen (Model: 104) Impact Tester as per Standard ASTM D 256-10 and a sample size of 62.5 × 12.5 × 3 mm was used.

The values of tensile, flexural and impact behaviors of uncoated and coated sisal-epoxy composites observed are given in ***Table 3***. Fabrication conditions such as fiber length and fiber loading were selected as per a literature survey for short sisal fibers. The uncoated sisal-epoxy composites exhibited better values of tensile, flexural and impact strength of 31 MPa, 51.5 MPa and 43.1 kJ/m^2, respectively, for an uncoated sisal fiber length of 55 mm and fiber loading of 30% by weight. The coated sisal-epoxy composites exhibited better values of tensile, flexural and impact strength of 43.9 MPa, 68.9 MPa and 58.5 kJ/m^2, respectively, for the coated sisal fiber length of 55 mm and fiber loading of 30% by weight.

Applications of coated sisal fiber reinforced epoxy composites

The use of sisal fiber reinforced epoxy composites in engineering applications such as the automobiles, construction and other industries are proposed because of its low cost, acceptable strength, low weight and ease of degradation. The coating of sisal fiber for improvement in the mechanical behaviors of sisal-epoxy composites may spread the application of these composites in the following areas.

- Sports goods applications (child toys, table tennis racquets)
- Automobile applications (rear flaps, mud guards, engine guards)
- Structural applications (door panels, name plates)
- House hold applications (knife holders, trays)
- Electrical applications (switch boards)

Conclusions

A continuous thick Ni-P alloy layer was coated on the surface of sisal fibers by electroless plating. The coating thickness ranged between 1.42-8.23 μm, and a maximum coating thickness of 8.23 μm was observed for a coating duration of 60 minutes. There is no appreciable change in the coating thickness beyond 60 minutes of the coating duration. The SEM images reveal dense, uniformly distributed nickel deposition on the sisal fiber. The EDAX spectrum revealed that an increase in nickel % with respect to the coating duration clearly indicates the preferential deposition of nickel on sisal fiber. Sisal – epoxy composites were fabricated using uncoated and coated sisal fibers. A 40%, 32% and 35% improvement in tensile, flexural and impact behaviors, respectively, were observed for coated sisal fibers in epoxy composites.

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