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INVESTIGATION OF Ni-P COATED BAMBOO FIBRE/NANO-TiO_2 POLYESTER MATRIX COMPOSITE PROPERTIES

RAZISKAVA LASTNOSTI Ni-P OPLAŠČENIMI BAMBUSOVIMI VLAKNI IN NANO DELCI TiO_2

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Natural-fibre-reinforced polymer composites are most commonly employed in the automotive, aerospace and structural industries. This research focuses on the synthesis and analysis of the dynamic properties of a bamboo fibre used with a polyester matrix in a nanocomposite produced with titanium oxide nanoparticles. Bamboo-fibre samples were prepared using the electroless plating method in both their uncoated and nickel-phosphorus-coated states. Bamboo-fibre contents of (4.5, 9, 13.5 and 18) *w*/% and nanoparticles of (0.5, 1, 1.5 and 2) *w*/% with and without the coating were examined for these qualities. Coated bamboo fibres were mixed with 13.5 *w*/% and 1.5 *w*/% of titanium oxide. It was concluded that dynamic and thermomechanical properties of the polyester matrix reinforced with bamboo fibres coated with nanoparticles allow excellent bonding as compared to the nanocomposite of the polyester matrix reinforced with uncoated bamboo fibres.

Keywords: bamboo fibre, nano-TiO_2, dynamic and thermomechanical properties, nickel-phosphorus

Polimerni kompoziti ojačani z naravnimi vlakni so danes razvojni trend in zato se zelo pogosto uporabljajo v avtomobilski in letalski industriji, kot tudi v strojništvu in gradbeništvu. V raziskavi predstavljeni v članku, so se avtorji osredotočili na sintezo in analizo dinamičnih in termo mehanskih lastnosti z bambusovimi vlakni in nano delci titanovega oksida (TiO_2) ojačanega polimernega kompozita s poliestersko matrico. Vzorce bambusovih vlaken so pripravili z uporabo metode elektrolitskega platiranja in uporabo dodatnega oplaščenja kompozitnih vlaken s spojino oziroma zlitino niklja in fosforja (Ni-P). V izdelanih vzorcih kompozitov je bila vsebnost bambusovih vlaken in nano delcev TiO_2 različna (4,5, 9, 13,5 in 18) *w*/% vlaken ter 0,5, 1, 1,5 in 2) *w*/% nano delcev). Vse izdelane oplaščene in neoplaščene kompozite so kakovostno ovrednotili. Najboljše lastnosti je imel kompozit s 13,5 *w*/% oplaščenih bambusovih vlaken in 1,5 *w*/% nano delcev TiO_2. Na osnovi raziskave so avtorji zaključili, da imajo izdelani kompoziti s poliestersko matrico ojačani z oplaščenimi bambusovimi vlakni boljše termo mehanske in dinamične lastnosti z odlično vezavno trdnostjo v primerjavi s kompoziti, ki vsebujejo neoplaščena vlakna.

Ključne besede: bambusova vlakna, nano titanov oksid, dinamične in termo mehanske lastnosti, nikelj-fosfor

I INTRODUCTION

Composites are made by physically combining/mixing two or more elements to create a new component with properties that are superior to those of individual components.[1–3] Natural fibre based polymer composites are nowadays most common in the industrial field; hence, they are used in a wide range of applications, exhibiting better properties. They are bio-degradable, renewable, inexpensive, eco-friendly and recyclable.[4,5] Natural fibres can replace synthetic fibres such as glass, carbon nanotubes and graphite due to their low cost, smaller energy consumption and better properties such as mechanical and dynamic mechanical properties, bio-degradability and damping capacity. Reinforcement is used when natural fibres combined with different matrix materials are studied. In the automobile field, 20–25 % of such polymer composites is used and 50 % of them is widely used in the construction field.[6–9] Natural-fibre-reinforced polymer-matrix composites were investigated in several research works to improve the composites in this way.[10–12] Nano-filler materials maximise interfacial adherence and uniform distribution.[13,14] Titanium oxide (TiO_2) nanoparticle has been the most studied metal-oxide nanoparticle[15,16] because of its one-of-a-kind mix of properties, including thermal stability, increased oxygen storage capacity and corrosion resistance.[17–20]

The mechanical properties of natural-fibre reinforcement in bio-composites are not significantly improved even after a chemical treatment process. As a result, despite the fact that natural fibre/plastic composites have been commercialised, their applicability in many industries is limited. This has motivated a lot of researchers to improve the thermal and mechanical properties of composites. The best way to increase the mechanical performance of natural-fibre-based composites is to combine nano-fillers like metal nanoparticles, nanoclay, and carbon nanotubes (CNT) with polymer resins such polyesters, epoxy and vinyl ester resins.[21–23] The systematised autocatalytic reduction of metallic salt[24] and electroless deposition/coating of abrasive particles are becoming increasingly common as a particle treatment method.[25] Several researchers have enhanced material properties

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using these coating techniques.[26] To improve the mechanical behaviour of polymer composites, an electroless technique was performed to achieve a particle reinforcement. The deposition of copper phosphorus on the silicon carbide particles was used in an electroless process.[27] The friction and wear behaviour of nickel-phosphorus coating on silicon particles was tested using the electroless deposition approach at an increased temperature, and it was established that the applied layer was robust.[28]

In the present study, bamboo fibres with and without a coat of nickel-phosphorous were added to a polyester matrix material and a small amount of titanium oxide nanoparticles were used. The crucial parameter in this investigation was the change in the nanoparticle and fibre content. The distribution of nanoparticles and the fracture surface of the nanocomposite were studied using high-resolution scanning electron micrographs (FESEM). The dynamic and viscoelastic properties of the polymer-matrix composite reinforced with bamboo fibres and nanoparticles were investigated.

2 EXPERIMENTAL PART

2.1 Materials

The polyester resin was purchased from a local polymer vendor, Kamatchi Polymer Ltd., Puducherry, India, while methyl-ethyl-ketone peroxide as the catalyst and accelerator used for cobalt was obtained from another local polymer vendor. Bamboo fibres were procured from Go-Green India ltd., Chennai (India). Titanium oxide nanoparticles were procured from Subra Scientific Ltd., Pondicherry (Sigma Aldrich) at a density of 6.4 g/mL at 28 °C with 99 % purity and a nanoparticles size of < 40 nm.

2.2 Electroless plating process

Ni-P is applied on a bamboo-fibre substrate using an electroless plating process. Bamboo fibres must be cleansed and pre-treated before this operation to achieve consistent coating. The bamboo fibre is an insulating substrate; hence, it should be pre-treated with a tin II chloride and palladium chloride solution as the catalyst, activating the bamboo fibre in 20 min. Electroless coating consists of a chemical solution such as nickel sulphate as the source of nickel, sodium hypophosphite, tri sodium citrate and ammonium chloride. The pH value is maintained in the bath during this process, using ammonium chloride. The temperature of chemical solution in the bath is controlled by a PID set-up. **Figure 1** shows the experimental set-up of the electroless-bath hot plate. The pH value and bath temperature are maintained at (80 ± 2) °C and 7–8 pH so that a uniform coating of the fibres is obtained. Sodium hypophosphate acts as the reducing agent, releasing free electrons during the electroless process. The free electrons react with the nickel source, nickel sulphate in the electrolyte bath, converting them into nickel particles. The excess electrons liberated from the electrolyte solution in the bath deoxidize the phosphorus ions in the reducing agent, resulting in phosphorus particles that are deposited over the entire surface of the fibres along with nickel. The nickel and phosphorus particles are placed over the fibre substrate after the palladium chloride solution is activated in the electroless bath.

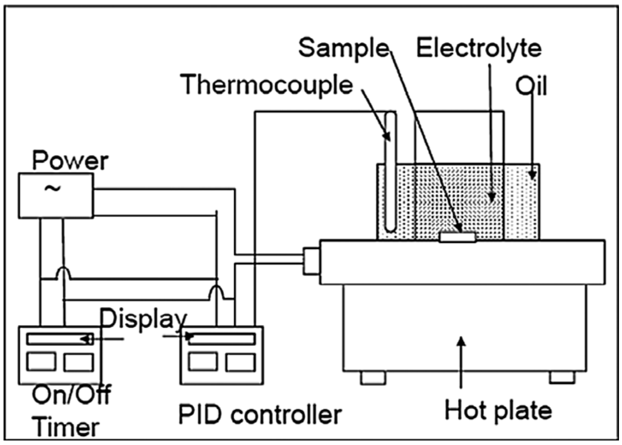

Figure 1: Experimental electroless plating set-up

Table 1: Prepared polyester nanocomposite specimens with various compositions

Sp. No.	Specimen ID	Composition of the material			
		Polyester resin (*w*/%)	Without Ni-P coated bamboo fiber (*w*/%)	With Ni-P coated bamboo fiber (*w*/%)	TiO_2 nanoparticle (*w*/%)
1	PUC-1	95	4.5	–	0.5
2	PUC-2	90	9	–	1
3	PUC-3	85	13.5	–	1.5
4	PUC-4	80	18	–	2
5	PC-1	95	–	4.5	0.5
6	PC-2	90	–	9	1
7	PC-3	85	–	13.5	1.5
8	PC-4	80	–	18	2

PUC – polyester uncoated composite, PC – polyester coated composite

2.3 Preparation of the composite material

The polyester nanocomposite is reinforced with bamboo fibres using the compression moulding method. The polyester composite samples are prepared without coated bamboo fibres and with nickel-phosphorous coated bamboo fibres with standard sizes of (250 × 250 × 3) mm in length, width and thickness of the specimen. The polyester resin matrix is weighed in a weighing machine; 250 g of it is placed in a 500 mL glass beaker; it is cleaned; then 4.5–18 *w*/% of bamboo fibre is added in intervals, each time in the amount of 4.5 *w*/% . Then (0.5, 1, 1.5 and 2) *w*/% of titanium oxide nanopowder is placed in the 100 mL beaker. The bamboo fibres with and without the coating are placed over the cleaned mould surface and then the nano titanium oxide powder is poured into the polyester resin. A mechanical stirrer is used to stir thoroughly both the resin and powder for 20 min. Thereafter, 0.2 mL of MEKP, as the catalyst, is added with a syringe into the polyester resin and also 0.1 mL of cobalt, as the accelerator, is added to the polyester resin with a disposal syringe. Both the catalyst and accelerator are stirred well for 2 min. The mould surface is cleaned and white wax, as the releasing agent, is applied. The prepared composite mixture is poured into the fabricated steel mould, coated with white wax as the releasing agent for different samples and left to be cured for 12 h at an applied pressure of 2.5 MPa at room temperature. After that the sample is removed from the steel mould. Eight different samples are prepared, having weight fractions of bamboo fibre of (4.5, 9, 13.5 and 18) *w*/% and additions of nano-TiO_2 of (0.5, 1, 1.5 and 2) *w*/%; samples can be without the coated fibre or with the nickel phosphorous coating. The fibre/polyester matrix nanocomposite is thus developed. The prepared eight specimens with different compositions are shown in **Table 1**.

2.4 Synthesis of TiO_2 nanoparticles

TiO_2 nanoparticles are synthesized using a slightly modified sol–gel process that was developed earlier for the synthesis of molecularly imprinted titania. Briefly, a solution comprised of absolute ethanol (40 mL) and deionized water (5 mL) is placed in a 100 mL two-neck round-bottom flask equipped with a septum, condenser and magnetic bar. The solution is warmed gently, and $TiCl_4$ (0.6 mL, 0.05 M) is injected into the solution through the septum. The mixture is stirred continuously for 1 h at 60 °C. Afterwards, a dilute solution of NH_4OH (0.05 M) is slowly dropped through the septum to maintain a pH of 9. The mixture is subsequently aged overnight at 60 °C with consistent magnetic stirring. The product is heated under vacuum at 100 °C to yield white TiO_2 powder. Later, this powder is subjected to different thermal and chemical treatments for the synthesis of crystalline or functional TiO_2 nanoparticles. **Figure 2** demonstrates the sol–gel process for the synthesis of TiO_2 nanoparticles.

3 RESULTS AND DISCUSSION

3.1 Characterization

The surface characteristics of the uncoated bamboo fibre and Ni-P coated bamboo fibre are shown in **Figure 3**. The surface of the bamboo fibre has a satiny appearance and clean texture, as shown in **Figure 3a**. However, there are many impurities on the surface, making the plating process more time-consuming. Hence, methanol, acetone and NaOH solutions are utilised to remove the impurities from the bamboo-fibre substrate. The bamboo-fibre surface is activated with palladium chloride, ensuring a better coating on the entire surface. Initially, the bamboo fibre is coated with Ni-P using the bath composition for plating mild steel, described by Elansezhian et al.[29] and presented in **Table 2**. This concentration is used to test the electroless Ni-P coating on the bamboo fibre. As an electroless bath decomposes in a very short period without a proper uniform coating over the surface, the coating of bamboo fibre (BF) using this bath composition is extremely challenging. As a result of the chemical concentrations, operating temperature and the potential of hydrogen (pH) values for the electroless bath are adjusted with a proper coating on the surface based on trial and error.

Finally, the suitable bath composition for proper BF plating is determined, as shown in **Table 3**. A field emission scanning electron microscope (FESEM) image of the bamboo fibre plated with Ni-P is seen in **Figure 3b**

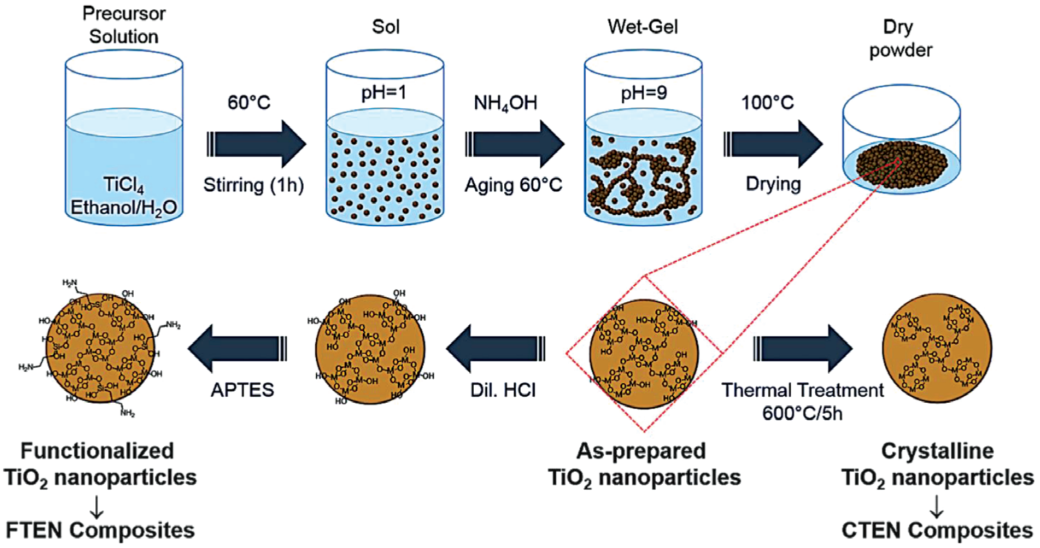

Figure 2: Schematic representation of the synthesis and functionalization of TiO_2 nanoparticles

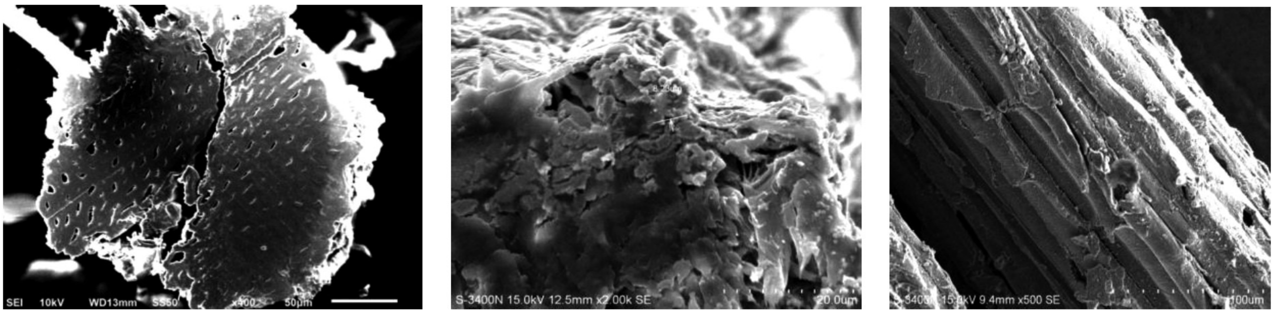

Figure 3: FESEM images: a) neat bamboo fibre without the coating, b) and c) Ni-P coated fibre at 200×, c) Ni-P coated fibre at 500×

at a 200× magnification and another image at a 500× magnifications is shown in **Figure 3c**. The Ni-P deposition over the BF is achieved with a uniform, thick and unbroken structure, as illustrated in **Figure 3b**. The Ni-P particles on the substrate are silvery grey. In addition, a considerable number of Ni-P particles are evenly distributed across the entire surface of the BF and are securely bonded to it, as seen in **Figure 3b**.The structure of the Ni-P particles is clearly shown to be oblate, as seen in **Figure 3c**.

Table 2: Electrolyte-bath solution components

Chemical elements in the EN bath	Bath components in EN (g/L)
Nickel sulphate as the nickel source	32
Sodium hypophosphite as the reducing agent	45
Tri-sodium citrate as the stabilizer	20
Ammonium chloride as the complex agent	40

Table 3: Electrolyte bath solution for proper coating determined on the basis of **Table 2**

Chemical elements in the EN bath	Bath components in EN (g/L)
Nickel sulphate as the nickel source	45
Sodium hypophosphite as the reducing agent	50
Tri-sodium citrate as the stabilizer	40
Ammonium chloride as the complex agent	55

3.2 Morphological properties of the composite

The TiO_2 nanoparticles distributed into the bamboo fibre-reinforced matrix material are shown in **Figure 4a**. This figure shows a FESEM image with a varied nanoparticle content that correlates well with the mechanical tests. When compared to the uncoated composite sample, **Figure 4a** clearly shows that the reduction in the rough lines suggests that the nanoparticles are spread equally within the fibre-reinforced matrix. The pulled-out fibre and the surrounding matrix are separated by a large void in **Figure 4b**. This shows that the neat uncoated composite has poor interfacial adhesion. **Figure 4c** shows the components that are adhered effectively, where the nanoparticles stimulate an increase in the specific surface area to improve interfacial bonding. Clumps arise when the amount of nanoparticles added exceeds a particular threshold. This is due to the agglomerates that become weak areas when loaded. **Figure 3d** clearly shows the agglomeration of the nanoparticles. These findings are consistent with those obtained with tensile testing.

3.3 EDAX analysis on the bamboo fibre

EDAX spectroscopy of the Ni-P plating of the BF is shown in **Figure 4** where we can clearly see a set of strong peaks on the curve indicating the deposition of Ni-P particles on the substrate. According to the EDAX analysis of Ni-P/BF, the concentration of the Ni-P particles over the bamboo-fibre substrate includes about 91.36 *w*/% of nickel and 8.53 *w*/% of phosphorus. The FESEM image and EDAX spectrum indicate the presence of the Ni-P particles, effectively coating the entire surface of the bamboo-fibre substrate after the electroless coating technique has been successfully used. Daramola et al.[30] obtained a similar outcome for long-bamboo-fibre bundle/PLA composites.

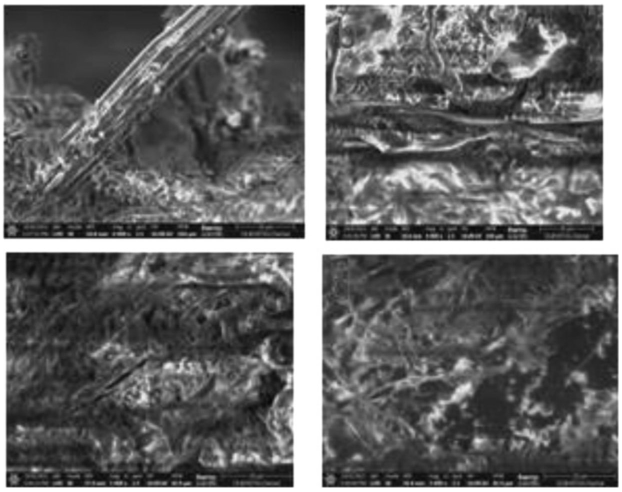

Figure 4: FESEM images of the polymer composite with the Ni-P coating and nanoparticles: a) 0.5 *w*/%, b) 1 *w*/%, c) 1.5 *w*/%, d) 2 *w*/% of nano-TiO_2

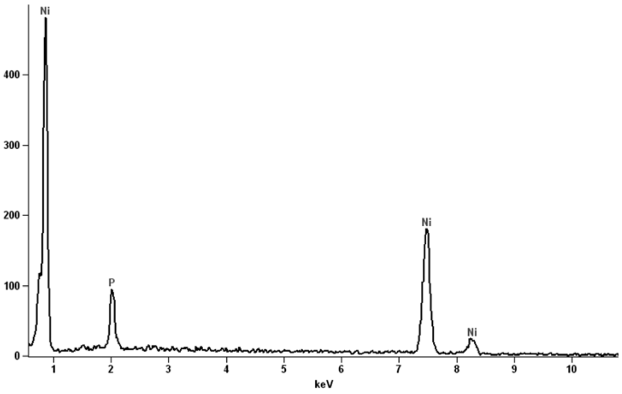

Figure 5: EDAX spectrum for the Ni-P coated bamboo fibre

3.4 Thermogravimetric analysis (TGA)

The thermograms of various hybrid bamboo-fibre composites are clearly shown in **Figure 6**. A thermogravimetric analysis was carried out to evaluate the thermal stability of the PMBF composites at high temperatures. The results show that when the Ni-P coated BF is utilised as reinforcement, the thermal stability of the polyester composite is increased. It also indicates that a higher concentration of TiO_2 nanoparticles, as the reinforcement in the polyester composite, improves the thermal stability, which is confirmed by the experiment result.[30] During the thermal investigation of the polyester composite material, three different phases of degradation are identified, as shown in **Figure 6**. The beginning of decomposition is very slow due to the presence of a small amount of moisture that evaporates, and it lasts up to 300 °C. In the thermogravimetric testing, this is referred to as the initial stage of decomposition. During the second stage, the hybrid composite degrades rapidly, with over 85 % of the composite material degraded in the case of the coated BF-reinforced composite. In this stage of the coated bamboo fibre composite, the starting and finishing temperatures are 300 °C and 425 °C, respectively. If the Ni-P coated BF is utilised as the reinforcement, the starting temperature tends to rise, and this trend continues as the concentrations of the TiO_2 nanoparticles and Ni-P/BF reinforcement increase. The end temperature of this stage also increases significantly, from 425 °C to around 485 °C for 1.5 *w*/% of TiO_2 nanoparticle and 13.5 *w*/% of Ni-P/BF. This happens due to increased adhesion force and better interfacial bonding between the added nanoparticles with the matrix material and the nickel phosphorus-coated BF. As a result, the temperature at which the degradation occurs rises.[31–33] In this stage, the temperature is high because the composite material is hardened.[34,35] The residual stage is the last step. For the polyester hybrid composite containing 1.5 *w*/% of nanoparticles, a maximum residue of 60 % is left over when the nanoparticles are added to the composite and the temperature resistance increases.

3.5 Dynamic mechanical analysis

For polymer composites, the dynamic mechanical analysis (DMA) is increasingly being utilised to analyse the mechanical properties of a composite material subjected to dynamic loading conditions. It is also utilised to investigate the polymer chain mobility and interfacial bonding between the polyester matrix, TiO_2 nanoparticles and coated or uncoated bamboo fibres in composites. The dynamic mechanical behaviour of a polyester hybrid nanocomposite depends on different factors such as filler concentration, fibre loading, resin wettability, reinforcing material orientation and matrix/reinforcement interfacial area.

3.6 Storage modulus

The storage modulus is the most crucial parameter for determining the load-carrying capacity of polymer composites. **Figure 7** displays the storage modulus of the

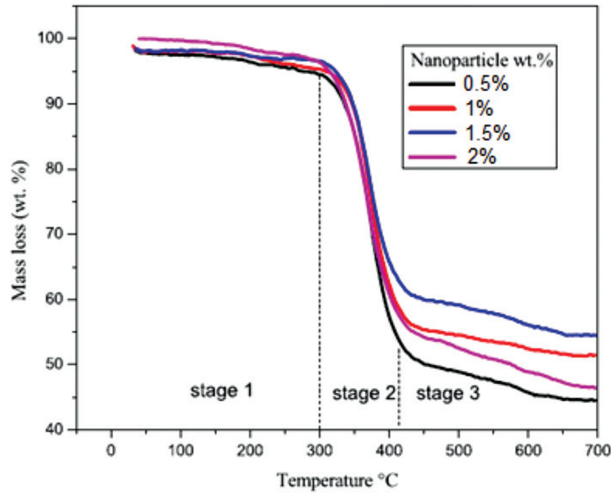

Figure 6: Temperature vs. mass loss (*w*/%) plot of the polyester/Ni-P BF nanocomposites

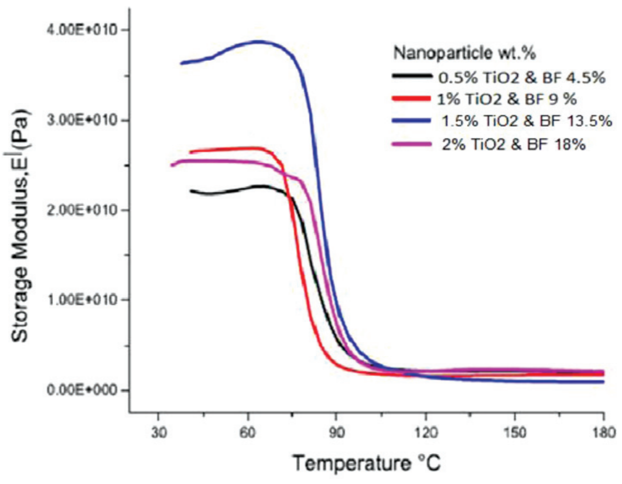

Figure 7: Storage modulus vs. temperature plot for the polyester nanocomposites

composites with different compositions at room temperature. The value of storage modulus E1 increases when TiO_2 nanoparticles are added. For the polyester composite with 1.5 *w*/% of nanoparticles the maximum storage modulus is determined to be 80 % of the matrix material. This is due to an increase in the stiffness of the polyester composite and a wide area of interfacial interactions created by adding TiO_2 nanoparticles. The nanoparticles reduce the mobility of the polyester matrix, leading to an increased storage modulus.[36,37] This is also most likely due to the increased matrix stiffness provided by the support of Ni-P/BF, allowing a better stress transfer at the matrix/bamboo fibre interface. The tensile strength of the sample of the coated composite with 1.5 *w*/% of nanoparticles has a higher storage modulus. The storage modulus is increased mostly due to the excellent dispersion of the nanoparticles and Ni-P/BF.

3.7 Damping factor (tan δ)

The damping factor is evaluated from the ratio of storage modulus and loss of modulus. The damping factor of a material determines the viscoelasticity of a polyester matrix composite. The peak value of tan δ is used to calculate the glass transition temperatures, T_g, of the uncoated polyester composite and other samples. **Figure 8** shows that the reinforcement with coated BF and TiO_2 nanoparticles raises the value of T_g. The T_g value for the uncoated polyester composite is 85 °C, while the T_g value of the composite including 13.5 *w*/% of the coated fibre and 1.5 *w*/% of the TiO_2 nanoparticles reaches 91.3 °C. **Figure 8** shows the tan δ values for all the composites. The viscoelastic properties of the polyester-matrix composite is increased due to the addition of nanoparticles and coated bamboo fibre so that the polymer composite can withstand higher temperatures. The maximum value of T_g is raised at the optimum loading of the coated fibre and nanoparticles.

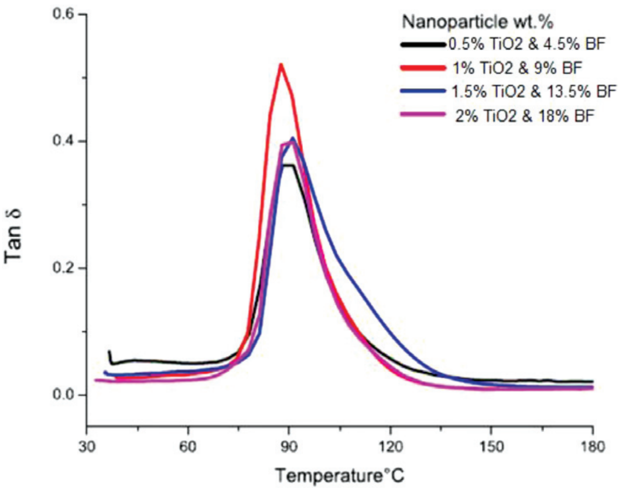

Figure 8: Tan δ vs. temperature plot for the polyester nanocomposites

4 CONCLUSIONS

Bamboo fibres are coated with nickel-phosphorous using the electroless plating method and trial and error method. The presence of nickel-phosphorus particles over the entire surface of the bamboo fibre indicated by spherically shaped bubbles is shown on FESEM images.

Polyester composites with the reinforcing bamboo fibre that can be coated or uncoated, and titanium oxide nanoparticles were developed using a compression moulding process and various concentrations. An exfoliated structure of the polyester-matrix nanocomposites was revealed using an XRD analysis. The polyester nanocomposite material with an addition of 1.5 *w*/% of titanium oxide nanoparticles had stable thermal properties and a high residual content.

The storage modulus (E1) of the polymer matrix reinforced with coated bamboo fibres and 1.5 *w*/%. of nanoparticles was also high. Values of the storage modulus and thermal temperature of 3.96×10^4 MPa and 91.3 °C were obtained with 1.5 *w*/% of titanium oxide nanoparticles. The FESEM result determined the surface properties of the composite.

The coated bamboo fibre and polyester matrix exhibited effective interfacial bonding with the nanoparticles. Finally, the experimental result showed that the momechanical and dynamic mechanical properties of the polyester matrix reinforced with the coated bamboo fibre and nanoparticles allow excellent bonding as compared to the composite including the polyester matrix reinforced with the uncoated bamboo fibre.

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