Mechanical and Tribological Behaviour of Hybrid Multi Fibre Reinforced Nylon 6-6 Nanocomposites

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Abstract. The investigation on the effect of adding silane modified chopped E-glass fibre and Aluminium metal wire-mesh into nano silica toughened nylon 6-6 thermoplastic composites on mechanical, drop load impact, fatigue and tribological behaviour is studied in this paper. The primary aim of this research work is to develop a hybrid Nylon 6-6 nanocomposites having high stiffness, toughness and wear resistance. The chopped glass fibre and Al wire-mesh was surface treated with the help of 3-Aminopropyletrimethoxylane (silane) and acid etching. The tensile results revealed that additions of glass fibre and Al mesh into nano - silica toughened nylon 6-6 composite gives improved tensile and flexural strength. Similarly, the Izod impact strength of Al-mesh reinforced nano silica (1vol.%) toughened nylon 6-6 gives superior energy absorption up to 6 Joules/cm. The drop load impact penetration of composite N3 (59% - Nylon 66, 20% - E-glass fibre, 20% - Al wire mesh and 1 % - nano silica) shows very limited penetration than other composites. Highest fatigue life of 16391 cycles was observed for the composite designated N3, which contains 1 vol. % of nano silica, whereas the composite containing 2 vol.% of nano silica gives very lower specific wear rate and Co-efficient of friction. The developed composite which has better modulus, stiffness, wear resistance and fatigue life could be possibly used in automobile power transmission gears, domestic equipment and farm related machineries.

Keywords: thermoplastic, aluminium wire-mesh, nano-silica, fatigue, tribology

1. Introduction

Thermoplastic composites are predominantly used in various sectors when compared to other types of composite materials, since they are cost-effective, easy to fabricate and readily available in huge quantities [1]. When these composites are virgin and plain their demand is literally low compared with reinforced polymer composites. The addition of fibres and filler particles into these polymers could improve the mechanical, thermal and energy absorption behaviours [2]. Recently, polymer matrix composites are widely used in automobiles than metallic materials because of their low cost, sustainability, deployability and bio eco-friendly nature [3]. For structural engineering applications, thermoplastics are more preferred than thermosets because of their inbuilt flexible molecular structure, recyclability and higher formability, whereas the thermosts are very rigid, high brittle, one-time processing limitation and non-bio degradable [4]. In general thermosets and thermoplastics could be strengthened through two routes namely fibre strengthening and particle strengthening. The fibre strengthening improves the load bearing characteristics whereas the particle strengthening improves the micro level properties such as wear and thermal stability [5]. In some cases, metal mesh also could be used as load bearing element when the particular application is focused for high damping and energy absorbing application. Given the choice of study, researchers prefer nylon 6-6 as matrix, since it is a commercially available thermoplastic, economically cheap and easy processing methodologies [6]. The glass fibre and aluminium wire mesh could be selected as reinforcements, since both are easily available and highly performing engineering reinforcements.

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Glass fibres are foreknown material for better mechanical and load bearing characteristics, similarly aluminium wire mesh is having very high tensile strength and high energy absorbing capabilities [7]. Moreover, for particle strengthening the silica nano particles could be used since, silica is very hard, possess high thermal stability, high wear resistance and very high melting temperature. Apart from these superior properties, they are easily available and cost effective compared with other rare oxides [8]. The surface-modified particles actively react with polymeric matrix and also the particles could disperse uniformly [9]. Dinesh et al [10] studied the effect of adding surface-treated iron (III) oxide particles in epoxy resin matrix on thermo-mechanical behaviour. It was observed that the surface-modified reinforcements improve the mechanical and thermal properties compared with unmodified reinforcements in a matrix. Arunprakash et al [11] studied the effect of adding SS-304 wire-mesh and Al-6061 wire mesh into epoxy resin along with E-glass woven mat. Adding wire-mesh along with glass fibre improves the drop load energy absorption rate and penetration depth. Parthipan et al [12] conducted an experiment on effect of nano-silica addition into epoxy resin matrix. The authors observed that addition of nano silica of desirable percentage improved the mechanical and wear behaviour of epoxy composite. Similarly, the acid leached metal wire-mesh could be more porous after surface modification, which could improve the adhesion of polymer matrix with leached portion of wire-mesh. Thus, the surface-modification via silane substance or acid leaching may be important while preparing high performance polymer composite materials. In this research work, the tribological behaviour of Nylon 66 reinforced with E-glass fibre, Al wire mesh and nano silica is studied. These mechanically strengthened, high load bearing, high wear resistance and fatigue failure resistance of nylon composites could be used as gears in power transmission areas such as automobile gear box, power tool exchangers and machinery parts.

2. Materials and methods

2.1 Materials

The commercial nylon 6-6 with density 1.15 g/cm$^3$ and molecular weight 224 g/mol was purchased from Alpha chemical company, Chennai, India. The chopped E-glass fibre of density 2.3 g/cm$^3$ and aluminium wire-mesh of density of 2.65 g/cm$^3$ and wire diameter of 0.25 mm was purchased from Metro metal traders Pvt. Ltd, Chennai, India. The nano silica of size 20 nm and silane coupling agent 3-Aminopropyletrimethoxylane (APTMS) were purchased form Sigma Aldrich, USA. The solvent ethanol was purchased from METRICK, India, Ltd. Figure 1 shows the SEM images of E-glass fibre, aluminium wire-mesh and nano silica particles used in this investigation.

![Figure 1. SEM images of (a) Chopped E-glass fibre, (b) Aluminium wire-mesh and (c) Silica nanoparticles](image-url)
2.2. Surface-modification of second phase reinforcements

The chopped E-glass fibre and nano silica particles are surface-treated by silane coupling agent by aqueous solution method which is the ideal method [13]. In this method a concentrated aqueous solution was used to perform surface-modification. Required amount of APTMS of approximately 4 wt.% of silane was used for modifying the surfaces of E-glass fibre and nano silica particles. Similarly, the aluminium metal wire-mesh was surface leached by concentrated H₂SO₄. In this, the wire-mesh was briefly dipped immersed into concentrated acid and soaked for 10 min to allow surface leaching [14]. Figure 2 shows the SEM images of aluminium wire-mesh after surface-modification process.

![Figure 2. Surface leached Al wire-mesh](image)

2.3. Composite fabrication process

The hybrid composites were prepared by liquid casting method. In this process, the required composite size steel mould with induction heated set up was pre-formed with required dimension of 5mm thickness. The aluminium wire-mesh of 3 distinguishable layers (20 vol.%) and chopped E-glass fibre of 20 vol.% was filled in the pre-set induction heated steel mould. The melted Nylon 6-6 mixed with nano silica particles and stirred continuously for uniform mixing. The resulted colloidal suspension was then poured into the pre-heated (250°C) mould for 5 min then allowed for curing in ambient temperature. The casted nylon hybrid composites were taken out from mould and cleaned with cotton cloth and checked for any visual defects like mis-run and rat tail. Table 1 shows the composition and designations of nylon 6-6 hybrid composites prepared in this present study.

| Composite Designation | Nylon 6-6 (vol %) | Glass fibre (vol %) | Al wire mesh (vol %) | Nano silica (vol %) |
|-----------------------|------------------|---------------------|---------------------|--------------------|
| N₀                    | 100.00           | -                   | -                   | -                  |
| N₁                    | 80.00            | 20                  | 20                  | -                  |
| N₂                    | 59.50            | 20                  | 20                  | 0.5                |
| N₃                    | 59.00            | 20                  | 20                  | 1.0                |
| N₄                    | 58.00            | 20                  | 20                  | 2.0                |

2.4. Test specimen preparation

The fabricated hybrid composites were checked for dimensional adequacy for making test specimens. Minimum five identical specimens were prepared for each test. The test specimens were prepared in-accordance with ASTM standards by using an abrasive water jet machining. The test specimens were machined with required process parameters such as operating pressure of 200 MPa,
abrasive flow rate of 0.30 g/cm³ and nozzle diameter of 0.75 mm. The abrasive particle selected for the machining was SiC of particle size 5 µm.

2.5. Characterization

2.5.1. Mechanical

The tensile and flexural tests were conducted based on ASTM D 638 and 790 respectively. A universal testing machine (INSTRON 3000 series, UK) of 20 Ton loading capacity with feed rate of 1.15 mm/s along with hydraulic gripper setup is used for the present investigation. The Izod impact test was conducted with a mini-impactor (Krystal India Pvt. Ltd) with 20 J as maximum capacity based on ASTM D 256. The Shore-D hardness of hybrid composite was measured using Durometer followed by ASTM 2240. The indenter was made with 35°±1.0° included angle.

2.5.2 Low velocity drop load test

The low velocity impact behaviour of fabricated composites was tested based on ASTM D-4762 using a drop load impactor (INSTRON 9000 series, UK). The impactor has sharp nose tip mass of 1.0 Kg. A constant velocity of 2 m/s was maintained for all composite designations. The specimens were held with the help of hydraulic grippers for better stability.

2.5.3. Fatigue behaviour

The tensile fatigue behaviour of composites was done by using a fully automated fatigue tester (MTS, 1500, USA) with hydraulic gripper mechanism attached. The test was carried out with the guidance of ASTM D 3479. A 5 Hz loading frequency, R=0.1 stress ratio, 1.28KN of maximum load, 6.00 GPa of elastic modulus, and 23°C as working ambience were set as process parameters.

2.5.4. Tribology behaviour

The tribological behaviour of hybrid composites was investigated using a Pin-on-disc setup. The test was conducted in-accordance with ASTM G-99. A wear track diameter of 165 mm, sliding speed of 800 rpm, applied load of 5 N and sliding distance of 400 m was set as process parameters. The test was repeated 3 times for computing average results.

2.5.5. Morphology

The fractured surface morphology of Nylon 6-6 hybrid composites was scanned using a scanning electron microscope (HITACHI, S1500, JAPAN). The samples were spurted with gold before scanning to avoid charging during electron beam passing.

3. Results and discussions

3.1. Mechanical

The mechanical properties of the fabricated polymer composites is presented in Table 2. It can be observed that the pure nylon 66 possess a low tensile strength of 60 MPa due to the soft nature of nylon66, which is bounded with no strengthening mechanisms. The plastic deformation occurs at a much lower value of yield strength. It is observed that, additions of 20 vol.% of E-glass fibre and 20 vol.% of Al-6061 wire-mesh into nylon 66 improves the tensile strength by 25% for N₁ composite. This improvement is because of high load bearing E-glass fibre and high ductile Al-6061 wire-mesh with in matrix [15]. The presence of E-glass fibre spreads the load in uniform direction bi-directionally throughout the matrix and improves the modulus. Also, the presence of soft aluminium wire-mesh in matrix further increases the load bearing capability of matrix, thus higher tensile load is observed.

It is further observed that additions of nano silica of 0.5, 1.0 and 2.0 vol.% in to nylon matrix boost up the tensile strength. Tensile strength improvement of 32%, 41% and 38% is observed for composites, which contains nano-silica along with E-glass fibre and aluminium wire mesh. The presence of nano-silica stiffens the matrix, also reduces the stress concentration and stress intensity factor on the inbound
micro cracks thereby increasing the tensile strength [16]. But it is further noted that adding more volume percentage of nano-silica up to 2 vol.% reduces the tensile strength. The decrement in tensile strength is due to the agglomeration of large volume of nano-silica though they have been functionalised. At larger particle volume they get agglomerated and forms as a cluster. These clusters on matrix tend to improve the crack initiation and propagation thus reduction in tensile strength is observed [17].

| Composite designation | Tensile strength (MPa) | Flexural strength (MPa) | Izod impact (J/cm) | Hardness (D-shore) |
|-----------------------|------------------------|-------------------------|-------------------|-------------------|
| N₁                   | 60                     | 95                      | 0.6               | 77                |
| N₂                   | 80                     | 115                     | 3.6               | 77                |
| N₃                   | 88                     | 128                     | 4.5               | 84                |
| N₄                   | 102                    | 135                     | 6.0               | 88                |
| N₅                   | 96                     | 120                     | 5.2               | 94                |

The scanning electron microscopy images of fractured portion on surface-modified tensile specimen is shown in Figure 3. The fibre and wire-mesh shows highly reacted phase with nylon 66 due to surfacetreatments like silane on E-glass fibre and acid etching on wire-mesh surface. When the load is applied, these surface activated fibre and metal wire-mesh effectively shares the load and distributed through molecular reaction. There is no fibre pull-out and no delamination noticed on fibre-matrix interface. This indicates effective integration between reinforcements and matrix phases. Moreover, the other place on fractured portion shows almost flat fracture. There is no dimple formation and pit marks. The river marks on matrix shows the comparative brittle nature of matrix due to the presence of nano-silica particles.

Figure 3. SEM images of (a) before fracture and (b) after fractured portion of N₄ composite designation

It is observed that the flexural strength of nylon hybrid composite gets increased with additions of reinforcements. Adding E-glass fibre and aluminium metal wire-mesh of 20 vol.% into nylon 66, improves the flexural strength by 17%. This improvement is because of effective load sharing behaviour of E-glass fibre and high ductile aluminium wire-mesh. Further additions of nano-silica also greatly improved the flexural strength by improving flexural modulus. The improvements of 26%, 30% and 21% were noted for composites N₂, N₃ and N₄ respectively. This improvement is because load absorption behaviour of nano-silica particles with controlled volume up to 1 vol.% the flexural strength is higher beyond which, it decreases. Infusion of higher concentration of nano silica leads to agglomeration of nanoparticles and creates discrepancy in load transfer behaviour of matrix, which intern reduced the flexural strength [18]. Since nano-silica is hydrophilic in nature, it tends to attract more negatively charged ions in matrix and forms high cross-linking density. When forming high cross-linking density, the molecules lose their stretchability and become hard and strong with apparent decrease in ductility and behaves comparatively brittle. This brittle nature reduces the flexural strength and modulus at higher concentration [19].

Similarly, the impact toughness of nylon hybrid composite shows improved values. It is observed that the pure nylon 66 gives very low impact toughness of 0.6 Joules. While adding E-glass fibre and
nano silica into nylon 66 matrix, the energy absorption increases. The increment of 83% is observed for N2 composites. It is because of the presence of load absorbing E-glass fibre and aluminium wire-mesh in nylon 66 matrix. The reinforcements in matrix acts as barriers to the micro crack propagation, which in turn increases the energy absorption. Further addition of ultra-fine nano silica aids in improving energy absorption. The increment of 86%, 90% and 88% in Izod impact toughness is seen for composite N2, N3 and N4 respectively. For composite N4 there is a small drop in impact toughness due to higher particle loading. At higher concentration, nanoparticles forms cluster and creates discrepancy in load transferring phenomenon [20].

The shore-D hardness of Nylon 66 composite gives an improved hardness. The addition of hard nano-silica particle in nylon matrix occupies molecular voids and produces very hard surface, which in turn increase the hardness. The highest hardness of 94 Shore-D is observed for composite N4, which contain 2 vol.% of nano silica particles. The improvement of 8%, 13% and 18% were observed for composites N2, N3 and N4 respectively. In contrast with tensile, flexural and impact strength of composites the hardness increases with increase in concentrations of silica nanoparticles. Higher concentration of nano particles increases the immobility of the polymeric chains, arrests its motion against indentation and offers very high resistance against deformation [21].

3.2. Drop load impact behaviour

The drop load impact behaviour with respect to energy absorption from the hitting impactor is as shown in Figure 4. It is noted that the pure nylon 66 gives very poor load absorption due to the inefficient load absorbing mechanism within it. It is observed that the addition of 20 vol.% of E-glass fibre and aluminium wire-mesh into nylon 66, the energy absorption increases. The improvement of 90% was observed for composite N1, which contains reinforced fibre and wire-mesh elements. This improvement is because of high energy absorption of ductile aluminium wire-mesh, which could transfer and absorbs the suddenly applied load. The process of silane surface-modification greatly supports in load sharing and exhibits better bonding nature with matrix [22]. This high adhesion improved fibre and metal wire-mesh also arrests the formation of metal surface to matrix interfacial delamination and crack growth. The presence of metal wire-mesh receives maximum velocity of impactor by absorbing very high momentum. Thus, the penetration of impactor got reduced, which leads to higher drop load impact absorption. The presence of chopped E-glass fibre also acts as energy absorber during sudden impact of load. When the impactor hits the specimen, the load is uniformly shared in the matrix. When the matrix is reinforced with short fibres, the load is absorbed and diminished subsequently thus no force effect will create much damage on the matrix phase [23].

It is further noted that the additions of 0.5, 1.0 and 2.0 vol.% of nano-silica particle into Nylon 66, fibre/metal wire-mesh composite, the drop load impact energy absorption improves further. The improvement of 91.3%, 93.2% and 94% were observed for composite designations N2, N3 and N4 respectively. On comparing other composite designations, the nano-silica dispersed Nylon 66 composite possess improved energy absorption. This improvement is the cause of improved micro load sharing ultra-size nano-silica particles in the matrix, which could arrest the micro crack creation and propagation. This results in superior drop load impact behaviour of the composite [24].

It is observed that the composite N3 has the highest energy absorption capacity than other composites. Meantime the composite, which contains larger volume fraction of nano-silica (2 vol.%) greatly affects the toughness of matrix. As discussed earlier, high volume fraction of nanoparticles leads to clustering due to super saturation of particle in polymer voids. This reduced void content in polymer matrix makes it very rigid due to low molecular movement. When impact load acts on the top surface of the composite, the surface will generate cracks. These cracks further propagate immediately resulting in permanent deformation. Even though the particles are surface-treated, the dense matrix medium makes the particles to settle in a place due to super saturation. Hence, poor energy absorption is found in higher particle loaded nylon 66 composite [25].
3.3 Fatigue behaviour

The fatigue behaviour of Nylon 66 hybrid composites is shown in Figure 6. It is observed that the pure nylon 66 has very low fatigue life of 629 cycles. This lower fatigue life is the cause of poor load bearing capacity of nylon 66 polymer. There are no micro load sharing phenomena taking place, thus the cracks easily develop and propagates. It is further noted that the inclusion of 20 vol. % of E-glass fibre and aluminium wire-mesh into nylon 66, restricts the formation of micro cracks. The improved life cycle of 9001 counts is observed for composite N₁. This improvement is the due to the presence of glass fibre and metal wire-mesh which uniformly shares the applied load and reduces the formation of micro cracks [26].

It is further noted that, the addition of nano-silica along with E-glass fibre and aluminium wire-mesh improves fatigue life cycle. The improvement of 95%, 96% and 95% were observed for composites N₂, N₃ and N₄ respectively. This improvement is because of reduction of micro crack formation by the presence of nano silica particles. The nano silica particles absorb tensile load from every cycle and reduce the risks of climb dislocations, which causes permanent deformation. When tensile load is applied as high frequency for long cycle the accrued residual stress results in a permanent failure but the presence of nano silica acts as a barrier on matrix for restricting the mobility of crack growth. This phenomenon is similar like precipitation strengthening on metals. The presence of tiny silica particles present on nylon
matrix provides significant restriction to form notable cracks leading to better resistance and does not deform during high cycles of load [27].

It is observed that the larger volume fraction of particles on nylon matrix marginally reduces the fatigue life cycle of composites. Large concentration makes the particles to agglomerate and tend to form particle cluster. These clusters are possibly creating more micro cracks around the clusters. In higher frequency of same load, slowly the dislocations (micro crack) capture one by one and forming a continuous and notable deformation. Figure 6 shows the scanning electron microscope images of nylon 66 hybrid composites. Figure 6(a) shows the microstructure of composite N3 under fatigue test. There is no interfacial cracking found near the fibre-matrix interphase. Figure 6(b) shows the SEM image of broken surface of N4 composites. It indicates that, more micro cracks developed near to the fibre and wire mesh side, which leads to complete plastic deformation.

![SEM images](image_url)

**Figure 6.** SEM images of (a) N3 and (b) N4 composite designation

### 3.4. Tribology

The Co-efficient of Friction (COF) and specific wear rate of nylon 66 composites is shown in Figure 7. It is noted that the pure nylon 66 gives COF and specific wear rate of 0.7 and 0.14 respectively. This very high value of COF induces larger sp. wear rate when this composite is exposed to abrasive wear disc. This larger sp. wear rate is because of soft nylon 66 molecules, which could not withstand external shear force. When the test specimen is exposed to abrasion disc, the surface layer of nylon 66 gets eroded easily. It is further noted that adding 20 vol. % of E-glass fibre and aluminium wire mesh in to nylon 66, both the COF and sp. wear rate decreases considerably. The COF of 0.55 and sp. wear rate of 0.12 is observed for composite N2. This improvement in wear resistance is the cause of inbuilt wear resisting materials. The addition of E-glass fibre and aluminium wire mesh reduces the contact area of pure nylon 66 in the abrasion disc during operation. Instead of nylon 66, the E-glass fibre and aluminium wire-mesh approaches the abrasion disc thereby improving the wear resistance [28].

It is further noted from Figure 7 that, the addition of nano-silica particles in to fibre-nylon 66 hybrid composite further improves the wear resistance. The COF decreases by 26%, 29% and 34.2% and corresponding specific wear rate also decreases by 43%, 49% and 60% for composites N2, N3 and N4 composite designations respectively. This improvement in wear resistance is due to the presence of nano-silica particle. The high surface area nano silica particles are exposed to the abrasive disc and reduces the direct contact of soft nylon 66 in to the abrasion disc. This phenomenon improves the wear resistance of nylon 66 hybrid composites. Also, the presence of hard nano-silica particles may also act as an roller at nano and micro level, which may also induce a three body roller effect and hence avoids the direct contact of specimen with abrasive body and decreases the coefficient of friction between the working specimen and abrasion disc. The change in COF significantly influences the specific wear rate of the composites [29].
It is observed that the addition of large volume of nano-silica particles greatly supports the reduction of coefficient of friction and sp. wear rate. The presence of large volume of nano silica improves the slippery between abrasion disc and composite specimen both by rolling and sliding action, which improves its tribological properties of hybrid composites with large volume fraction of nano silica.

The scanning electron microscope image of worn surface of N2 and N4 composites is shown in Figure 8. The composite, which contain only E-glass fibre and aluminium metal wire-mesh (N2) produces wavy and high pit marked worn surface. This indicates the ductile nature of nylon 66 matrix and high COF between test specimen and abrasion disc due to sticky action and ploughing action as well. But in Figure 8(b), the worn surface shows almost flat surface and there is no trace of pit marks, which indicates very hard surface of composite and reduced coefficient of friction [30].

4. Conclusions

Based on the experimental work carried out, the salient results of the present study are summarized below.

The incorporation of surface-modified E-glass fibre and aluminium wire-mesh into nylon 66 thermoplastic matrix improves the tensile, flexural and impact properties.

The presence of high ductile aluminium wire-mesh improved the energy absorption behaviour of composite to a greater extent.

The nano-silica infusion at lower concentration (1.00 vol.%) improves the tensile and flexural properties, whereas for higher concentration (2 vol.%), properties decrease marginally.

The presence of fine hard nano-silica of 2 vol.% gives very high hardness among other composites.

The fatigue behaviour of composites shows improved fatigue life for composites infused with E-glass fibre, aluminium wire-mesh and nano-silica additions compared with Nylon 66. At 2 vol.% of nano-silica, the fatigue behaviour comes down.
The wear behaviour of nylon 66 hybrid composites shows improved wear resistance. For the nanoparticle concentration of 2 vol.%, the coefficient of friction and specific wear rate is the least. Thus, the additions of E-glass fibre and aluminium metal wire-mesh along with nano silica toughened nylon 66 composites provides an opportunity to use it as power transmission gears in automobile and other engineering devices where high strength to weight ratio is desirable.

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