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Effect of Alkali Treatment of *Alstonia macrophylla* (AS) fiber on Dynamic Mechanical and Machinability Properties of Polypropylene (PP) Composites reinforced with Unidirectional AS fiber

E. Sakthivelmurugan[a*]***, G. Senthil kumar***[a]***, S.M. Vinu Kumar***[b]

[a]*Bannari Amman Institute of Technology, Department of Mechanical Engineering, Sathyamangalam, Erode district, Tamil Nadu, India.*
[b]*Sri Krishna College of Technology, Department of Mechanical Engineering, Kovaipudur, Coimbatore district, Tamil Nadu, India.*

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Dynamic mechanical analysis (DMA) and drilling performance of the polypropylene (PP) composites reinforced with different volume fractions (0, 10, 20, 30, 40 and 50 vol.%) of untreated and alkali treated novel *Alstonia macrophylla* (AS) fiber were investigated. DMA reports showed that 40 vol.% fiber loaded PP composites imparted good thermomechanical properties in terms of storage modulus (E'), and loss modulus (E") with a remarkable 366% and 331% improvement than virgin PP laminates respectively. In addition, study also disclosed that, alkali treated composites exhibited higher DMA properties than untreated PP composites owing to strong interfacial bonding of fiber and matrix as result of alkali treatment. Drilling study was also performed to investigate the effect of chemical treatment and drill parameters viz., drill point angle (90°, 118°, and 130°) and feed rate (20, 40, and 60 mm/min) on machinability property of PP/AS composites. Drilling outcomes conveyed that alkali treated PP/AS composites showed slight resistance to drilling than untreated composites owing to strong interfacial strength which played a vital role in resisting thrust force. Field emission scanning electron microscopy (FESEM) was used to capture the images of the drilled surface to understand the morphology of the PP/AS composite.

Keywords: *DMA, Drilling, Alstonia macrophylla, Polypropylene, Natural fiber composites, Alkali treatment, Thrust force, Torque, FESEM, Delamination.*

1. Introduction

Newer technological advancement in manufacturing firms, mostly automotive, aerospace, marine, electronic appliances and consumer products, created the demand for polymer composites to continuously increase[1,2]. Manufacturing of the synthetic fiber reinforced polymer composites such, glass fiber and carbon fiber based polymer composites consumes higher material and synthesing cost. In order to curb those costs, recent studies have showed the way for new technologies for producing the natural fiber based polymer composites, especially where applications don't require extreme mechanical properties[3]. Natural fiber reinforced polymer composite (NFRPC) have replaced most of the conventional synthetic fiber composites in automotive industries[3,4] and studies are under way to explore their potential usage to continue in aerospace and marine industries as well[3]. Though NFRPCs have advantages like, environmental friendly, lower density, higher degree of flexibility, and low manufacturing cost, yet their applications are limited owing to their hydrophilic characteristics with high disparity of mechanical properties[4,5]. This limitation can be averted to maximum extent by prior chemical treatments. Commonly used chemical treatments are alkalization, acetylation, and silanization[6,7].

NFRPCs manufactured from thermoplastic polymers have an upper hand over the thermoset composites because of their better impact resistance, easier recyclability, higher damage tolerance, ease of processing and low price of the polymer matrix[7]. Therefore, natural fibers reinforced thermoplastic (NFRT) composites have taken special places in packaging, transportation, structural and building industries[8]. Amongst the thermoplastics, polypropylene (PP) is perhaps the sought after polymers for producing NFRT composites due its affordability and exhibiting good physical, chemical and mechanical properties. PP has a major demerit. It is non-polar candidate because of the deficiency of functional group in its structure as a result of which it possesses low surface energy resulting poor interfacial adhesion between polymer and fiber member[9]. This can be overcome by employing appropriate surface treatment techniques. Literature suggests that PP has been reinforced with several natural fibers such as, flax, kenaf, abaca, jute, sisal, ramie and hemp[8]. However, there are numerous natural fibrous materials existing in the biomass which are yet to be exposed in order to utilize their potential to become reinforcement candidate. *Alstonia Macrophylla* (AS) tree is one such which is majorly found in South East Asia. It is sometime referred as Hard alstonia, Hard milkwood or Big-leaved macrophyllum[10].

*e-mail: sakthi.glen@gmail.com

Nowadays, NFRT composites experiences various kind of unprecedented stress while performing as component in applications. Thus it is foremost to understand their dynamic characteristic which can be explored under creep, stress relaxation and DMA studies[11]. Amongst them, DMA provides quite good information on viscoelastic properties of the composite for a short span of time as compared with other two techniques. Hence, insight into DMA properties of the NFRT composites is essentially required to establish the mechanical performance of the end product. DMA properties are studied in terms of storage modulus (E'), loss modulus (E") and damping factor. Storage modulus (E') refers to the energy stored in the system, whereas its viscous nature in the form of energy dissipation during the process is described by loss modulus property (E")[12]. Few studies have reported on viscoelastic behavior of the natural fiber reinforced PP composites[13-16]. Main outcomes of natural fiber reinforced PP composites[13-16] were on effect of fiber reinforcement and laminae layers[13,15], influence of chemical treatment on interfacial bonding[13,15], and dominance of the fiber reinforced PP composites over the neat PP on DMA properties.

Drilling is most sought after operations in secondary machining process of the composites. In order to facilitate the mechanical fastening it is inevitable to make the hole on the composites parts, particularly for making parts used in assembly. Despite the NFRPCs manufacturing in good quality forms, their performance and properties along with ability to endure drilling force is another issue. Drilling of NFRPCs are unlike drilling of metals and alloys because they are immiscible in nature and drill bit has to cut the two phases namely, fibrous and polymer layers which induces severe damages around the hole. Drilling induced damages relied on the thrust force and torque generated. Drilling forces should have minimized as possible for producing the higher quality of drill hole. This is accomplished by controlling of various process parameters such as, drill bit geometry, feed rate, and cutting speed.

From the exhaustive literature studies it is learnt that only few researchers have explored the effect of chemical treatment on machinability property of the composites, otherwise most of the papers[17-20] were limited with the optimization of the drilling parameters (types of drill bit, spindle speed, feed rate, drill point angle and etc.,). Moreover, studies pertaining to the influence of the chemical treatment on DMA performance of the thermoplastic composites is also still lacking attention. Thus, for the first time PP composites reinforced with novel *Alstonia macrophylla* fiber have been developed and studied the effect of chemical treatment on their DMA and drilling characteristics.

2. Materials and Methods

2.1. Materials

In this study, dry seed pods from the *Alstonia macrophylla* tree were collected as it is found vastly in the region of Sathyamangalam Taluk, Erode district, Tamil Nadu, India. Dry seed pods were soaked in the water for the specified period of time in order to extract their fibers using water retting process. The PP sheets were procured from Ghanshyam Polyplast, Coimbatore. PP sheet and AS fiber were employed as matrix material and reinforcement member respectively for fabricating the PP/AS composites and shown in the Figure 1.

2.2. Chemical treatment of AS fibers

Raw AS fibers were soaked in 5% (w/v) aqueous solution for 60 minutes at room temperature (29°C). Afterwards, neutralization process was followed, where fibers were allowed to soak in water containing 1% (w/v) acetic acid for reducing any traces of NaOH remaining on the surface of the AS fiber. Alkali treated AS fibers were kept in air oven at 65°C for 120 minutes to make them moisture free. Finally, untreated and alkali treated AS fibers are used as reinforcement member for making their respective PP composites.

2.3. Fabrication of PP/AS composites

PP/AS composites are made by employing film stacking technique and following its curing in hot compression moulding machine. In this technique, PP sheets and AS fibers were stacked alternatively in the aluminium mould and subjected to incessant pressure (25 bar) and temperature (180°C) in hot compression moulding machine for time duration of 15 minutes. During the course of curing under compression, slight higher pressure in the scale of 0.5 bar was applied when the melting point of PP is reached to enhance stacking ability of laminae.

Figure 1. (a) Pure PP sheets (b) Raw-*Alstonia macrophylla* fiber.

Then entire mould was cooled under normal room temperature and once curing is done, PP/AS composite laminate was removed from the aluminium mould and cut into required shape for further investigations. The schematic representation of the fabrication of PP/AS composite is depicted in Figure 2 and Prepared PP/AS composites are shown in the Figure 3. Detail on the designation and composition of PP/AS composite is indicated in the Table 1.

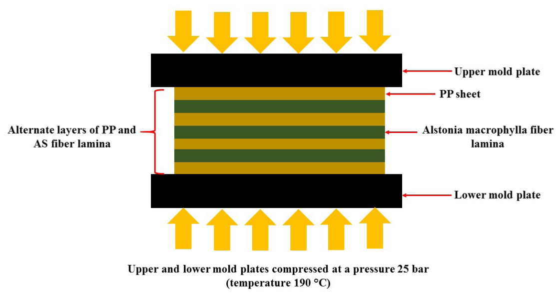

Figure 2. Fabrication of PP/AS composites.

2.4. Dynamic Mechanical Analysis (DMA) study

Viscoelastic properties of PP/AS composites were studied in terms of storage modulus, loss modulus, glass transition temperature and damping factor using dynamic mechanical analyser (Make: SII Nanotechnology Japan-DMS 6100) under three-point bending configuration for the temperature range 25°C–150°C with a heating rate of 3°C min−1 operated at a frequency 10 Hz.

2.5. Drilling Setup

Drilling was performed on PP40AS composites using 8mm diameter HSS twist drill in a vertical CNC machine. In this work, spindle speed is kept constant (2500 rpm), whereas feed rate (20 mm/min, 40 mm/min, and 60 mm/min) and drill point angle (90°, 118°, and 130°) is varied to study their effect on drilling responses; thrust force, torque and delamination. Figure 4 depicts that drilling setup and different drill point geometries used for drilling the PP/AS composite.

The composite specimen was placed over the force dynamometer (Type: Make: Kistler) by means of fixtures to execute the drilling operation. The thrust force, torque signals were recorded via dynamometer and it is processed further through data acquisition system to arrive for their numerical values.

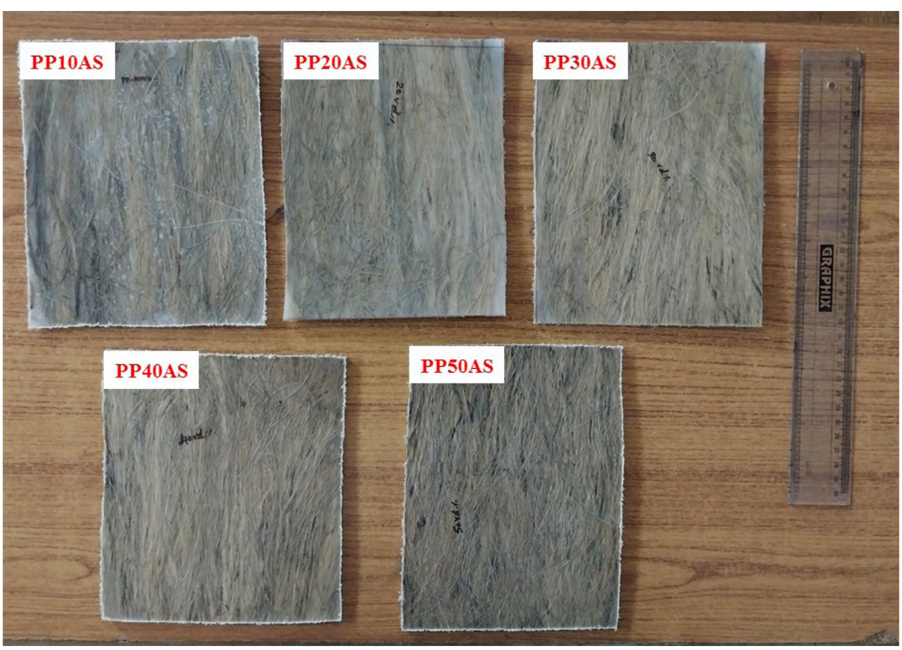

Figure 3. Fabricated PP/AS composites.

Table 1. Composition of the PP/AS composites.

Sample code	Reinforcement and matrix member				Volume fraction (Vol.%)	
	Type of fiber	Chemical treatment	AS Fiber layers	PP layers	AS Fiber	PP
Neat PP	-		-	3	-	100
PP10AS	Unidirectional fibers	1. Untreated fibers	2	3	10	90
PP20AS		2. Alkali treated fibers	2	3	20	80
PP30AS			4	6	30	70
PP40AS			4	6	40	60
PP50AS			6	8	50	50

Furthermore, quality of the drilled holes in terms of delamination was observed using tool makers profile projectors. Equation 1 was used to determine the delamination factor of the PP/AS composites[17,18].

$$d_f = D_{max} / D_{nominal} \quad (1)$$

Where, d_f denotes delamination factor; D_{max} and $D_{nominal}$ represents the maximum diameter and nominal diameter of the drilled hole as indicated in the Figure 5.

2.6. FESEM Analysis

Morphology of the surface of the drilled hole of PP/AS composites were examined using Field emission scanning electron microscopy (FESEM) (Make: Carl Zeiss Sigma-300, Schottky FEG) under different magnifications.

3. Results & Discussions

3.1. Storage modulus of untreated and alkali treated PP/AS composites

Figure 6 depicts the comparison of the storage modulus of Neat PP and PP/AS composites at different fiber loadings for both untreated and treated PP composites. The storage modulus of neat PP is around 0.95 GPa which is increases with increase in the fiber loading because addition of AS fibers in PP provides restriction to the matrix molecules resulting in increase in storage modulus[15]. Moreover, extent of increment of storage modulus property varies depending upon volume percentage of AS fiber percentage in the PP composites[21]. Also, it is evident from the figures that, as compared

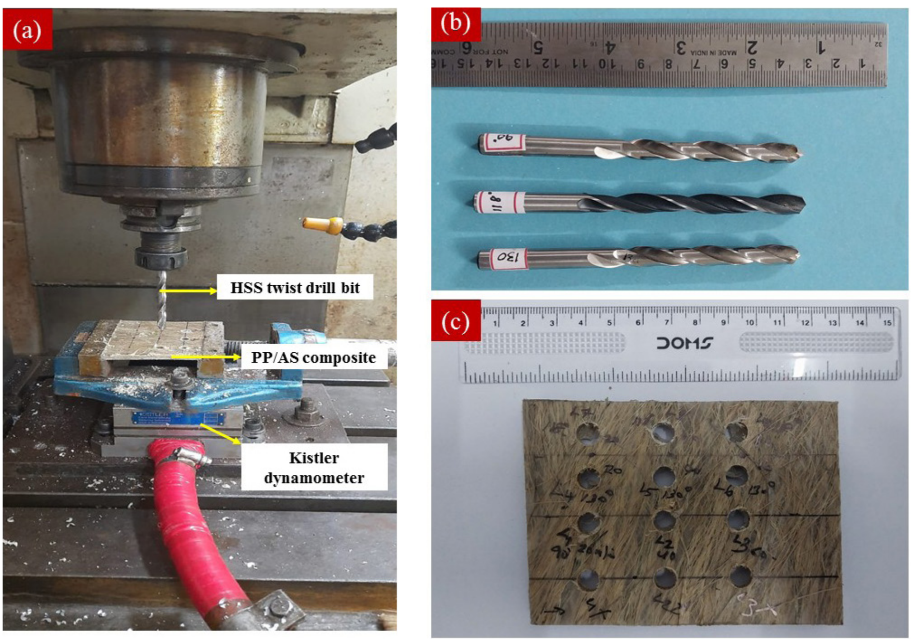

Figure 4. (a) Experimental drilling set up (b) Different geometry of HSS drill bit used (c) Drilled hole on PP/AS composites.

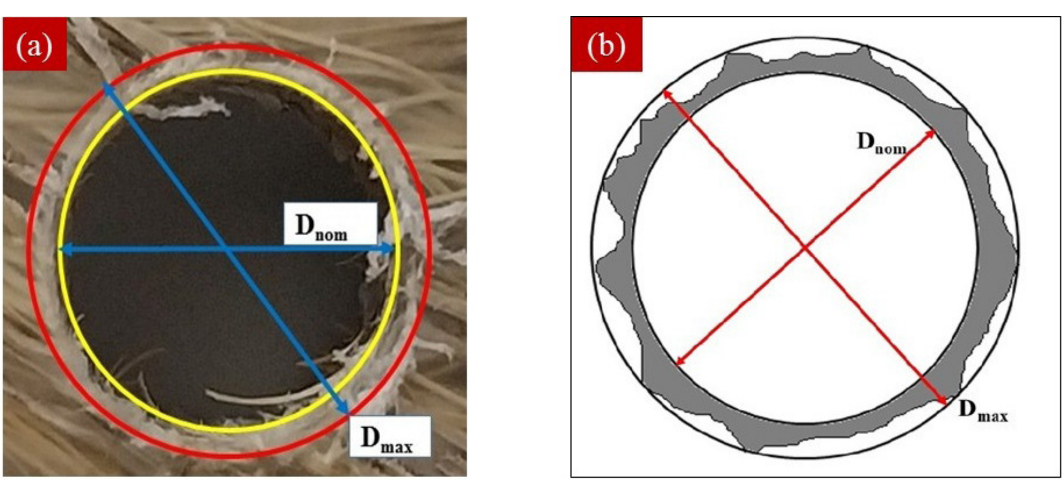

Figure 5. Evaluation of the Delamination factor.

to untreated PP10AS, PP20AS, PP30AS, PP40AS and PP50AS composites, their alkali treated have shown 32%, 22%, 31%, 13% and 12% improvement in the storage modulus property. Amongst the prepared lots, maximum value of storage modulus is attained by alkali treated PP40AS composites (3.45 GPa) which is 13% higher than its untreated PP composites. This may be ascribed to the modification of the fiber morphology by the alkali treatment as it enhances the interfacial bonding of fiber and matrix. Furthermore, it can be seen that both untreated and alkali treated PP/AS composites (PP50AS) at higher fiber loading exhibited lower storage modulus which may be owing to the agglomeration of the AS fibers as the plying increases[16,22]. PP/AS composites retain higher storage modulus in rubbery zone (80°-120°C) compared to Neat PP, may due to the hindrance possessed by the AS fibers when the matrix molecules are at higher mobility level[12]. The percentage increase in storage modulus of the alkali treated PP40AS composites than its virgin PP laminate is 0.613 GPa (366%), followed by PP30AS and PP50AS composites by 0.576 GPa (343%) and 0.544 GPa (323%) respectively. These findings are in line with the work carried by other researcher on PP based composites[23].

3.2. Loss modulus of untreated and alkali treated PP/AS composites

Figure 7 represents the loss modulus vs. temperature curves of Neat PP and PP/AS composites at different AS fiber loading for both untreated and treated composites. Treated PP/AS composites showed higher loss modulus property than untreated composites. Amongst the fabricated PP composites, PP40AS composite exhibited higher loss modulus value followed by PP50AS, PP30AS, PP20AS and PP10AS composites. This trend is remained same in both treated and untreated PP/AS composites. With AS fiber loading percentage increases, loss modulus of the PP/AS composite is also increases when compared to neat PP. Loss modulus peak of neat PP is 0.025 GPa attained at 66.62°C which increases gradually with addition of AS fiber and the highest value (0.11 GPa) is obtained for PP40AS composites. The reason for increase in loss modulus may be attributed to the mechanical restrain provided by the addition of AS fiber to the free movement of molecular chains of the PP matrix[24]. As it evident from the figures that, compared to the untreated PP10AS, PP20AS, PP30AS, PP40AS and

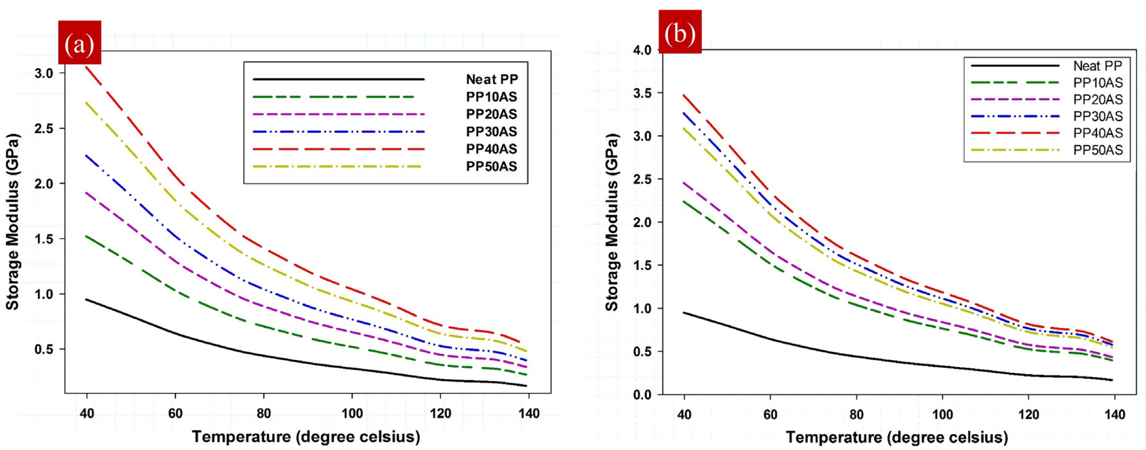

Figure 6. Storage modulus vs temperature curves of (a) Untreated PP/AS and (b) Alkali treated PP/AS Composites at different fiber loadings.

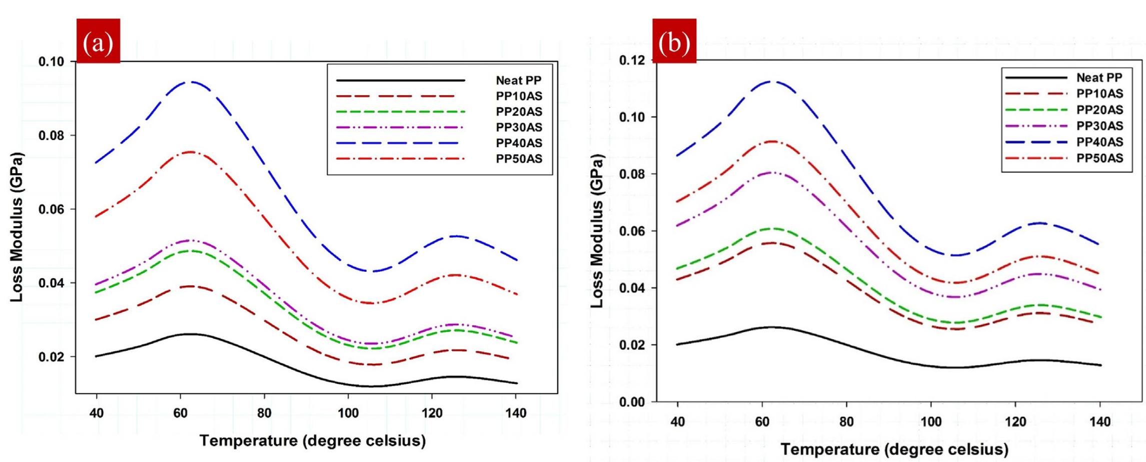

Figure 7. Loss modulus vs temperature curves of (a) Untreated PP/AS and (b) Alkali treated PP/AS Composites at different fiber loadings.

PP50AS composites, their alkali treated PP/AS composites have shown 30%, 20%, 36%, 26% and 28% improvement in the loss modulus property. This may be due to the fact that, PP/AS composite may have a higher energy dissipation at the fiber and matrix interface as result of chemical treatment. Moreover, loss modulus of the PP/AS composites decrease with respect to the increase in temperature, may be ascribed to the increased free movement of the polymeric chains as maximum energy is dissipated[25]. Similar to the storage modulus curves, all composites retain higher loss modulus in rubbery state as compared to neat PP[26].The distortion in the loss modulus trend at higher fiber loaded composite PP50AS is inevitable because of the plying effect of the PP layers as well as that of overcrowding of the AS fibers which results in frictional sliding of the fibers[27].

3.3. Damping property of untreated and alkali treated PP/AS composites

Tanδ vs temperature curves of Neat PP and PP/AS composites at different fiber loadings for both untreated and alkali treated PP/AS composites are shown in Figure 8 and it is evident from that the increase in the AS fiber content in PP has reduced the damping ratio of the PP composites may be because, increased fiber content restrains the segmental mobility of the matrix molecules, consequently storage modulus values of the composites increased more than the loss modulus values, thus resulting in lower Tanδ value[25]. The Tanδ peak for Neat PP is obtained at 74.17°C with a highest damping factor of 0.10, while PP40AS composite shows the lowest (0.05), as it restricts the mobility of the polymer chains endorsing a material with greater rigidity, thus decreasing the damping of the material. Untreated PP/AS composites showed higher damping value than treated PP/AS composites indicating that they are good damping materials as there exist a good interaction between fiber and matrix. Of the prepared PP composites, alkali treated PP40AS composite exhibited better damping property than its untreated composites due to the increased in the surface roughness property of the AS fiber as a result of NaOH treatment. Moreover, it is interesting to see that PP/AS composites exhibited lowest values of Tanδ before attaining the transition phase because at this stage, polymer endures restriction to its molecules movement. Subsequently with increase in temperature the molecular mobility of the polymer molecules increases, resulting in higher damping[28].

3.4. Drilling behavior of untreated and alkali treated PP/AS composites

3.4.1. Effect of feed rate and drill point angle on thrust force

For the current study, drilling signals were recorded using piezoelectric drill dynamometer (Kistler-Type 9272). Figure 9 and Figure 10 shows thrust force signals generated for untreated and alkali treated PP/AS composites while drilled using HSS twist drill bit by varying two parameters namely; feed rate and drill point angle. Figures clearly shows that, in the first stage of drilling there is an intense increase of thrust signal as cutting tool touches the composite. Following, steady zone and drastic decrement of drilling signal in the stage II and Stage III respectively. Former indicates composite drilling is underway, whereas in latter, drilling operation is completed as cutting tool exit completely from the laminate[17,20].

Figure 11 shows that the thrust force of both treated and untreated PP40AS composite increased with increase in the feed rate. It is noticed that both types of composites have followed the similar trend, expect mere deviation when the composites were drilled at feed rate 20 mm/min using 90° drill bit. Alkali treated composites drilled at feed rates 20 mm/min, 40 mm/min and 60 mm/min have shown 9%, 6% and 12.5% higher thrust force as compared to the untreated PP/AS composites, which clearly indicates that, chemical treatment of AS fiber has increased fiber and matrix interaction as lignin removal enhanced its wettability property thus resulting good mechanical bonding and so thrust force generated while drilling the composite was found higher. During drilling of PP/AS composite, if the bond between fiber and matrix resin is not stronger unlike in treated composites, then they slide each other and fails early when they are subjected to drill force and hence exhibits lower thrust force. Alkali treated PP/AS composites showed higher thrust force values because alkali treatment has heightened interface and interphase toughening mechanism in the PP/AS composites.

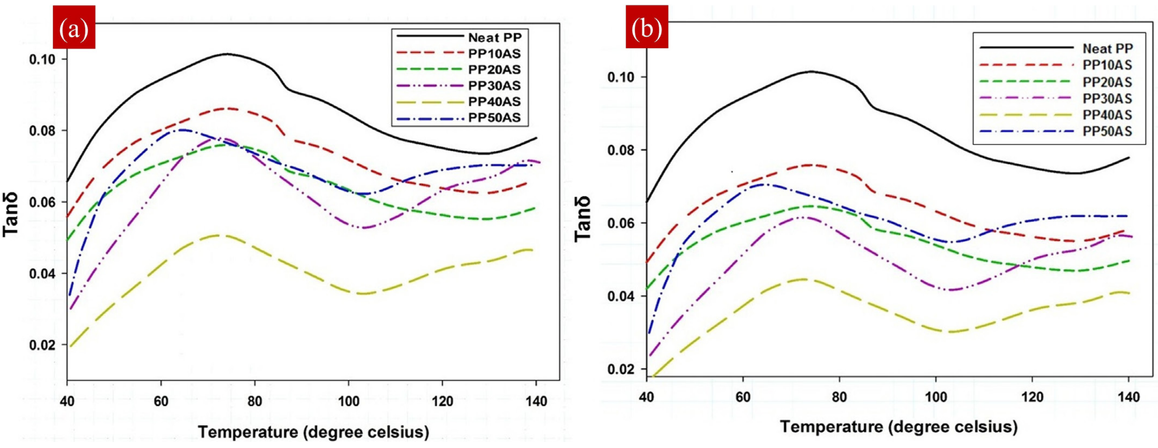

Figure 8. Tanδ vs temperature curves of (a) Untreated PP/AS and (b) Alkali treated PP/AS Composites at different fiber loadings.

From the Figure 11, it can be observed that increasing feed rate increases thrust force mainly attributed to the elevation of the shear area. Moreover, this increase in thrust force might have also occurred due to increase in the cross sectional area of the undeformed chip[29]. It is learnt from the literatures that, not only spindle speed and feed rate defines the quality of the drilled hole but tool geometry decides it by knowing thrust force value[30,31]. From the Figure 11 it is evident that thrust force increased with increase in the drill point angle. Higher thrust force 41.20 N is exhibited by the PP/AS composites when it is drilled with 130° drill point angle tool at the feed rate of 60 mm/min. Around 64% reduction of thrust force is observed when the same composites drilled using 90° point angle at the feed rate of 20 mm/min.

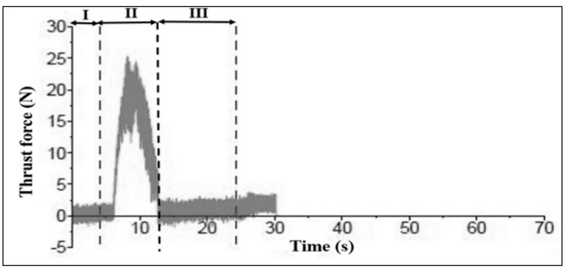

Figure 9. Thrust force signal for untreated PP/AS composite (Feed rate 40 mm/min; Drill point angle 118°).

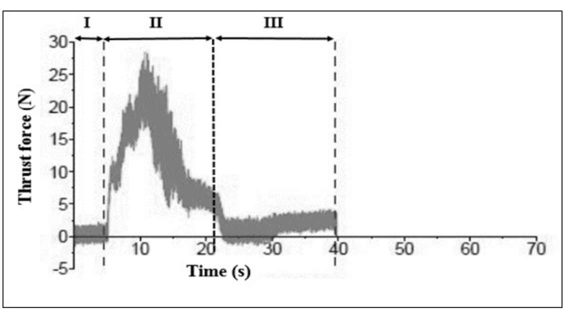

Figure 10. Thrust force signal for treated PP/AS composites (Feed rate 40 mm/min; Drill point angle 118°)

This reduction is mainly due to the decrease in the length of cutting edge which clearly indicates that drill tool can easily enter into the polypropylene composite specimen since its bending resistance along the axial direction is reduced[29].

3.4.2. Effect of feed rate and drill point angle on Torque

Figure 12 depicts the variation of the torque generated for untreated and alkali treated PP/AS composites when drilled at different feed rates and drill point angles. It can be seen from the Figure 12 that torque increases with increase in the feed rate and drill point angle. Alkali treated PP40AS composite exhibited higher torque compared to the its alkali treated one. This may be because of its higher physical and mechanical properties as imparted by the chemical treatment of fiber therefore its resistance to the mechanical drilling is higher which eventually leads to increase in required torque and thrust force[32]. Both untreated and alkali treated PP40AS composite experienced and lower and higher torque when they were drilled at the feed rate 20 mm/min using 90° drill point angle and 600 mm/min using 130° drill point angle, respectively. Moreover, drilling PP40AS composites at a higher drill point angle with increment of feed rate led to significant increase in torque. This may be attributed to drill geometries; as different drill geometries induce their own unique cutting mechanisms while drilling the composite material[18].

3.4.3. Effect of feed rate and drill point angle on Delamination

Delamination is a critical damage factor which has to be addressed carefully for producing good quality of holes. Not only that, mechanical and fatigue resistance composites will also be reduced and may land in catastrophic failure of the components. It is evident from the Figure 13 that, delamination factor of the alkali treated PP40AS composites found to be higher than its untreated composites may be because while drilling, treated composites experiences higher thrust force than untreated ones and eventually higher thrust force leads to higher delamination in the composites. Figure 14 clearly shows that alkali treated PP40AS composite endures more delamination damages as compared to the untreated composites. This may be attributed to strong interlaminar bond strength of the composite as a result of alkali treatment.

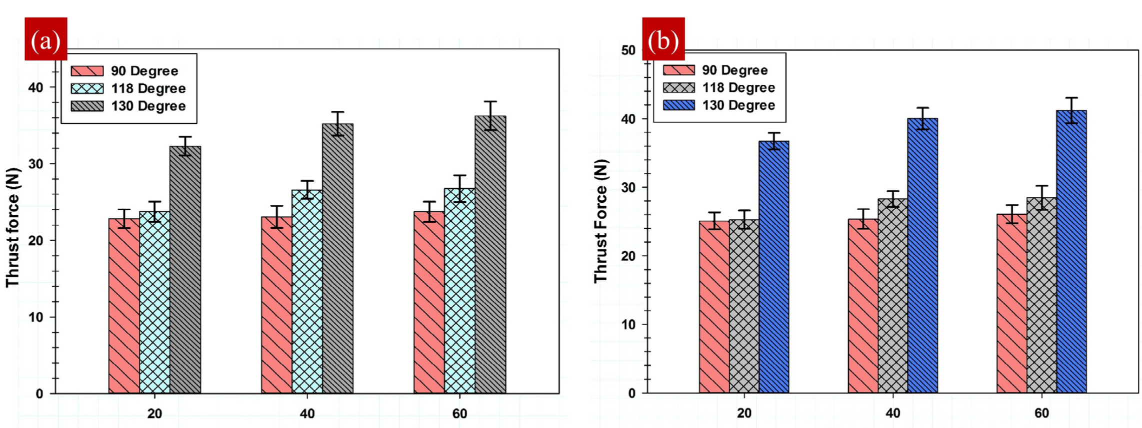

Figure 11. Thrust force vs Feed rate of the (a) Untreated and (b) Alkali treated PP/AS composites.

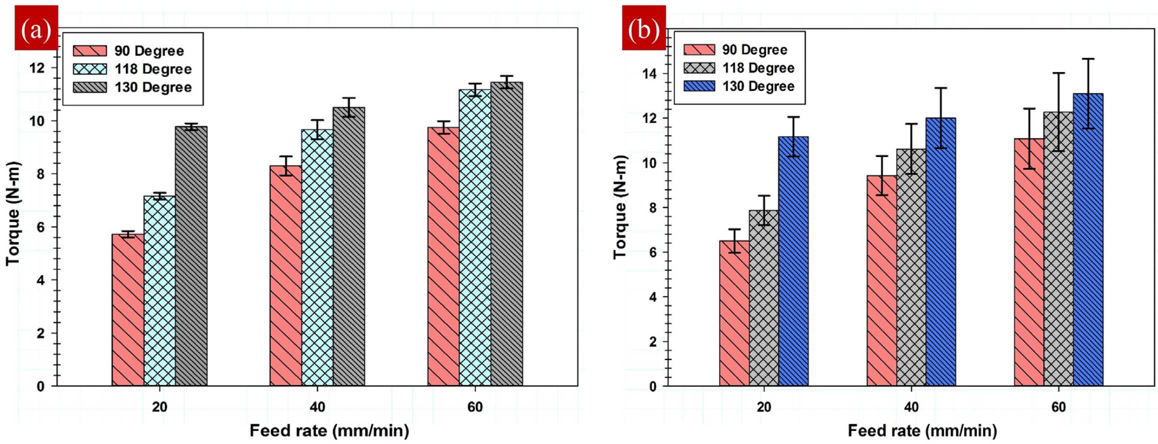

Figure 12. Torque vs Feed rate of (a) Untreated and (b) Alkali treated PP/AS composites.

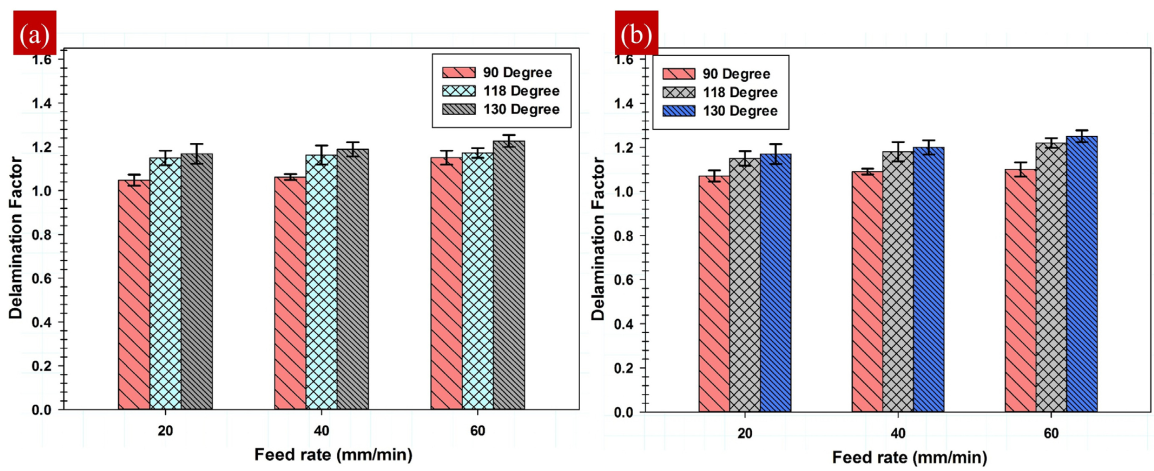

Figure 13. Delamination factor vs Feed rate of (a) Untreated and (b) Alkali treated PP/AS composites.

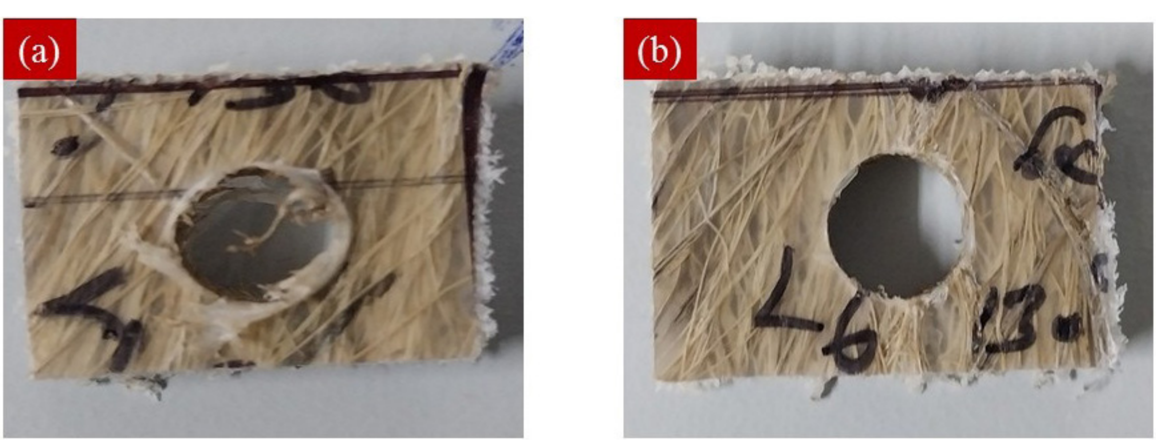

Figure 14. Delamination damage produced at 20 mm/min using 90° drill point angle for (a) Alkali PP40AS composites (b) untreated PP40AS composites.

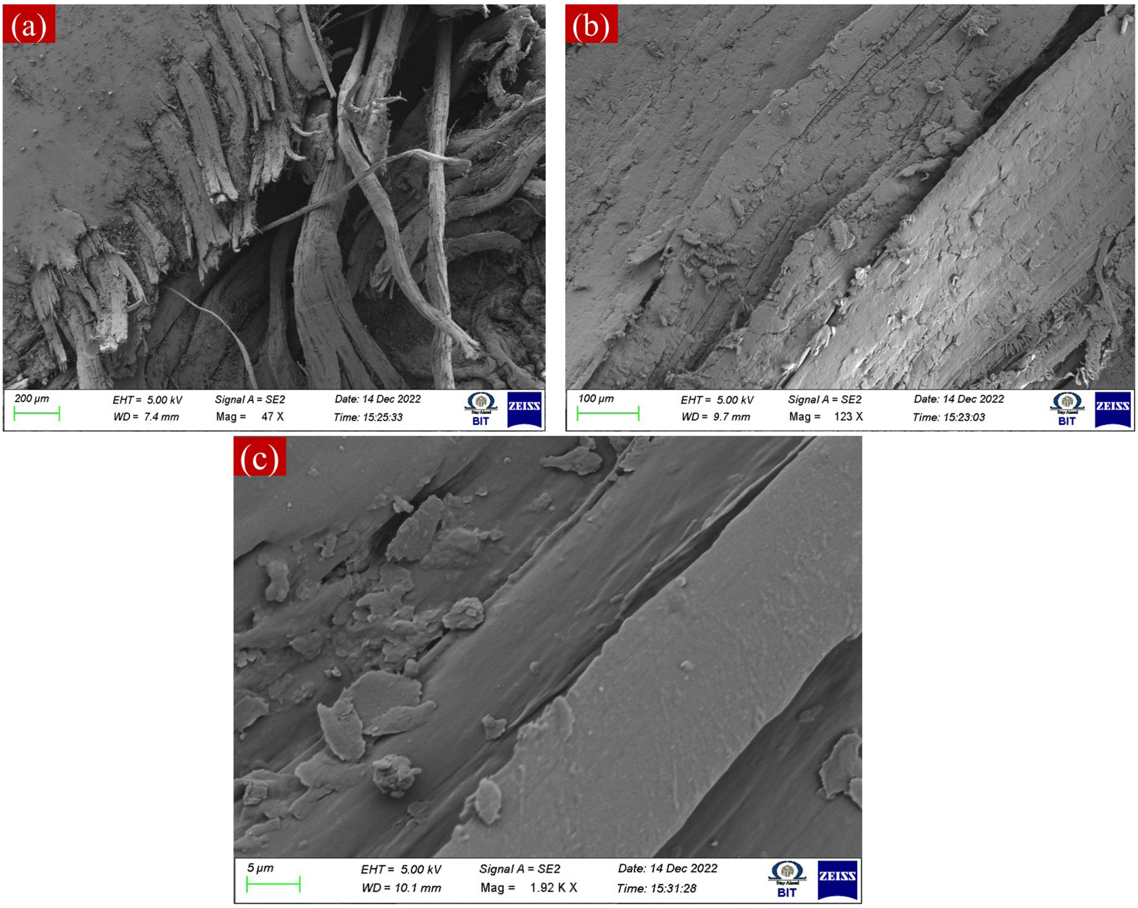

Figure 15. FESEM images of the drill surface of the PP40AS composites.

Delamination transpires only when applied loading exceeds the interlaminar shear strength of the composites. Besides, delamination situation may not rise until and unless the PP laminate is fully penetrated by the drill[33].

It is noticed in the figure that, irrespective of the composite materials drilled, increasing feed rate resulted in increasing delamination. This is because PP material deform under loading incurs the fiber splitting. Moreover, this amount of fiber splitting increases as drill point angle increases as reported by the researchers[29,34]. In other words, as drill point angle increases delamination also increases. From the figure that, for both untreated and alkali treated PP40AS composite, good surface quality of the hole with lower delamination can be produced if the PP/AS composites were drilled at the combination of lower drill point angle tool and minimum feed rate. These findings are in good agreement with the reported literatures[35,36].

3.5. FESEM analysis of the drilled hole surface of PP/AS composites

From the Figure 15, matrix fracture, fiber pull out and interfacial damage of fiber-matrix are observed and these may not be much severe when compared to the hole produced at higher feed rate for alkali treated PP composite. Because, lower feed rate resulting lower thrust force and hence drill bit doesn't damage the PP/AS composite this remain true in both untreated and alkali treated PP composites. Moreover, unlike thermoset matrix damages, crack formation around drilled hole is not significant owing to the ductility behaviour of the polypropylene material[3,17].

4. Conclusion

In this research work, DMA and drilling characteristics of the untreated and Alkali treated PP/AS composites were experimentally studied and based on which following inferences are arrived:

1. Storage modulus (E') and Loss modulus (E") properties of the PP/AS composites increased with increased in the fiber volume fraction up to 40 vol.% fiber loading and decreased beyond this fiber loading. PP40AS composites exhibited maximum E' and E" values of 0.613 GPa and 0.110 GPa respectively. Highest damping factor (Tanδ) of 0.10 was found for neat PP while 0.05 lowest for PP40AS. Moreover, alkali treated PP/AS composites showed better dynamic mechanical properties than untreated ones. Amongst the prepared lot, chemically treated PP40AS showed better property followed by PP50AS, PP30AS, PP20AS and PP10AS.

2. Alkali treated PP40AS composite experienced higher thrust force of 41.20 N when compared to its untreated composites (35.25 N) when they were drilled at feed rate 60 mm/min using 130° drill point angle tool. Also, it was evident that higher thrust force and feed rate will result in higher delamination. Minimum delamination damages were recorded at the feed rate 20 mm/min when the PP40AS composites drilled using 90° drill point angle tool.
3. Torque developed during the drilling of PP/AS composite have followed the footstep of the thrust force. Results clearly showed that torque found to be increased with increase in feed rate and drill point angle. Highest torque 13.09 N-m experienced by alkali treated PP40AS composite when drilled at higher feed rate 60 mm/min using 130° drill point angle tool than untreated composites (11.45 N-m). Torque of the alkali treated PP40AS was reduced to 48% by switching to different drilling parameters; at feed rate 20 mm/min and drill point angle 90°.
4. FESEM images of the PP40AS composite showed few prominent features like matrix fracture, fiber pull out and interfacial damage of fiber-matrix.

5. References

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