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Dynamic and Thermo-Mechanical Properties of Polypropylene/Date Palm Nano Filler Biocomposites

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ABSTRACT

Lignocellulosic based nano-sized filler are effective substituent for switching from manmade nanofillers (i.e. carbon, glass etc.) to reinforce polymer composites owing to its lightweight, plentifulness, and fully compostable characteristics. The present study adapted a dry mechanical ball mill assisted defibrillation process to produce nanoscale filler from waste date palm agro residue. Then, the melt mixing technique was used to fabricate biocomposites of polypropylene (PP) with 1-5 wt.% loading of this nanofillers.[0] A dimensional stability changes of the composites were studied by thermo-mechanical analysis (TMA). The thermomechanical analysis indicated that all these composite samples exhibits low coefficient of thermal expansion (CTE). The CTE values for the highest nanofiller loaded sample (5 wt.[20]%) was found to be 95 and 138 at 40°C and 80°C, respectively, when compared to neat PP, which is 80.4 and 125 at the same temperatures. [1]Moreover, the viscoelastic parameters of the biocomposites, such as storage modulus (E′ or G″), loss modulus (E″ or G″[2]), and damping factor (tan δ) were examined by using dynamic mechanical analysis (DMA) in both solid and molten state.[2] It is observed that the both E′ and E″ decrease with temperature in all composites compared to the neat PP in the solid state.[20] The storage modulus (E′) difference between the biocomposites and PP at -50°C was found to be only 9%.[1] On the other hand, G′ and G″ were found to increase with the entire angular frequency range. The storage modulus at 0.1rad/s of the neat PP is 732 Pa, while the sample with 1wt.% of nanofiller loading shows storage modulus 850 Pa in the molten state.[20] Overall, these composites demonstrated good dimensional stability in a given temperature range and frequency in the solid state, as well as typical viscoelastic behavior of entangled polymeric liquid in the molten state.

Keywords: Polypropylene, Date Pam Fiber, Nano Filler, Composite, Dynamic Mechanical Analysis, Thermomechanical Analysis.

1.[1] Introduction

An increasing focus on polymer composites based on natural fibers/fillers has been stimulated by the necessity of low carbon generation, sustainability consideration, combined with high performance that can be utilized for high engineering applications. Natural fibers/fillers reinforced polymer composites offer great potential in automotive, aerospace, prosthesis, sports and ballistic applications where light weight and high resistance are crucial parameters (Al Rashid, Khalid, Imran, Ali, & Koc, 2020; de Queiroz, Banea, & Cavalcanti, 2021; Malalli & Ramji, 2022; Mansor, Nurfaizey, Tamaldin, & Nordin, 2019; Marichelvam et al., 2021; Nurazzi et al., 2021). In fact, all major automotive manufacturers, including Toyota, Ford, Audi, Volkswagen, Volvo, Porsche, and McLaren, now using natural fiber reinforced polymers in non-structural body components for vehicles such as the underroof, instrument and door panels, sun visor, seat backs, oil air filters, boot liners, internal engine covers, and preforms (J. S. Neto, de Queiroz, Aguiar, & Banea, 2021).

Generally, plant fibers are classified based on the parts of the plant from which they are isolated, for example, bast, straw, leaf, seed, fruits and wood fibers. Several types of plant fibers such as flax, jute, sisal, kenaf, oil palm are already being well established and used in a various application. Additionally, the academic community continues to investigate other waste crop residues as fillers to determine their suitability for polymer reinforcement. One example is the date palm plant which is abundantly available in arid regions, and produces bast fibers that have been used for household applications since centuries. Aside from this, its high cellulose content, durability, and good tensile strength, make it unique plant parts (Faiad et al., 2022). Nonetheless recent studied have demonstrated that date palm agro residue can be used in high engineering applications due to their competitive technical characteristics (Benzidane et al., 2022).

[8] However, the molecular properties of natural fibers/fillers derived from any lignocellulosic source differ considerably due to heterogeneity caused by the factors such as origin, age, and part of the plant, crystallinity of the cellulose component, amount of lignin, hemicellulose, and other inorganic contents such as ash (Seki, Selli, Erdoğan, Atagür, & Seydibeyoğlu, 2022).[8] Therefore, filler's chemical and/or physical modification are also required in order to achieve good thermal and mechanical properties from composites based on natural filler. Additionally, various compatibilizers are utilized to increase the bonding between the filler and matrix.[8] On the other hand, the molecular and microscopic characteristics of the polymer matrix can be altered to varying degrees by the addition of these fillers. Similarly, among commodity polyolefins, polypropylene has appealing attributes such as low cost, a wide processing temperature range, thermo-oxidative stability, and resistance to various chemicals. A review has been published by Faruk et al.(Faruk, Bledzki, Fink, & Sain, 2012) on bio composites reinforced with natural fillers including treatment/modification of the natural fiber to be incorporated within polymer matrix. This indicate that effort to develop bio composite materials with improved performance for global application is ongoing process.[0]

Moreover, analyzing and validating the characteristics and performance of natural filler-based composites are crucial, especially when such composites are subjected to periodic stresses such as damping.[0] It is well known that dynamic mechanical analysis is widely used for determining such properties as a function of temperature, frequency and time by applying sinusoidal force on composite materials.[1] The modulus of the polymer is divided in to storage modulus (E´) and loss modulus (E´´).[28] As temperature increases, the E´of polymers decreases while the E´´and damping factor (tan δ) increases to the glass transition (Tg) point (Seth, Aji, & Tokan, 2018).[17] The stiffness of the particular composite of polymer is known to responsible in decreasing E´ while E´´is associated with energy dissipation induced by the viscous portion of the polymer (Haris et al., 2022a). However, various factors such as composite's morphology, heterogeneity, morphological transformation and relaxation causes internal molecular friction of the molecular chains and thereby effects dissipation of energy (J. S. Neto et al., 2021).[1] Similarly, the internal mobility of polymeric molecular chains is correlated with the damping factor (δ), which is calculated by dividing the storage and loss modulus (tan δ=E"/E'), which demonstrates the impact of the filler/matrix interactions.[8] A low tan delta value suggests lower mobility of the polymer chain indicating good filler-matrix interaction, while high tan delta value indicates that the more energy is being dissipated than stored in the composite system (J. Neto et al., 2019).

To determine how thermoplastic and thermoset composites perform in terms of thermomechanical performance, numerous amounts of research have been conducted in the past. This study, however, will focus primarily on lignocellulosic biomass based filler and polypropylene matrix composites. Mohd Izwan S et al., (S. Mohd Izwan, S. Sapuan, M. Zuhri, & A.[1] Mohamed, 2021) investigated the dynamic mechanical and thermal stability of polypropylene composites reinforced with chemically treated sugar palm and kenaf fiber (first alkalization, then benzoylation). These fibers were melt blended with polypropylene in varying ratios up to 20wt. % to construct hybrid composites. It was shown that an equal proportion of these fibers, whether treated or untreated, results in the highest E´in a PP composite.[20] The PP composite with untreated fibers have E´´of 1300MPa, 958Mpa, and 695 at 20°C, 40°C, and 60°C, respectively, while treated fiber composites have storage moduli of 1360MPa, 991Mpa, and 711 at the same temperature range. Furthermore, the loss modulus (E") of the same composites with untreated and treated fiber is 80.7Mpa and 86.2 MPa, respectively, while the damping factor (δ) was reported to 0.0585Pa and 0.0531 Pa.

In another work, a polypropylene-based composite was made by mixing 5% to 20% Carpinus betulus L. (CB) powder.[2] It was found that increasing the weight fraction of the filler enhances the stiffness of the composite, as evidenced by the composite with the maximum storage and loss modulus values having 20 wt.% CB (Atagur et al., 2020). In another study, hybrid composites were generated using a secondary filler system that included halloysite nanotubes (HNTs) in a sisal fiber/polypropylene system. Short sisal fiber was treated with alkali and high intensity ultrasound before being combined with halloysite nanotubes (HNTs) and polypropylene by internal mixing and compression molding. It has been stated that a blend of 10% sisal fiber and

6% HNT filler demonstrates a significant improvement in E´as analyzed by dynamic thermal analysis (Krishnaiah et al., 2020).

Another study investigated the dynamic mechanical characteristics of polypropylene biocomposites composed of alkali and silane treated bagasse microfibers (Hidalgo-Salazar, Luna-Vera, & Correa-Aguirre, 2018). The E' and loss modulus E" decreased to 55% and 52%, respectively, as the temperature rose from 25°C to 75°C.[2] Furthermore, the increase in the value of α relaxation and the stability of E' with temperature was linked in this work to bagasse fiber, that improves the stability of the storage module of the PP matrix with the temperature. In a similar work, Doum palm shell particles were reinforced with polypropylene and their dynamic mechanical assessment was performed.[0] In this case also, increased modulus and decreased tan delta value were ascribed to effective interfacial adhesion between matrix and particles, which causes restriction of polymer chain mobility and offers the composites thermal stability (Seth et al., 2018).

In contrast, thermomechanical analysis (TMA) is another methodology for examining how a composite material's dimension vary when it is subjected to both temperature and a fixed load. [27] Besides this, it can also be used to study volumetric changes, molecular structure, surface roughness, cross-linking polymerization under dynamic and static loads, and curing of polymers and composites.[0] The coefficient of thermal expansion (CTE) of a hybrid composite made of polypropylene and treated sugar palm-kenaf fiber with various ratio was also examined by Mohd Izwan S et al., (S Mohd Izwan et al.,[23] 2021) In order to determine the CTE from the linear slope of the strain-temperature curve, the specimen of these composites was heated from -50 °C to 100 °C with 0.05 N loading of the probe.[0] It was stated that, porosity of the samples began to shrink during the testing when an external load was applied in the axial direction with temperature, and the samples displayed deformities in various phases. Consequently, it was reported that thermal expansion coefficient (CTE) of the PP composite reinforced with an equal amount of chemically treated fibers at 45°C was 7.32 as compared to 3.21 for untreated fibers. Furthermore, the thermal expansion coefficient rose from 24.93 to 30.11 at 105°C for untreated and treated fibers, respectively. At lower temperature, minor changes in CTE were linked to transition state and moisture evaporation, which prohibited excessive expansion of the composite, however at higher temperature, significant increases were reported owing to melting of composite.

[2] In another study, the coefficient of thermal expansion (CTE) of a polypropylene/rice husk composite was determined by adding up to 50% of the filler.[0] The CTE of pure PP was reported to be 123 x 106/°C in the temperature range of 10 to 50°C, increasing to 163 × 10−6/°C in the temperature range of 50-100°C. These values were reported to decrease by 30-62 % percent after addition of the 50wt.% of the filler. A similar observation was made with the PP-based composite material, where the thermal expansion decreased as amount of reinforcement increased (Atagur et al., 2020; Doan, Brodowsky, & Mäder, 2016; Reixach et al., 2015a).[90]

The significance of this study in this context is, in the development of biocomposites/green composites reinforced with the least amount of date palm nanofillers in polypropylene. This filler was made using a simple mechanical approach, avoiding the use of hazardous chemicals for the treatment process and consequently eliminating the multi-step purification step (Alothman, Shaikh, Alshammari, & Jawaid, 2022).[40] To the best of our knowledge, no relevant research on the thermo-mechanical characteristics of date palm nanofiller/polypropylene composites has been reported.[40] The information gleaned from these analyses could make it conceivable to comprehend, how the reinforcing of such fillers influences the characteristics of the polypropylene matrix, that can be advantageous in outdoor applications in dry climate with significant temperature variation.

2. Experimental

2.1.[16] Materials

The homopolymer polypropylene (PP 3030, melt flow index (MFI) of 3 g/10 min and a density of 0.9 g/cm^3) obtained from the National Industrialization Company (TASNEE) was utilized to formulate biocomposites. Maleic anhydride grafted polypropylene compatibilizer (PRIEX-25097) was purchased and used from Addcomp, The Netherland. Similarly, the nano size filler was also produced using planetary ball milling with 99 cycles, each lasting 15 minutes at 300 RPM (Pulverisette 7 Premium, Fritsch Co. Germany). Fig. 1 display the scanning and transmission electron microscopy (SEM and TEM) images of these nanofillers. These fillers typically had a diameter of 30-50nm and a length of 1-10mm.

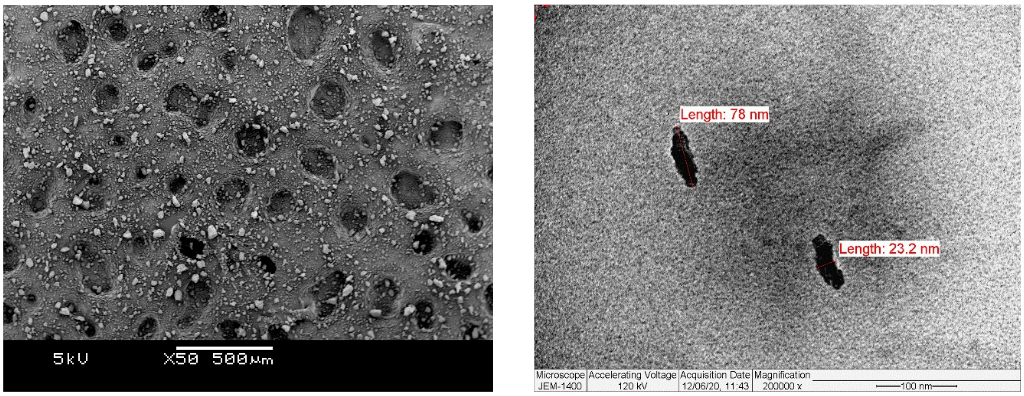

Fig. 1. SEM and TEM of date palm nanofillers (DPNF)

2.2. Preparation of Composites.

The dried date palm nanofillers in the range of 1-5 wt.% loading and polypropylene modified maleic anhydride compatibilizer (PRIEX-25097, Addcomp, The Netherland) were melt blended with the polypropylene by using twin screw melt compounder (DSM Xplore micro-compounder, 15 cm^3, The Netherlands).[16] A melting temperature of 200°C, a screw speed of 100 rpm, and a mixing period of 10 minutes was kept in a speed-controlled mode. This molten mass was then collected for injection molding to make standard specimen. These specimens are termed as PP/NFD-1, PP/NFD-2, PP/NFD-3, PP/NFD-4, and PP/NFD-5, where the number reflects the nanofiller's loading percentage. Table 1 tabulated formulation of composites and used for thermomechanical characterization.

Table 1. List of biocomposites produced in this work

Sample ID.	Composition (wt. %)-
Neat PP	0% DPNF (0) + PP (100)X
PP/NFD-1	1% DPNF (1) + PP (97)X+ PRIEX (2)
PP/NFD-2	2% DPNF (2) + PP (96)X+ PRIEX (2)
PP/NFD-3	3% DPNF (3) + PP (95)X+ PRIEX (2)
PP/NFD-4	4% DPNF (4) + PP (94)X+ PRIEX (2)
PP/NFD-5	5% DPNF (5) + PP (93)X+ PRIEX (2)

2.3. Characterization of the composites

2.3.1.[18] Thermo-Mechanical Analysis

Thermal mechanical analysis (TMA) was carried out according to ASTM D696 using a TA instrument Q400, under nitrogen with rate 50mL/min, in the temperature range from 30°C to 160°C at a heating rate of 5°C/min. The dimensions of TMA sample are 5 × 5 × 3 mm^3.[28] The objective was to investigate the dimensional changes in the thickness direction to evaluate the CTE of the composites.

2.3.2.[0] Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) was conducted according to ASTM D4065-01, as a function of temperature, on solid samples with dimensions of 60 × 12 × 3 mm^3 to determine the viscoelastic behavior PP/NFD composites.[30] DMA tests were performed using a TA (DMA Q 800) instrument, operating in the three-point bending mode and frequency 1 Hz, and the temperature was ramped from -50°C to 150°C with a heating rate of 5°C/min. Furthermore, melt viscoelastic properties of these composites were also analyzed by AR-G2 Rheometer (TA instruments, USA).

The samples were compressed into a disc that fits inside the rheometer's circular plates.[8] First, linear viscoelastic measurements were carried out for melt at 190°C using a parallel plate geometry with a diameter of 25 mm and a gap of 1000 µm.[8] Dynamic frequency sweeps were performed within the linear viscoelastic region of the materials, ranging from 0.1 to 100 rad/s. To maintain linearity, the strain was kept constant across the frequency range. This strain was selected from a dynamic strain sweep test, which was performed from 0.[4]01–100% strains at a fixed frequency of 1 Hz and the deviation strain from linearity was tracked.[24] The frequency sweep test was performed at constant temperature.[52] Additionally, time sweeps were also performed to ensure that no thermal degradation taking place and the material is stable during the length of measurement. Each measurement was performed on a fresh sample and repeated measurements had been conducted to ensure the reproducibility of the experimental results.

3. Results and Discussion

3.[58]1 Coefficient of thermal expansion

The dimensional stability of PP/NFD biocomposites and neat PP can be evaluated through the estimation of the coefficient of thermal expansion (CTE). Fig. 2 showed the dimension change vs temperature of the biocomposites and neat PP. It was showed that the dimension changes of the biocomposites and neat PP increase as temperature increase. The coefficient of thermal expansion at temperature 40℃ and 80℃ biocomposites and neat PP was tabulated in Table 2. Among the biocomposites addition of 5wt% of nanofillers shows the slightly highest CTE compared to other biocomposites sample.[0] In general, is observed that addition of NFD have negligible effect on the CTE of the biocomposites throughout the temperature range. This showed that the cellulose filler maintained the composite's dimensional stability by reducing significant thermal expansion. It also denotes an effective filler-reinforcing effect, minimizing shape change and warping effect, and therefore these composites can be employed for dimensional stability while subjected to thermomechanical stress. However, Reixach et al (Reixach et al., 2015b), on the other hand, observed a contrasting behavior for other polypropylene wood-based composites.[2] They reported that as the amount of wood fiber in the PP composites increased, the thermal expansion decreased.[0] This behavior was attributed to the lower coefficient of expansion of the reinforcement compared to that of the matrix. Similarly, Mohd Izwan et al. (S. Mohd Izwan, S. M. Sapuan, M. Y. M. Zuhri, & A. R. Mohamed, 2021) found that the CTE of sugar palm/kenaf fiber reinforced PP at 45℃ was much lower than that of neat PP. The authors observed that when the temperature raised to 105℃, a huge expansion occurred for these composites. They attributed this increase in CTE to the melting state of the bio composites. Similar conclusion has reported by Poletto et al. (Poletto, 2018)

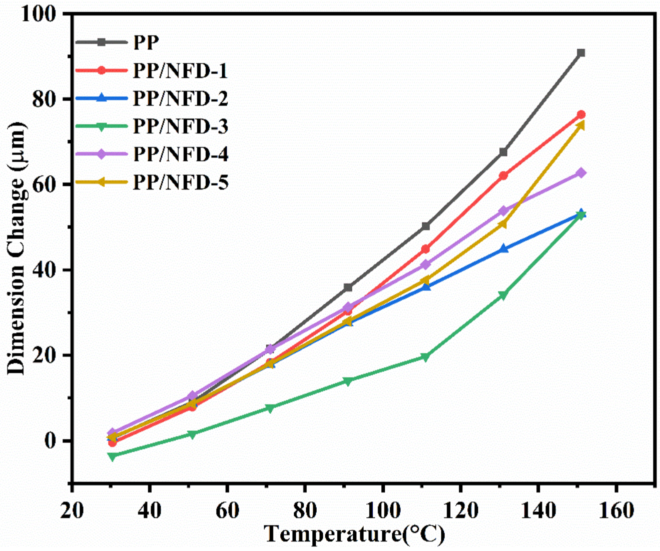

Fig.2. Coefficient of thermal expansion (CTE) of nano date filler-based PP composite

Table 2: The coefficient of thermal expansion at temperature 40℃ and 80 ℃biocomposites and neat PP

Sample ID.	CTE at 40℃	CTE at 80℃
Neat PP	80.4	124
PP/NFD-1	85.9	120
PP/NFD-2	89.4	128
PP/NFD-3	88.4	119
PP/NFD-4	83.5	121
PP/NFD-5	95.4	138

3.2. Dynamic Mechanical Analysis

3.2.1.[2] Viscoelastic response of in the solid state (Storage modulus, E´)

The effect of palm date nanofiller (NFD) on the viscoelastic response of the polypropylene in the solid state was also investigated through dynamic mechanical analysis (DMA). Fig.[1]3 illustrated the effect of NFD on the storage modulus (E´) of the polypropylene composite. It is observed that, incorporation of nanofillers showed slightly improvement in E´of polypropylene. Table 3 showed the E´of the PP/NFD biocomposites and neat polypropylene at -50 ℃ (glassy region) and 150℃ (rubbery region). It worth to mention that storage modulus, however, decreased as the temperature increased. As temperature rises, the E'of composite decreases, which is caused by an increase in molecular mobility and free volume. Nonetheless, temperature has a significant impact on molecular mobility and free volume as molecules absorb more energy at higher temperature. When this energy becomes comparable to the energy barriers, large-scale conformational rearrangements of molecules can occur.[20] This region is referred to as the rubbery region, and has an E' value that is lower than that of the glassy and transition regions.[3] Moreover, at higher temperature (rubbery region) the modulus of the composites are dominated by the intrinsic matrix modulus (Karamipour, Ebadi-Dehaghani, Ashouri, & Mousavian, 2011; Majid, Hassan, Davoud, & Saman, 2011). As a result, there are no significant different in E´for PP/NFD biocomposites are observed. Similar trends of viscoelastic properties of polymer reinforced with different natural fiber have been reported (Hansen, Borsoi, Dahlem Júnior, & Catto, 2019; Haris et al., 2022b; Joseph, Mathew, Joseph, Groeninckx, & Thomas, 2003).[4] However, various other factors such as fiber and matrix type, the presence of fillers, fiber content, and fiber orientation, the chemical treatment of the fibers, amount of loading, and size effect the final properties of composite materials.

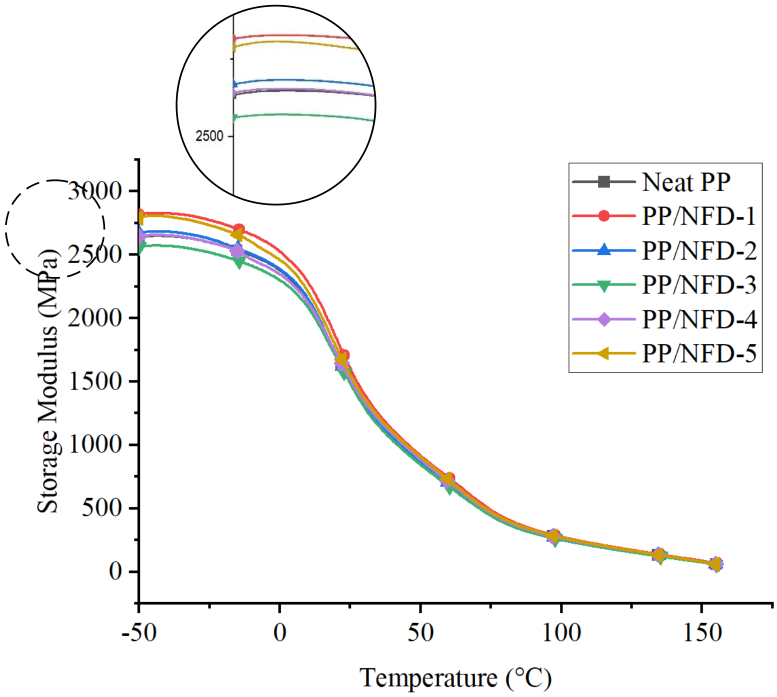

Fig.3. Storage modulus(E') of nano date filler-based PP composite

Table 3: The storage modulus of the NFD/PP biocomposites and neat polypropylene at glassy region and rubbery region

Sample ID.	Storage modulus at Temperature -50℃, E'_G (MPa)	Storage modulus at Temperature 150℃, E'_R (MPa)	Percentage difference in storage modulus compared to PP at Temperature -50℃ , E'_G (MPa)
Neat PP	2631	79	-
PP/NFD-1	2812	85	6.87
PP/NFD-2	2864	76	8.58
PP/NFD-3	2758	74	4.82
PP/NFD-4	2739	80	4.10
PP/NFD-5	2783	79	5.78

3.2.2. Analysis of Loss modulus, (E'')[31] and damping Factor (Tan δ)

Similarly, the loss modulus and tan delta of PP/NFD biocomposites along with neat PP are depicted in Fig. 4 and Fig. 5 respectively. It is observed that biocomposites with 1wt.% filler loading shows a slight improvement in loss modulus compared to neat PP, whereas other samples display no significant difference in loss modulus. Also, the tan delta follows the same pattern.[1] The dynamic mechanical characteristics of several natural fiber-based PP composites have been examined, and it was shown that a decrease in tan delta value results in more elastic nature of composites than pure PP matrix (Tajvidi, Falk, & Hermanson, 2006).[17] The decreased in the delta value is attributed to the reduced viscoelastic lag between the stress and the strain (Han, Han, Cho, & Kim, 2008).[1] The glass transition temperature of the biocomposites and neat PP can be determined from the peak of loss modulus and tan delta.[32] Table 4 shows the glass transition temperature of the composites.[1] The result demonstrates that the glass transition of the biocomposites is not appreciably influenced by the addition of NFD.[81] The results of earlier research (Modesti, Lorenzetti, Bon, & Besco, 2006) that reported the addition of nano filler had no significant impact on the glass transition temperature also support this fact.[20] Additionally, a neat polymer and its composite may have a glass transition temperature similar due to a neutral or weak interaction between the fillers and polymer matrix (Bashir, 2021).[0] Typically, the glass transition temperature determined using tan delta is 5°C-10°C higher than the loss modulus.[1] The tan δ values were lowered in the biocomposites compared to the PP matrix because of less matrix by volume is available to dissipate the vibrational energy.

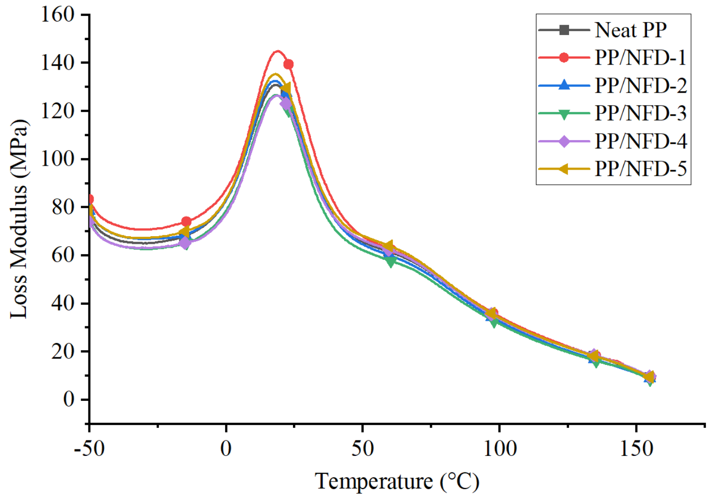

Fig.4. Loss modulus(E”) of nano date filler-based PP composite

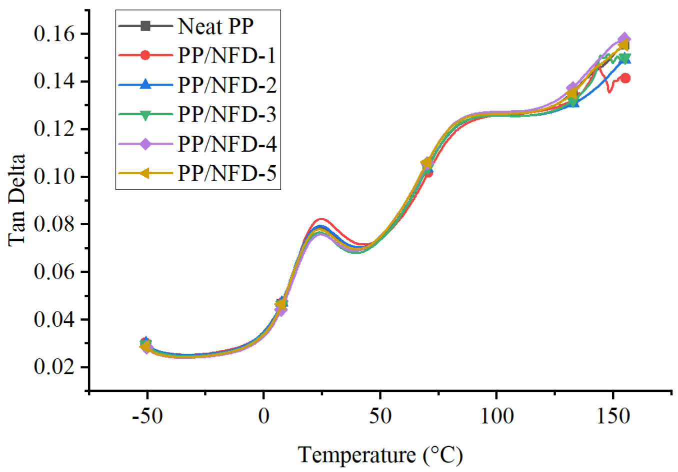

Fig.5. Damping factor/Tan (δ) of nano date filler-based PP composite

Table 4: The glass transition temperature extracted from the peak of loss modulus and tan delta

Sample ID.	Tg from Loss modulus (℃)	Tg from Tan delta (℃)
Neat PP	18.28	24.07
PP/NFD-1	19.04	24.63
PP/NFD-2	17.75	24.31
PP/NFD-3	18.00	23.86
PP/NFD-4	18.78	24.91

PP/NFD-5	18.13	23.98

3.3.3. Viscoelastic response of in molten state (Storage modulus, G´)

The molten polymeric fluid's elastic response is measured by its storage modulus(G'). The storage modulus denotes the capacity to store energy applied by external forces, and it increased with angular frequency over the entire applied frequency range. Fig. 6 displays the variation between the storage modulus (G′) and the frequency. The storage modulus (G′) of the composites filled with 1 wt.% loading (PP/NFD-1) of natural fillers shows slightly higher modulus at lower frequency than the neat PP. The storage modulus at 0.1rad/s of the neat PP is 732 Pa, while the sample PP/NFD-1 show storage modulus 850 Pa, and at 1 rad/s it is 5524 Pa, and 5700 Pa respectively.[8] This behavior could be due to the more interaction between the fillers and the matrix, which caused increased elasticity in this system compared with remaining composite samples.[3] It has been well established fact that the poor compatibility of filler and the matrix could decrease of both storage modulus and loss modulus.[3] The rest of the samples, on the other hand, had a lower storage modulus than the neat PP across the whole frequency range.[53] These results indicate that the storage modulus changes as a result of the formation of the polymer-filler network. The difference in storage modulus between the composites reduced as the frequency rose. These might occur as a consequence of the particles' inherent stiffness or aggregation.[62] It should also be noted that the inclusion of the filler may cause variations in the relaxation time spectrum, resulting in changes in the composites' viscoelastic characteristics. According to Marcovich et al. (Marcovich, Reboredo, Kenny, & Aranguren, 2004), a composites sample comprising 50% wood flour reinforced PP matrix exhibited solid-like behavior at low frequency. [2] Furthermore, it was reported that the presence of the wood filler had no effect on the relaxing mechanism of the PP. However, when the wood filler loading increases the corresponding relaxation times also increased.

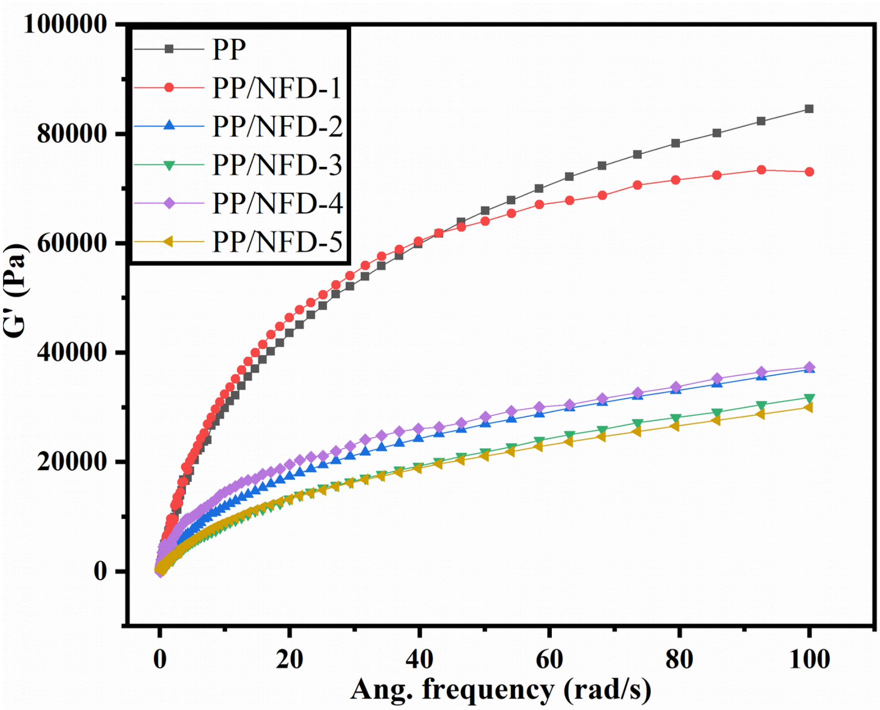

Fig.6. Comparison of composite's storage modulus with frequency

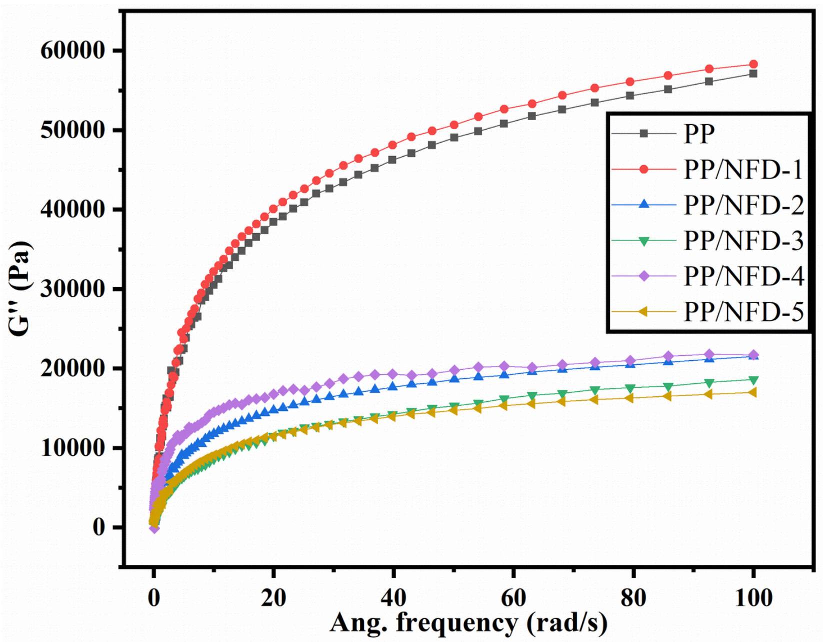

Fig.7. Comparison of composite's loss modulus with frequency

3.3.4. Analysis of Loss modulus, (G'')[33]

As stated earlier, the loss modulus related to the energy dissipation in the system. The loss modulus variation along with the frequency is shown in fig 7.[4] The loss modulus increased with increase in frequency. At 0.1 rad/s, the loss modulus of neat PP is 1703 (Pa), 1453 Pa for PP/NFD-1, 677 Pa for PP/NFD -2, 262.6 Pa for PP/NFD -3, 495.2 Pa for PP/NFD -4 and 390.3 Pa for PP/NFD -5 respectively. The discrepancies in loss modulus between these composites can be related to differences in particle size distribution. For all samples, the loss modulus appears to rise linearly with frequency. The loss modulus differences amongst the composites remained almost constant as the frequency increased. As previously stated, a larger particle concentration resulted in more particle-particle interactions. Even at high shear rates, this impact remained consistent as the shear rate increased. It was therefore concluded that both the storage module (G′) and loss modulus (G′) shows the less frequency dependency characteristic. It is clear that G' G" and it has been usually interpreted as the condition at which the natural filler particles are connected throughout the PP composite sample (Ren et al., 2014). Similarly, fig.[91] 8 depicts the tan δ (which is the ratio of loss modulus (G″)/storage modulus (G′)) vs. frequency of the biocomposites. In general, all these samples show a solid-like behavior with G″ ≫ G′ and with almost similar tan δ value at lower frequency. They exhibit a solid-like behavior even at higher frequencies (which correlate to shorter relaxation time), and both PP and biocomposites with only a slight change in tan δ values. Furthermore, the rheological threshold in composites is observed when the tan δ become equivalent at higher and lower frequency at particular filler content (Poulose et al., 2018).

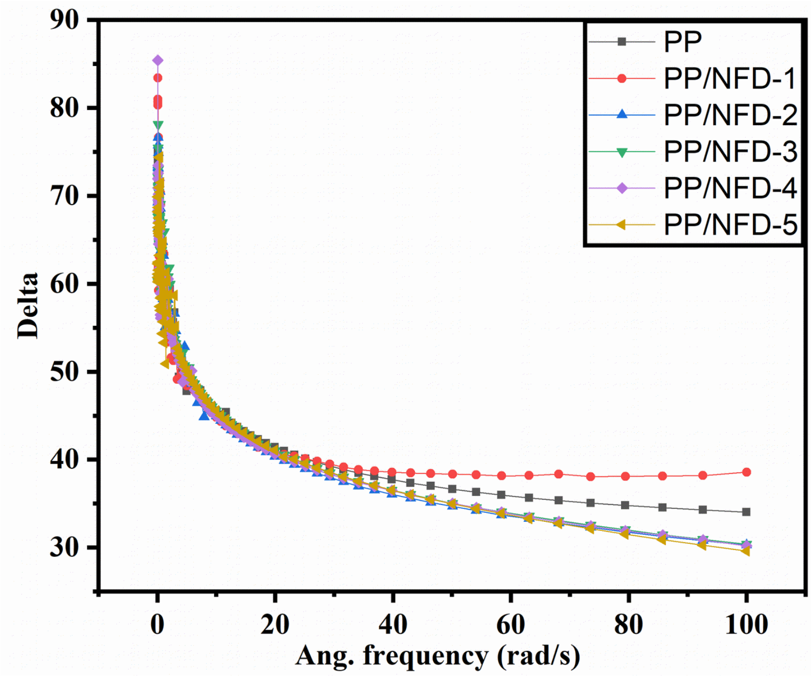

Fig.8. Tan (δ) of composite with frequency (ω)

4. Conclusion

In this work, we successfully formulated biocomposites of polypropylene based on date palm nanofillers. The industrial viable process such as melt blending method was used to make these composites. These composites then analyzed by their thermomechanical characteristics.[16] Because the thermal stability of these composites is found to be stable, they can be used for dimensional stability when subjected to thermomechanical stress. Moreover, dynamic mechanical analysis in solid state reveled that these biocomposites shows improvement of storage (E') modulus at 50℃ which is much higher than the room temperature. Their fore such composites can be useful in formulating household items in arid climate.[1] The Tg values obtained from loss modulus and tan delta was found to remain fairly constant.[16] Additionally, rotational rheometery was used to examine the rheological properties of these composites in the molten state. The sample with 1wt.% of loading shows highest storage modulus (G') and loss module (G”) with the frequency rates compared to other samples. The less frequency dependency characteristics are observed for the storage modulus (G') and loss module (G”) with the frequency rates. The frequency independence of storage modulus (G′) and loss module (G”) shows an elastic solid (solid-like) behavior of these biocomposites. Thus, our findings demonstrated that these biocomposites are thermomechanically stable and can be effective in dry climates.

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