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A Study of the Effects of Time of Heat Setting and Wet Processes on Shearing (Gf/Cm) Properties of Treated and Untreated Griege Knitted *Ingeo*™ Poly (Lactic Acid) (Pla) and Polyethyleneterepthalate (Pet) Fabric

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Abstract

This paper comparatively evaluated the effect of wet processes and heat setting on the shearing properties of treated and untreated griege *Ingeo*™ Poly Lactic Acid (PLA) and Polyethylene Terepthalate (PET) knitted fabrics. The treated samples were subjected to wet processes including scouring, dyeing, alkaline reduction clearing and softening. The KES-FS was used in measuring shearing rigidity ($G_1.G_2$) gf/cm. degree, shear hysteresis at 5%, shear angle ($2HG_1$, $2HG_2$) and shear angle at 5% ($2HG5_1$ and $2HG5_2$) of treated and untreated knitted *Ingeo*™ Poly (lactic) acid and Polyethyleneterepthalate fabrics in warp (G_1) and weft (G_2) directions. PLA samples of dimension 200 mm×200 mm were subjected to heatsetting at temperatures of 130˚C respectively and increasing times of 15 s, 30 s, 45 s, 60 s, 90 s, 120 s and 240 s using the Werner Mathis infra-red heatsetting equipment. Results showed that for shear rigidity, the control untreated PET exhibited a higher property both before and after undergoing wet treatments and heatsetting treatment. But for $2HG_1$ and $2HG_2$ the untreated griege PET exhibited a higher $2HG_1$ and $2HG_2$. The reverse became the case after wet treatment and heatsetting where PLA increased in both properties with increasing time of heatsetting and wet processes. G-Shear Rigidity, gf/cm.degree indicated the ease with which the fibers slide against each other resulting in soft/pliable to stiff/rigid structures. Large hysteresis means greater recovery forces will be required to overcome fabric internal friction. Smaller values of $2HG_5$ indicates good comfort and softness. Too large values of $2HG_5$ indicates inelasticity and stiffness.

Keywords: *Ingeo*™ Poly (lactic acid); KES-FB system; Shearing rigidity (gf/cm); Shearing hysteresis (gf/cm)

Introduction

Polylactic acid is aliphatic polyester based on renewable materials which have been widely applied industrially. The most outstanding characteristics of Polylactic acid are its biodegradability. PLA can be degraded to carbon dioxide and water completely in a natural environment without pollution. This implies that biodegradable PLA fibers are eco-friendly. Another attractive attribute of PLA is its abundance and availability. PLA can be produced from the polymerization of lactic acid [1-5] obtained from the fermentation of corn, sugar and vegetables, and does not require any fossil fuels. Lactic acid is one of the simplest chiral molecules and exists as the two stereo isomers, L- and D- Lactic acid. The L form differs from the D form in its effect on polarized light. For L- lactic acid the plane is rotated in a clockwise (dextro) direction, whereas the D form rotates the plane in anticlockwise (laevo) direction. Ingeo PLA is a melt-processable natural based fiber, high resilience, low flammability, low smoke generation, excellent UV stability, high resilience, excellent wicking, moisture management and comfort properties, compostable under appropriate conditions and low odor retention [6-10]. PLA is aliphatic polyester having no aromatic ring structure, but the yarn properties of PLA are relatively similar to those of PET. However, the density and refractive index of PLA are relatively lower to those of PET. PET fibers can be produced from terepthalic acid obtained by processing of benzene and ethylene glycol obtained from ethylene. The polymerization reaction occurs in a vacuum at high temperatures releasing water as a byproduct. This is followed by the melt extrusion process into staple, filament or tow form [11-15]. Polyester fibers are used in woven and knitted fabrics for apparel and household fabrics and in the construction of functional articles such as car seat belts, nets, ropes and fiber-fill. They are also applied in medical textiles [16-20]. PET exhibits very good mechanical properties. The melting temperature of PET lies between 254°C and 260°C [20-25]. PET exhibits a high light resistance, UV and high abrasion resistance [26-30]. When properly treated PET fabric exhibits good dimensional stability, crease resistance and solvent resistance. PET when compared with other synthetic fabrics exhibits hydrophobicity and relative stability to chemicals and processing conditions [30-35]. At 65% r.h and 25°C, PET exhibits a good moisture regain of between 0.2-0.4%. The dyeing process of PET initiates with dissolving of the dye in water and subsequently by the transfer of the dye molecules from the solution to the fiber surface. This is immediately followed by the replenishment of the dye bath solution by dissolution o sold material from the dispersion. Diffusion of the absorbed dye into the fiber is the final stage. Polyester fabrics may be contaminated with surface deposits of unfixed dyes after dyeing especially at heavy depths of shade. This could result in lowering of the color fastness of the dyed PET fabrics. The chemical treatment of the dyed fabric with caustic soda and sodium hydrosulphite (Na_2S_2OH) at 70°C around 10-15 minutes is an effective method for removing this unfixed dye from the surface of

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polyester fibers. Polylactic acid is hydrolytic sensitive and has a lower Tm (170°C) and hence cannot be dyed at high temperatures of 125°C -130°C normally used for aqueous phase dyeing of PET having Tm of 250°C. Therefore, lower dyeing conditions of 110-115°C for 15-30 min at pH 4.4-5 are recommended for Polylactic acid [36-40]. Disperse dyes generally behave differently on PLA fiber than on PET fiber [41] displaying lower exhaustion [42], with dyeing being brighter [43], of higher color yield [44] and with maximum wavelength occurring at a shorter wavelength than on PET [42]. The low sorption of disperse on PLA compared to PET has been explored [45] using solubility parameter concept [46] as well as modeling [45]; several researchers have sought to maximize dye uptake into PLA through the synthesis of specific disperse dyes. Scouring is an important process in the industrial wet processing of knitted PLA and PET fabrics. The essence of scouring is to remove impurities and surface contaminants. PLA is scoured typically at 60°C for 10 minutes. However, the glass transition temperature (T_g) of PLA is about 60°C and hence there is a high possibility that scouring could induce morphological changes in PLA especially as it occurs under wet conditions. Reduction clearing is a wet process of using caustic soda and sodium hydrosulphite ($Na_2S_2O_4$) to effectively remove dye which is unfixed at the surface of both PET and PLA fibers at 70°C within time duration of between 10 to15 minutes. This is because PET and PLA fabrics may be contaminated with surface deposits of unfixed dyes after the dyeing process, especially at heavy depths of shade since there is a tendency for water insoluble disperse dyestuff to aggregate into relatively large particles as the dye bath cools down to below 100°C [46]. The application of softeners introduces a level of softness which may not be attained by mechanical finishing or modification of fabric construction. The type of finish applied is a function of the end use application, fashion or individual preferences. Softening agents may be classified into Cationic, Anionic, Non-Ionics and Amphoterics. The ability of a softening agent to perform depends on the ionicity and formulation relative to parameters like compatibility with other finishes, effects on dyestuff and type of handle. Heat setting the fiber thus introduces enhanced dimensional stability to the fibers improving fiber morphology and orientation. The heat setting process is determined by the temperature, time of heat setting, the medium of heat setting (air, solvents or water) and the tension applied to the substrates during heat setting. The heat setting temperature should be higher than the maximum temperature of the subsequent wet processes such as dyeing and ironing temperature so as to ensure the fabric attains dimensional stability [47]. For PET, heat setting occurs at temperatures of 130°C-140°C in steam or 190°C 220°C in dry air in the presence of some tension and since T_g of PET is approximately 80°C, heat setting of PET above its T_g would allow the polymer structure attain dimensional stability [45]. The KES-FB system measures fabric properties at small loads that are equivalent to those the fabrics are subjected to at normal end use application. KES Shear Test measured the ease with which knitted PLA and PET both treated and untreated (control) fibers slide against ease other to determine properties such as softness, pliability to stiffness/rigidity. Shear properties are measures of inter-yarn friction forces and shows the stability of a fabric to withstand in-plane mechanical distortion. They were measured to a maximum shear angle of ± 8°. The samples were measured for two parameters namely Shear Rigidity G, gf/cm.degree and Shear Hysteresis at 0.5° shear angle (2HG) and at 5° angle ($2HG_5$), gf/cm.

Materials and Methods

Ingeo Poly Lactic Acid (PLA) and Polyethylene Terepthalate (PET) fabrics

The knitted Ingeo Poly (lactic acid) and Polyethylene fabrics used for this investigation was supplied by NatureWorks LLC, USA. Sixteen samples of pique knitted fabrics obtained from 150/144d Tex/filament PLA and PET were used for this study. The treated fabrics were subjected to wet treatments including scouring, dyeing, alkaline reduction clearing and softening processes after heat setting treatments at 130°C at increasing time duration of 15 s, 30 s, 45 s, 60 s, 90 s, 120 s, and 240 s respectively. The untreated 'pique' knitted fabrics were used as control.

Dye

The dye used for this work was Dianix Yellow C-5G 200% having chemical name of 1-Ethyl-1, 2-dihydro-6-hydroxy-4-methyl-2-oxo-3-pyridinecarboxamide and molecular formula $C_9H_{12}N_2O_3$. The formular weight is 196.2 and the chemical structure is shown below (Figure 1) (Table 1).

Experimental

Heat-setting procedure: The heat setting of knitted PLA and PET fabrics were achieved using the Werner Mathis AG (Textilmaschinen Niederhashi/Zurich heatsetting equipment). The samples of dimension 200 mm by 200 mm were held on the sliding aluminum frame at a constant length and heated in dry air at a constant temperature of 130°C which is the maximum temperature for stabilizing PLA and PET as recommended by Cargill Dow. The samples were pinned on the sliding aluminum frame pins and heat set for time durations of 15 s, 30 s, 45 s, 60 s, 90 s, 120 s and 240 s respectively. The essence of prolonged heatsetting of the fabrics was to ascertain the behavior of PLA at high heatsetting time duration. After heatsetting, the fabric samples were allowed to cool down at room temperature for 24 hours.

Scouring procedure: They heat setted PLA and PET samples of dimension 200 mm by 200 mm and total weight of 83 g were scoured in 450 mls of water using a Mathis LABOMAT Scouring equipment of rpm 55 revs/min for 20 minutes at 60°C in an aqueous solution containing 1.66 g/l ERIOPON R, a non-ionic detergent and 0.83 g/l sodium carbonate (soda ash). This process was carried out at a liquor ratio of 10:1 using a beaker at a continuous stirring. The essence of scouring all knitted fabrics is to extricate all knitting lubricants, oils, waxes, dirt and other forms of impurities before commencing subsequent wet processing operations like dyeing, alkaline clearing and softening. Scouring reduces any propensity for uneven dyeing, stains

C.I.Number	Commercial name	Strength	Molecular Weight
Disperse Yellow C-5G	Dianix Yellow C-5G (DyStar)	200%	196.2

Table 1: Characteristics of selected disperse dye.

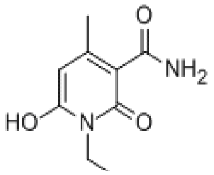

Figure 1: Chemical structure of Dianix Yellow C-5G 200%.

PLA Samples	1	2	3	4	5	6	7
Weight of Samples (g)	11.40	12.00	11.67	11.60	11.50	12.35	8.92
Weight of Dye (g)	0.23	0.24	0.23	0.23	0.23	0.25	0.20
Liquor Ratio	10:1	10:1	10:1	10:1	10:1	10:1	10:1
Total bath (mls)	114	120	117	116	115	124	60

Table 2: PLA dye values.

and dye fastness through the removal of oils, waxes and fats that may abide in the fabric. After scouring, they fabrics were rinsed with cold water and dried at room temperature.

Dyeing of knitted PLA and PET fabrics: Dyeing of PLA fabrics subsequently followed scouring, rinsing and drying. This took place at 110°C for 45 minutes using a laboratory scale Mathis LABOMAT Infra-red dyeing machine at a liquor ratio of 10:1 for each of the sample. The pH of the dye bath was maintained at 5 ± 0.1 through the application of acetic acid. 2% of selected disperse dye Dianix Yellow C-5G 200% was used though the quantity applied to each sample was calculated from the percentage weight of the fabric sample numbered from 1 to 7 for easy recognition and assessment. The total dye bath of each sample was also calculated from the weight of the fabric and liquor ratio. Table 2 below shows the individual values as determined from the experiment.

The Mathis LABOMAT Infra-red Uniprogrammer calibrations for the knitted PLA fabric are given in Table 3. The Dyeing procedure for PLA as represented by Mathis LABOMAT Infra-red equipment is shown in Figure 2. The dyeing quantities used in dyeing PET was determined the same method with those of PLA. 2% Dianix Yellow C-5G 200% was also used in dyeing PET at 130°C for 45minutes using acetic acid to maintain pH at 5 ± 0.1. The equipment used in dyeing was also Mathis LABOMAT infra-red dyeing equipment. The dye quantities as determined are shown in the Tables 4 and 5.

Uniprogrammer Calibrations PLA	Quantities
Rate of Temperature rise	30°C/min
Temperature	110°C
Time	45 min
Gradient (Rate of cooling)	5°C/min
Revolution/mm	50 rpm

Table 3: Mathis LABOMAT Uniprogrammer Calibrations for PLA.

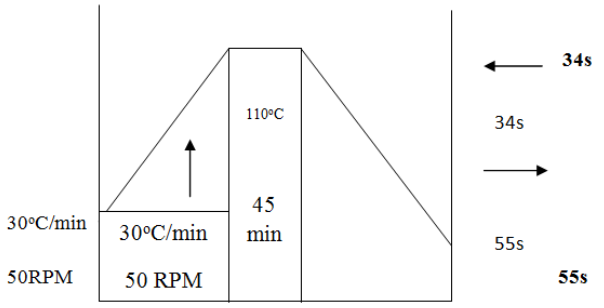

Figure 2: Mathis LABOMAT Uniprogrammer PLA dyeing procedure.

PET Samples	1	2	3	4	5	6	7
Weight of Fabric (g)	0.08	5.64	6.16	6.15	6.42	6	6.04
Weight of Dye (g)	0.12	0.11	0.12	0.12	0.13	0.12	0.12
Liquor Ratio	10:1	10:1	10:1	10:1	10:1	10:1	10:1
Total Bath (mls)	60.8	56.4	62	62	64.2	60	60

Table 4: PET dye values.

Mathis LABOMAT Uniprogrammer Calibrations for PET	Quantities
Start Temperature	20°C
Solution Temperature	20°C
Gradient	3°C/min
RPM	50 rpm

Table 5: Mathis LOBOMAT Uniprogrammer for PET.

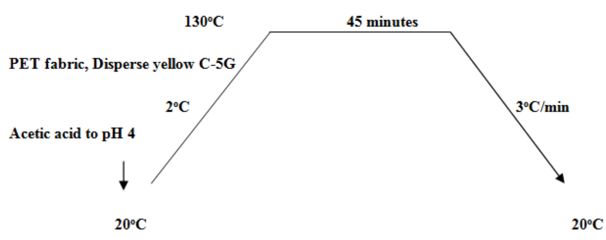

Figure 3: Procedure of disperse dye applied to PET.

Parameters	Quantities
Total weight of all samples	122.7 g
Sodium Hydroxide	12 g
Sodium dithionite	6 g
Warm water	2 Liters
Temperature of plate	70°C-80°C
Time	10-20 minutes

Table 6: Alkaline reduction Clearing Parameters.

$$\underset{\text{Colored}}{R_1 - N = N - R_2} \xrightarrow[\text{[H]}]{} \underset{\text{Colorless}}{R_1 - NH_2 + NH_2 - R_2}$$

Figure 4: Dye decolorization during alkaline reduction clearing process.

After dyeing, the PET fabrics were rinsed for 5 minutes using warm water and 2 minutes using cold water and subsequently dried at room temperature. A schematic representation of the dyeing procedure used for PLA and PET in this study is as shown in Figure 3.

Alkaline reduction clearing procedure: Alkaline reduction clearing is a process, which occurs after dyeing and air drying in order to extricate surface disperse dye [23-24]. All the samples used for this study were subjected to the same alkaline reduction clearing procedure. The quantities of chemicals used were calculated from a combination of the total weight of PLA samples. The quantities are shown in Table 6.

From the above table, alkaline reduction clearing of both Knitted PLA samples occurred within 70°C to 80°C for duration of 10-20 minutes. 6 g of Sodium dithionite and 12 g of sodium hydroxide were used to create enabling alkaline conditions needed for clearing to take place and for accurate comparative analysis. The efficiency of alkaline reduction clearing is a function of the chemical structure of the disperse dye [27-32]. When disperse dyes are treated with reducing agents, due to their azo group content, they are sensitive to treatment with a reducing agent usually in form of alkaline solution of sodium dithionite (hydros). The reducing agent destroys the azo chromophore, resulting to a loss of its color through the splitting of the azo chromophore into two colorless amino compounds [33-36] as shown in the Figure 4.

The softening agents [25-26] used in softening the PLA fabrics were Ciba® Sapamine® HS and Siligen CSM which were applied on the samples through padding using the Werner Mathis AG padding equipment calibrated at a pressure of 2 bar and roller speed of 2.5 m/min. The time of padding was 2 minutes at a temperature of 30 to 40°C. The two softeners were combined at 30 g/l whereby 3 mls of each were mixed with 200 ml of water to affect the softening process. The liquor ratio was 10:1 at a pH of within 5-6 sustained through the use of acetic acid. The liquor pick-up was about 90%. Ciba Sapamine is chemical composed of fatty acid ester, silicone, emulsion of fatty acid amide and polyalkylene. It is non-ionic/cationic in character with a pH of 4-5.5. Siligen® CSM is a hydrophilic silicone based softener, a registered trademark of BASF, composed of wax, polysiloxanes and non-ionic surfactants. After the padding process, the softened PLA fabrics were subjected to a drying procedure at a temperature of 110°C in 2 minutes using Werner Mathis AG equipment. The fabrics were then kept for storage for 7 days at room temperature and atmospheric pressure.

The Kawabata Evaluation System:

Shear tester [Model KES-FB1]: The specimen was clamped between two chucks each of 20 cm long. A constant force of 200 g was applied by attaching a weight to the specimen end on the front chuck. When the tests starts the back chuck constantly slides initially right to an angle of 8° and left to an angle of 8°, then back to its original position. The deformation rate was 0.416mm/s (Figure 5).

The parameters obtained were:

- **G-Shear Rigidity, gf/cm.degree:** This indicated the ease with which the fibers slide against each other resulting in soft/pliable to stiff/rigid structures.

- **2HG and 2HG$_5$-Shear hysteresis at 0.5° angle (2HG$_5$), gf/cm:** Measurements of energy loss is mainly caused by yarn to yarn friction at cross over points. Large hysteresis means greater recovery forces will be required to overcome fabric internal friction. Smaller values of 2HG$_5$ indicates good comfort and softness. Too large values of 2HG$_5$ indicates inelasticity and stiffness.

Fabric finishes generally reduces G and 2HG$_5$ values due to internal lateral pressure reduction and stress relaxation. Smaller contact area at yarn cross over points also reduces G and 2HG$_5$.

Too low values of G result to difficulties inlaying/cutting and handling due to fabric distortion. Too low values of G also cause difficulties in overfeeding, fullness creation and adversely affect drape. Fabric construction also affects G.

Results and Discussion

Shear rigidity [G$_1$.G$_2$], gf/cm.degree

The KES System determined the values of the given samples in weft and warp direction. The warp direction is represented as G$_1$ while the weft direction is represented as G$_2$. The average value of both directions was represented as G or mean.

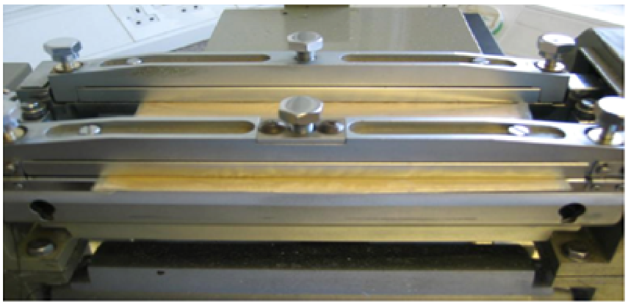

Figure 5: KES-FB Shear Tester [MODEL KES-FB1].

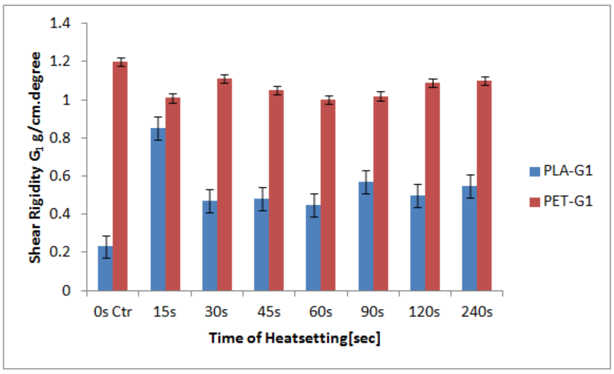

Figure 6: Comparative analysis of the effects of Heat setting and wet processing on Shear Rigidity G1 of treated and untreated knitted PLA and PET.

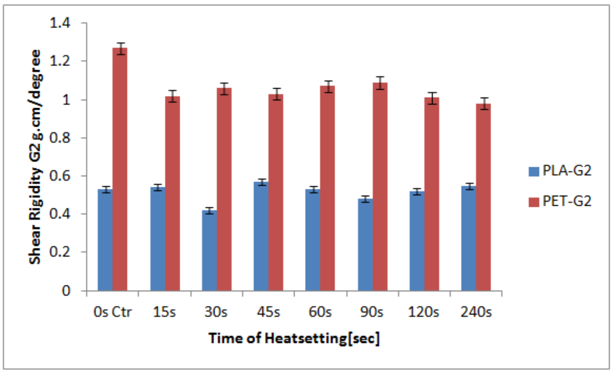

Figure 7: Comparative effects of increasing time of Heat setting and wet processing on G$_2$ of treated and untreated [Ctr] knitted PLA and PET.

Results from Figure 6 indicated that the control untreated PET exhibited a higher shear rigidity in warp direction G$_1$. After wet processes and heat setting treatments, PET continued with increased shear rigidity properties with increasing time of heat setting and wet processes. Though compared with the griege sample, PLA showed an increase in shear rigidity. This was the similar scenario in Figure 7 for G$_2$. This implies that PET exhibited a higher stiffness to PLA. Too low values of G will cause difficulty in laying/cutting and handling due to fabric distortion. Too low G values may lead to pucker as the extensibility in the bias direction becomes too high. Low values of G worsen Garment appearance. Too high values of G will cause difficulties in overfeeding and creating fullness and adversely affect drape. From the linear plot the G of PET increased with increasing time of heatsetting unlike that of PLA which decreased.

Figure 8 shows the mean of the effects on Shear Rigidity G with PET exhibiting a higher G. Fabric construction influences G. Loose fabrics have lower G because of a smaller number of yarn cross over points. Tight near jamming structures will have high G.

Figures 6 and 7 compared the Shear Rigidity G$_1$ and G$_2$ in warp and weft direction respectively of treated and untreated PLA and PET. Increasing time of Heatsetting and wet treatments increased values of G$_1$ and G$_2$ respectively. The values of G for PET remained higher than those of PLA for various times. The mean values are used for characterizing fabrics and Figure 8 showed PET exhibiting a high G

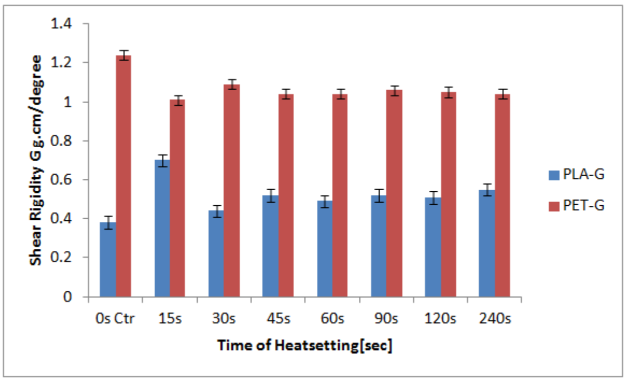

Figure 8: Comparative analysis of effects of heat setting and wet processing to mean Shear Rigidity G of treated and untreated knitted PLA and PET.

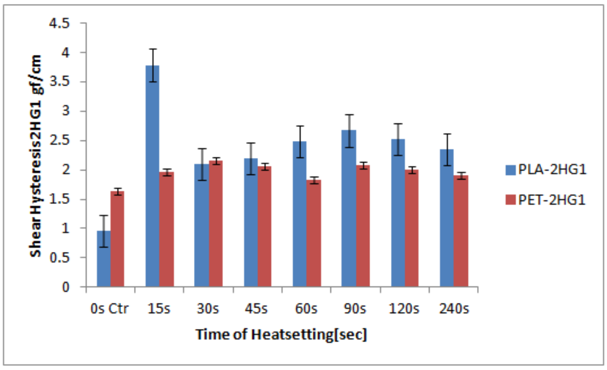

Figure 9: Comparative evaluation of the effect of increasing time of heat setting and wet treatments on $2HG_1$ of treated and untreated knitted PLA and PET.

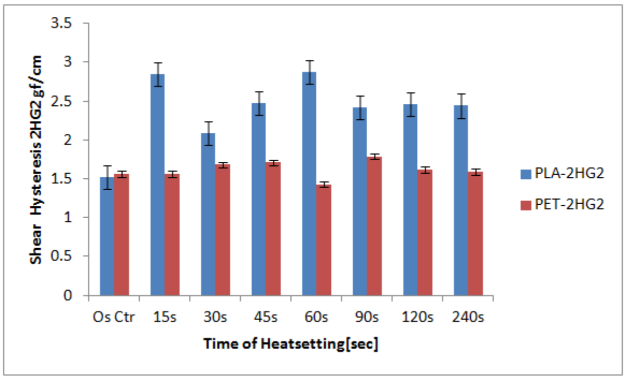

Figure 10: Comparative evaluation of the effect of increasing time of heat setting and wet treatments on $2HG_2$ of treated and untreated [Ctr] knitted PLA and PET.

unlike PLA. But increasing time of heatsetting reduced the values of G for PET and increased those of PLA.

Shear hysteresis at 0.5° shear angle ($2HG_1$, $2HG_2$) and at 5° shear angle ($2HG5_1$ and $2HG5_2$)

These parameters measures energy losses during shear deformation. These energy losses occur due to yarn to yarn friction at cross over points. Large hysteresis means greater recovery forces will be required to overcome fabric internal friction. The values obtained from KES evaluation for knitted PLA and PET are discussed below; Results indicated that knitted PLA has a higher Shear Hysteresis than knitted PET. These are mostly applied in hand value calculations. Measurements of energy loss is mainly caused by yarn to yarn friction at cross over points. Large hysteresis means greater recovery forces will be required to overcome fabric internal friction. Smaller values of $2HG_5$ indicates good comfort and softness. Too large values of $2HG_5$ indicates inelasticity and stiffness.

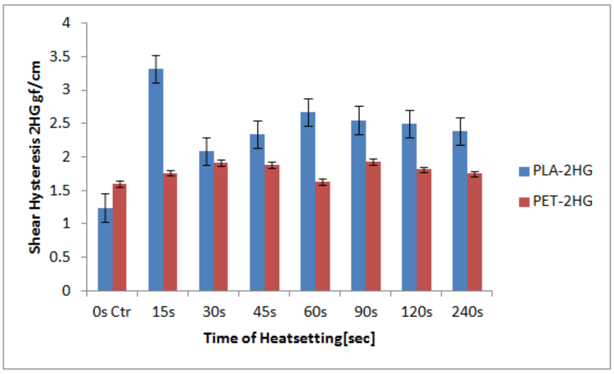

Figure 11: Comparative evaluation of the effect of increasing time of heat setting and wet treatments on Shear Hysteresis 2HG of treated and untreated PLA and PET.

Figures 9-11 comparatively evaluates the effect of increasing time of heatsetting and wet treatments on $2HG_1$, $2HG_2$ and 2HG respectively. Results indicate that untreated PET exhibited higher values for the parameters. But on heatsetting and wet treatments PLA increased across all parameters over PET. Hence it can be deduced that increasing time of heat setting and various applications of wet treatments increased the 2HG properties of PLA and relatively reduced those for PET. These values are used in calculating hand values for the fabrics. NatureWorks manufacturers of Ingeo PLA recommends a heatsetting time of 30 s at 110°C for PLA.

Conclusion

PET from results obtained from this study exhibited a higher Shear Rigidity than PLA. This may be attributed to the construction of the fabric structure. Fabrics that are loose exhibit lower G due to smaller number of yarns cross over points. Tightly closed structures exhibit a higher G. Shear hysteresis at 0.5° Shear angle (2HG) measured energy losses during shear deformation as a result of yarn to yarn friction at cross over points. Large hysteresis implies greater recovery forces will be required to overcome fabric internal friction. PLA exhibited a higher shear hysteresis than PET with increasing time of heatsetting and application of finishing treatments. Before application of treatments to both fabrics, PET exhibited a higher 2HG than PLA. This is due to the effect of heatsetting and wet processes which greatly reduced inter yarn friction in the fabrics and reduced number of fiber to fiber contacts at cross over points. Similar to bending properties, heatsetting after wet processes increases shearing properties which makes the fabrics more extensible with better elastic recovery.

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