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Comparative Evaluation of the Effects of Time of Heat Setting and Wet Processing on Shearing Properties of Knitted Ingeo™ Poly (Lactic Acid) (PLA) and Polyethyleneterepthalate Fabric

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Abstract This paper comparatively studied the effects of time of heatsetting and wet processes on 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 knitted Ingeo™ Poly (lactic) acid and Polyethyleneterepthalate fabrics in warp [G_1] and weft [G_2] directions using the KES-FS system of fabric evaluation. PLA samples of dimension 200 mm x 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 and subsequently treated to wet processes including scouring, alkaline reduction clearing, dyeing and softening. The KES Shear tester was used in determining the ease with which knitted PLA and PET fibers slided against each other to ascertain properties such as softness, pliability to stiffness/rigidity and inter yarn stability to mechanical distortion. They samples were measured to a maximum shear angle of ± 8°. Results showed that after subjecting the samples to various wet processes, PET exhibited higher shear rigidity G in both warp (G_1) and weft (G_2) directions with increasing times of heatsetting. Measurements of shear hysteresis at 0.5° shear angle (2HG) and 5° angle (2HG5) in gf/cm showed PLA exhibiting a higher shear hysteresis than PET with increasing times of heatsetting.

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

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1. Introduction

Ingeo is Cargill Dow's brand name for the first man-made fiber derived from 100% annually renewable resources. The process starts with corn, an abundant raw material that can easily and efficiently converted into plane sugars which subsequently undergo fermentation. The fermentation products are immediately transformed into high performance polymer called polylactide from which the branded Ingeo fibers and filaments are extruded [1,2,3,4,5].

Polylactic acid is aliphatic polyester which is considered as a green material due to its natural based origin and biodegradable properties. Lactic acid obtained from the fermentation of sugar obtained from corn or cassava is used as a monomer for PLA polymerization. Production of PLA is achieved by two major routes through direct condensation polymerization reaction of lactic acid and ring opening polymerization of lactide, a cyclic dimer of lactic acid, yielding poly(lactic acid, poly(d-lactic acid) or poly(d, l- lactic) acid depending on lactic acid isomers used. Polylactic acid can be produced by both melt spinning and solution spinning [6,7,8,9] though the former is used more regularly due to more eco friendliness and ease of processing. Thermal degradation of Polylactic acid can be prevented by the addition of a thermal stabilizer. The processing of Polylactic acid fiber/yarn is an important parameter in controlling the properties of the Polylactic acid fiber, yarn or fabric. Polylactic acid yarns passed through various yarn processing conditions possesses different physical properties and morphological characteristics, which invariably influences the ability of chemicals to penetrate the fabric during wet processing like scouring, dyeing and finishing [10,11,12,13,14]. The chemical structure of Polylactic acid is shown in Figure 1 below;

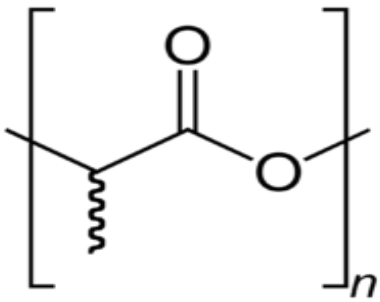

Figure 1. Chemical structure of Polylactic acid

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 [15,16,17,18]. The fundamental procedure for the production of Poly (ethylene terepthalate) was established by Carothers of Du Pont in 1928. This was followed suit by the development of the first commercial fiber-forming polyester in 1941 by Winfield and Dickson. Subsequently Imperial Chemical Industries Ltd marketed the first polyester filament in 1948. Presently, polyester is the most versatile synthetic fiber due to its very good textile properties and its affinity to blend very well with cotton [19,20]. The repeating unit of polyester molecule contains ester linkages in the main chains of macromolecules. PET is aromatic polyester, including a benzene ring in each repeat unit. 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 [22]. PET exhibits very good mechanical properties. The melting temperature of PET lies between 254°C and 260°C [15]. PET exhibits a high light resistance, UV and high abrasion resistance [23,24,25]. 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 [25-30]. 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. 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 to 15 minutes. Ingeo PLA fibers are dyed using disperse dyes though not all disperse dyes are good for dyeing Ingeo fibers [31]. The optimum dyeing conditions for PLA yarns as recommended by DyStar is 110°C for 30 minutes at pH between 4.5 and 5.0. Polyester is very difficult to dye due to its Crystallinity hence absorbs little water and does not swell [32,33,34,35]. Softening is a process which enhances the softening or handle of the fabric through the use of appropriate chemicals called softeners. The feel or handle of a textile fabric is a function of the mechanical and physical properties of its constituent yarns [36,37,38,39,40]. Heatsetting the fiber introduces enhanced dimensional stability to the fibers improving fiber morphology and orientation. The heatsetting process is determined by the temperature, time of heatsetting, the medium of heatsetting (air, solvents or water) and the tension applied to the substrates during heatsetting [41,42]. The Kawabata Evaluation System is an industrial standard of determination of fabric handle through an objective mode of assessment. KES measures a series of fabric properties at low stresses comparable to those the fabric undergoes during normal handling, tailoring, wearing and other end user applications [43]. The shearing properties as determined by the KES-FB system of fabric evaluation are shown below:

Table 1. KES-FB Shear properties and definition

SHEAR PROPERTIES	DEFINITION
Shear rigidity (G)	Average slope of linear regions of the shear hysteresis loop at ± 0.5° Shear angle
Shear Hysteresis (2HG)	Average width of the shear hysteresis loop at ± 0.5° shear angle

2. 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 150/144 d 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 heatsetting 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 (Table 2) and the chemical structure is shown below (Figure 2).

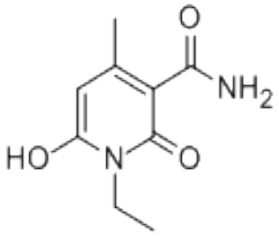

Figure 2. Chemical structure of Dianix Yellow C-5G 200%

Table 2. Characteristics of selected disperse dye

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

2.1. Experimental

2.1.1. 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.

2.1.2. 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 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.

2.1.3. 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 3 below shows the individual values as determined from calculations;

Table 3. PLA dye values

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

The Mathis LABOMAT Infra-red Uniprogrammer calibrations for the knitted PLA fabric initially read as follows:

Table 4. Mathis LABOMAT Uniprogrammer Calibrations for PLA

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

The Dyeing procedure for PLA as represented by Mathis LABOMAT Infra-red equipment is shown in Figure 3 below.

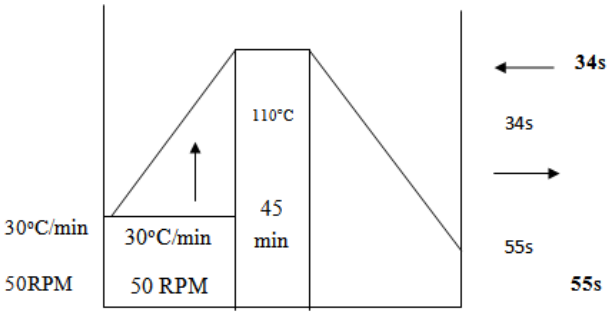

Figure 3. Mathis LABOMAT Uniprogrammer PLA dyeing procedure

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 45 minutes 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 table below:

Table 5. PET dye values

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 6. Mathis LOBOMAT Uniprogrammer for PET

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

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 below:

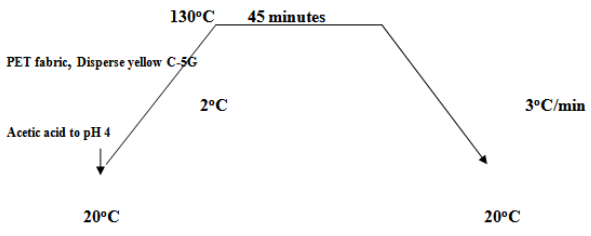

Figure 4. Procedure of disperse dye applied to PET

2.1.4. 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 7 below:

Table 7. Alkaline reduction Clearing Parameters

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

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 [33,34,35,36] as shown in the equation below:

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

Figure 5. Dye decolorization during alkaline reduction clearing process

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 mls 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.

2.1.5. 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 0.416 mm/s.

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 2HG5 – Shear hysteresis at 0.5° angle (2HG5), 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 2HG5 indicates good comfort and softness. Too large values of 2HG5 indicates inelasticity and stiffness.

Fabric finishes generally reduces G and 2HG5 values due to internal lateral pressure reduction and stress relaxation. Smaller contact area at yarn cross over points also reduces G and 2HG5.

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.

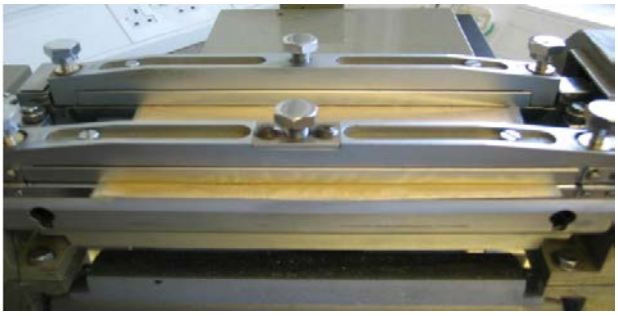

Figure 6. KES- FB Shear Tester [MODEL KES-FB1]

3. Results and Discussion

3.1. Shear Rigidity gf.cm/degree

The results of Shear Rigidity G in warp G_1 and weft G_2 directions are shown below:

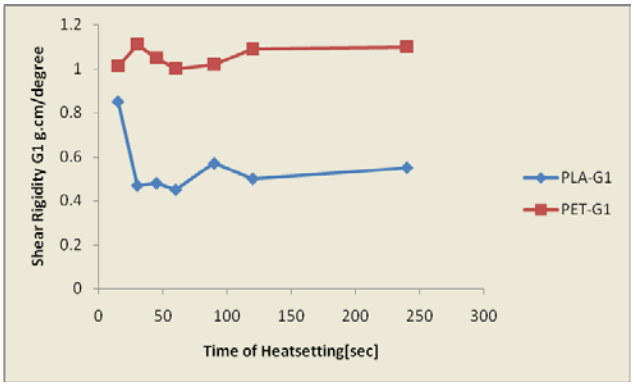

Figure 7. Effects of increasing time of Heatsetting and wet processing on Shear Rigidity G_1, gf.cm/degree of knitted PLA and PET

Figure 7 above shows PET exhibited higher shear rigidity over PLA due to wet processing and increasing times of heat setting. PLA decreased drastically from 0.5

g.cm/degree in warp direction to 0.4 g.cm/degree from 0-70 s while PET showed an increase within the same period. Also in weft (G_2) as shown in Figure 8, PET exhibited a higher Shear rigidity than PLA with increasing time of heat setting and wet processing. Theoretically G_1 and G_2 are same. A mean plot of the values shows PET exhibiting higher shear rigidity than PLA. This implies that PET is more rigid and stiffer than PLA while PLA is softer and more pliable than PET.

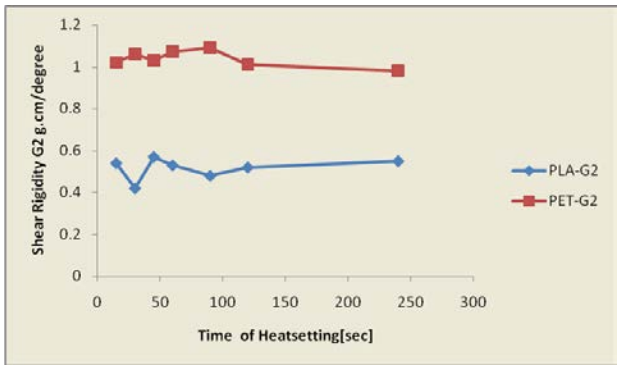

Figure 8. Effects of increasing time of Heatsetting and wet processing on Shear Rigidity G_2 gf.cm/degree of knitted PLA and PET

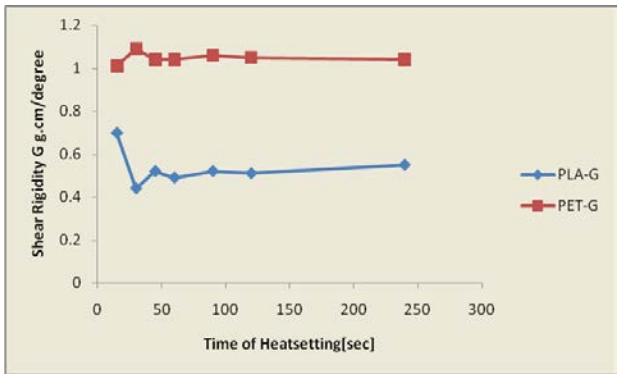

Figure 9. Mean Effects of increasing time of Heatsetting on mean shear rigidity G of Knitted PLA and PET

3.2. 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

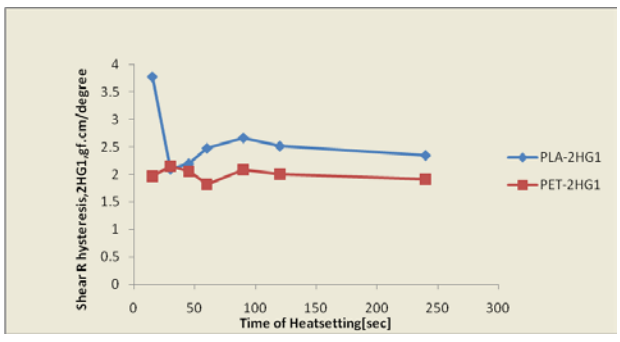

Figure 10. Effects of time of heatsetting and wet treatments on Shear Hysteresis $2HG_1$ of Knitted PLA and PET

Figure 10, Figure 11 and Figure 12 expresses the effect of increasing time of heat setting and wet processing on $2HG_1$, $2HG_2$ and 2HG respectively. Results indicate that knitted PLA has a higher Shear Hysteresis than knitted PET. These are mostly applied in hand value calculations.

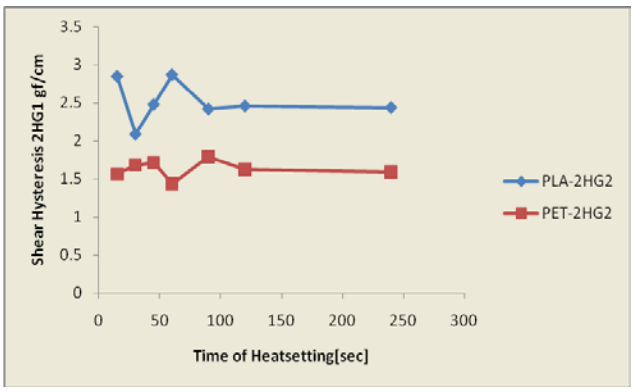

Figure 11. Effect of increasing time of heatsetting and wet treatments on Shear Hysteresis $2HG_2$ on knitted PLA and PET

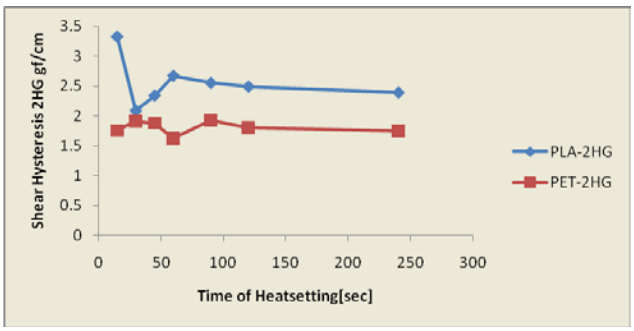

Figure 12. Effect of time of heatsetting and wet treatment on mean of Shear Hysteresis [2HG] of knitted PLA and PET

4. 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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