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Effects of Time of Heat Setting and Wet Processes on Tensile properties of Griege Knitted Ingeo™ Poly Lactic Acid (PLA) Fabric

Idumah CI[1]*, Nwachukwu AN[2] and Akubue B[3]

[1]School of Materials, University of Manchester, Manchester, United Kingdom
[2]Williamson Research Centre for Molecular Environmental Sciences, School of Earth, Atmospheric and Environmental Sciences, University of Manchester, Manchester, United Kingdom
[3]Ebonyi State University, Abakaliki, Nigeria

Abstract

Poly (lactic acid) (PLA) is an aliphatic polyester and ecofriendly material of a natural origin with biodegradable properties. The monomer for PLA is lactic acid obtained from the fermentation of sugar and vegetables like corn and cassava. This study investigated the effect of heatsetting and wet processes on the tensile properties of treated and untreated Ingeo™ Poly (Lactic acid) PLA knitted fabrics. PLA samples of dimension 200 mm×200 mm were subjected to heatsetting at a temperature of 130°C and increasing times of 15s, 30s, 45s, 60s, 90s, 120s and 240s respectively using the Werner Mathis infra red heatsetting equipment and subsequently treated to wet processes including scouring, alkaline reduction clearing, dyeing and softening. Four tensile parameters were determined in warp and weft direction using the KES-FB system of fabric evaluation. These tensile parameters were tensile extension EM [%], linearity of load extension LT [-], tensile energy WT [g.cm/cm²] and tensile resilience RT [%]. Results indicated a remarkable change in tensile properties of PLA with increasing times of heat setting and wet processes.

Keywords: Ingeo™; Poly lactic acid; KES-FB system; Linearity of load extension; Tensile energy; Tensile resilience

Introduction

Ingeo Polylactic acid is the only synthetic fiber available in large commercial quantities and wholly produced from an annually renewable raw material source that is not oil [1]. The fundamental raw material for the production of Ingeo PLA is corn [1,2]. 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 [3]. The fermentation products are immediately transformed into high performance polymer called polylactide from which the branded Ingeo fibers and filaments are extruded [4]. Polylactic acid is aliphatic polyester which is considered as a green material due to its natural based origin and biodegradable properties [5]. Lactic acid obtained from the fermentation of sugar obtained from cassava or corn is used as a monomer for PLA polymerization [5,6]. Production of PLA is achieved through two major routes through direct condensation polymerization reaction of lactic acid and ring opening polymerization reaction 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 isomers used [6,7]. PLA can be melt spun into different types of fibers including monofilaments, multifilaments, bulked continuous filaments, staple fibers, short–cut fibers and spunbond fabrics by conventional melt spinning machines [6-8]. The fibers are then drawn and annealed to give desirable mechanical properties such as high tenacity, good toughness and good dimensional stability [9].

Ingeo PLA fibers are dyed using disperse dyes though not all disperse dyes are good for dyeing Ingeo fibers. Research to ascertain appropriate disperse dyes for dyeing Ingeo has been initiated by DyStar Co [10-13]. Though PLA fibers exhibit characteristics similar to synthetic fibers, they are based on renewable natural sources and hence require re-engineered dyeing and finishing processes to obtain maximum benefits. DyStar recommended some group of disperse for use in dyeing Ingeo PLA fibers. Three azoic dyes comprising of medium energy and heat fastness of trichromatic combination, two anthraquinone dyes and three benzodifuranone were recommended by DyStar [10,11]. An understanding of the dyeability of PLA knitted fabric is imperative in producing aesthetically appealing fabrics of commercial value and enduring durability to washing and fastness. The dyeing properties of disperse dyes on PLA has been studied by many researchers in order to further the understanding of parameters that determine the dyeability of PLA [10,14-15].

Wet processes applied to PLA in this investigation are scouring, dyeing, alkaline reduction clearing, and softening processes. The essence of scouring is to remove impurities and surface contaminants. The scouring process is thought to have an impact on the fiber structure, properties and overall dyeing performance [16-18]. Generally, scouring is carried out using hot alkali which is a solution of caustic soda and detergent and usually occurring at significantly lower temperatures than those of heatsetting. Alkaline reduction clearing is a wet process of using caustic soda and sodium hydrosulphite ($Na_2S_2O_4$) to effectively remove unfixed dye at the surface of PLA fibers at 70°C and time duration of 10 to 15 minutes. This is because PLA fabrics may be contaminated with surface deposits of unfixed dyes after the dyeing process, especially at heavy depths of shade since there is tendencies for water insoluble disperse dyestuff to aggregate into relatively large particles as the dye bath cools down to below 100°C [19-21]. The softening process enhances the softening or handle of the fabric through the application

***Corresponding author:** Idumah CI, School of Materials, University of Manchester, Manchester, United Kingdom, E-mail: idugoldengate@yahoo.com

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of appropriate softening agents. The application of softeners produces a level of softness which may not be attained by mechanical finishing or modification of fabric construction [22].

PLA is a renewable synthetic fiber manufactured by extrusion through spinnerets to form filaments which in turn impact stress which are generated within the polymer structure and trapped in the material on cooling. Dimensional stability is in turn impacted into the fiber when exposed to wet or heat treatments. Heatsetting of fibers introduces enhanced dimensional stability to the fiber thereby improving fiber morphology and orientation. Heatsetting 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 [22-23].

The Kawabata Evaluation System is an industrial standard of determination of fabric handle through an objective mode of assessment. KES was used in this research to measure a series of fabric properties at low stresses comparable to those the fabric undergo during normal handling, tailoring, wearing and other end user application. Tensile properties evaluated using KES for this research are as shown in Figure 1.

Materials and Methods

The Ingeo Poly (lactic acid) fabric used for this investigation was supplied by NatureWorks LLC, USA. Sixteen samples of pique knitted fabrics obtained from 150/144d Tex/ filament PLA 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 15s, 30s, 45s, 60s, 90s, 120s, and 240s respectively. The untreated 'pique' knitted fabric was used as control.

Dye

The dye used for this work is 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 formula weight is 196.2 and the chemical structure is shown Figure 2 and Table 1.

Experimental

Heat-setting procedure

The heat-setting of knitted PLA fabrics were achieved using the

PROPERTY	DEFINITION
TENSILE	
Extensibility (EM)	% Extension at maximum applied load of 500g/cm
Tensile energy (WT)	Energy in extending fabric to 500g/cm
Linearity of load-extension curve (LT)	Linearity of load-extension curve
Tensile resilience (RT)	% Energy recovery from tensile deformation

Figure 1: PLA tensile properties evaluated using Kawabata Evaluation System.

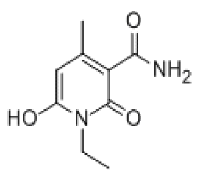

Figure 2: Chemical structure of Dianix Yellow C-5G 200%.

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 3: The Werner Mathis AG heatsetting equipment.

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 as recommended by Cargill Dow. The samples were pinned on the sliding aluminum frame pins and heat set for time durations of 15s, 30s, 45s, 60s, 90s, 120s and 240s respectively. The essence of prolonged heatsetting of PLA 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 (Figure 3).

Scouring procedure

They heat-setted PLA samples of dimension 200 mm by 200 mm and total weight of 83 g were scoured in 450 ml 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.

Dyeing of knitted PLA 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. Individual values as determined from the calculations (Table 2).

The Mathis LABOMAT Infra-red Uniprogrammer calibrations for the knitted PLA fabric as given in (Table 3).

The Dyeing procedure for PLA as represented by Mathis LABOMAT Infra-red equipment is shown in Figures 4 and 5.

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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.

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.

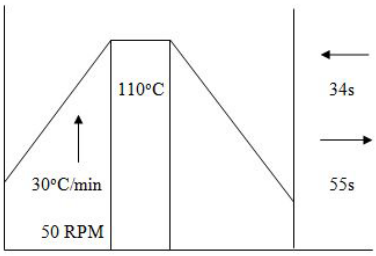

Figure 4: Mathis LABOMAT Uniprogrammer PLA dyeing procedure.

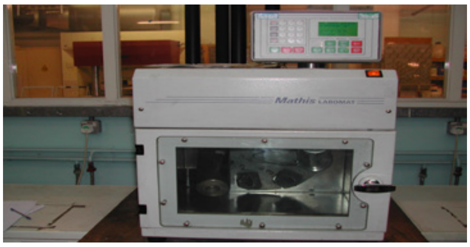

Figure 5: Mathis LABOMAT PLA dyeing equipment.

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 4: Alkaline reduction Clearing Parameters.

Alkaline reduction clearing procedure

Alkaline reduction clearing is a process which occurs after dyeing and air drying in order to extricate surface disperse dye. 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 4.

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 [24-28]. 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 [29-31] as shown in Figure 6.

They softening agents 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.

The Kawabata Evaluation System

The KES-FB system determines fabric properties at small loads

$$R_1 - N = N - R_2 \longrightarrow R_1 - NH_2 + NH_2 - R_2$$

Figure 6: Dye decolorization during alkaline reduction clearing process

Figure 7: Werner Mathis AG padding (softening) equipment.

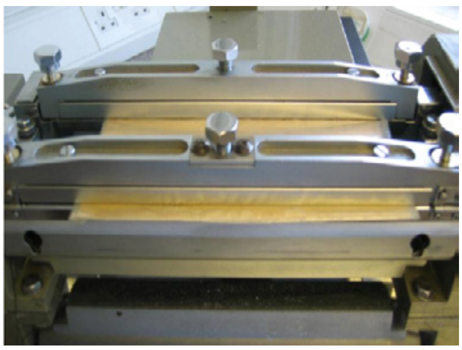

Figure 8: KES FB 1 Tensile equipment.

equivalent to those the fabrics are subjected to at normal end use application. The tensile properties determined were fabric extension [%], linearity of load extension, tensile energy [WT] g.cm/cm² and tensile resilience [%]. The specimen were clamped between two chucks each of 20 cm long. A constant force of 200 g was applied by attachinga weight to the front chuck of the specimen. When the test started, the back chuck constantly slided initially right to an angle of 8° then back to it's original position (Figure 8).

Results and Discussion

Figure 9 expatiates the effect of increasing time of heatsetting and various wet or finishing treatments on the extension of knitted PLA in comparison to the knitted loom fabrics or control. There is a remarkable change on the tensile extension [EMT] of PLA fabrics with increasing time of heatsetting and wet treatments especially at 90's. This implies that increasing heatsetting time of PLA beyond 90's may not necessarily improve tensile extension of knitted PLA. This is because finishing processes generally tends to alter fabric properties. PLA exhibited a consistent increase in tensile extension with increasing time of heatsetting and wet finishing applications. This implies that PLA may tend to exhibit enhanced fabric hand or softness and increased formability with increasing time of heatsetting and finishing applications.

Figure 10 below shows no remarkable change in linearity of load extension with heatsetting and wet treatments though a slight decrease in this parameter was noticed. This result indicated a softer

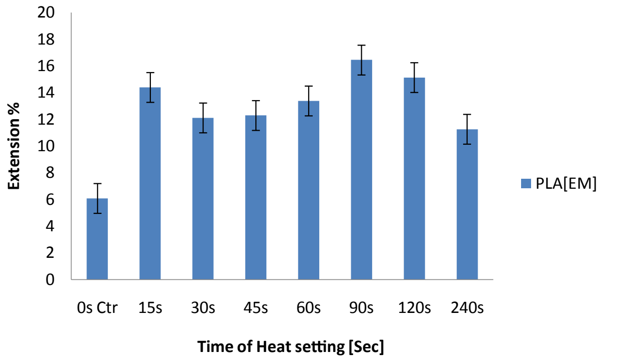

Figure 9: Comparative effect of heat-setting and wet processing on tensile extension (EMT) % of untreated control (0s Ctr) PLA and treated PLA fabrics.

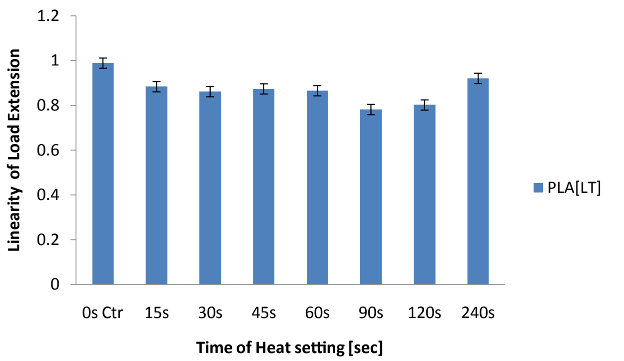

Figure 10: Comparative effect of increasing time of heat setting and wet processes on linearity of load extension LT of untreated (0s Ctr) control PLA and treated PLA fabrics.

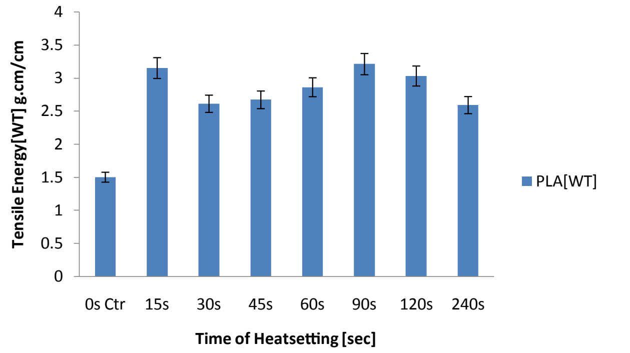

Figure 11: Comparative effects of heat setting and wet processes on Tensile Energy WT (g.cm/cm²) of untreated control and treated PLA fabric.

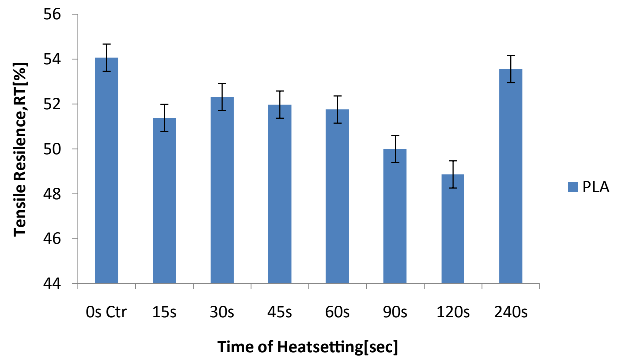

Figure 12: Comparative effect of heatsetting time and wet treatments on the tensile resilience of untreated control (0s Ctr) and treated PLA samples.

hand (larger extension in the initial low load region of load extension curve. Subjectively PLA fabric stretch is linked closely to LT (initial resistance to tension). PLA exhibition of lower LT with increasing time of heatsetting and wet treatments implies a better formability as extensibility under small loads represents in-pane compressibility which is equivalent to fabric formability (Figure 11).

This results show an increasing tensile energy of treated PLA fabric with increasing time of heatsetting and various application of wet treatments. Tensile energy (WT) g.cm/cm² is the energy required to extend a fabric to the prefixed maximum load and closely related to fabric flexibility, softness, gentleness and smoothness. At heatsetting time of 90s, the highest level of increase in tensile energy of PLA was noticed. Beyond this timing no better value was added to the tensile energy of PLA.

The result shows no remarkable difference in tensile resilience between the treated and untreated PLA fabrics. Tensile resilience, RT % is a measure of the ability of a fabric to recover after extension when applied force is removed. A lower RT as exhibited by PLA promotes softness (Figure 12).

Conclusion

The current research was initiated to comparatively analyze the effect of time of heatsetting and wet processes on the tensile properties of treated and untreated PLA samples to enhance further understanding of the tensile properties of Poly (lactic acid) fabric using the Kawabata Evaluation System (KES-FB) for fabrics.

The optimum time of heatsetting PLA yarns to minimize shrinkage during subsequent wet processing is within the range of 30-40s at 130°C.

There is a remarkable change in tensile extension (EMT) of PLA with increasing time of heatsetting and wet processes. This implies that PLA exhibited enhanced fabric hand or softness and increased formability after heat treatments and wet processes.

Treated PLA decreased in linearity of load extension LT with increasing time of heat setting and wet processes than the untreated PLA. A smaller LT as exhibited generally by PLA indicates a smaller hand or a larger extension in the initial low load region of the load extension curve. Subjectively, PLA fabric stretch is closely linked to LT (initial resistance to tension) and RT (resilience). PLA exhibition of lower LT with increasing time of heatsetting and wet treatments implies a better formability as extensibility under small load represents in-plane compressibility which is equivalent to fabric formability. PLA tailorability improves with improved fabric formability.

An increasing tensile energy is noticed with increasing time of heat setting and wet treatments on treated PLA when compared to untreated PLA. Tensile energy (WT) $g.cm/cm^2$ is the energy required to extend a fabric to the prefixed maximum load and closely related to fabric flexibility, softness, gentleness and smoothness.

Treated PLA exhibited no much difference in tensile resilience to untreated PLA. Tensile resilience, RT % is a measure of the ability of a fabric to recover after extension when the applied force is removed. A lower RT promotes softness and RT increases in fabric finishing as the inter fiber force are reduced.

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