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Study on flame retardant properties of poly(lactic acid) fibre fabrics

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Polylactic acid and its blended yarns with various fibres have been converted in to woven fabrics. These fabric samples have been scoured and then tested for mechanical and flame retardant properties. The results indicate that the high alkaline scouring severely damages polylactic acid fibre. Therefore, mild alkaline scouring is done for further studies. The scoured fabric samples are tested for various flame retardant properties and compared with polyester blended fabrics. Results indicate that polylactic acid fibre is not suitable for upholstery, apparel and work wear in terms of flame retardant properties.

Keywords: Cigarette ignition, Edge ignition, Horizontal flammability, Inclined flammability, Match flame, Oxygen index

1 Introduction

Poly (lactic acid) or PLA is aliphatic polyester being considered as a green material due to its natural-based origin and biodegradable properties. Lactic acid obtained from the fermentation of sugar and vegetables e.g. corn and cassava is used as a monomer for PLA polymerization[1]. The poly (lactic acid) fibre may be produced by various other methods[2].

It was reported[3-6] that PLA fibre has low moisture absorption, low flammability, and high resistance to UV light with other excellent mechanical properties. The various applications of PLA in composite have already been discussed[7]. The thermal characteristics of PLA filaments have been investigated[8] using modulated differential scanning calorimetry (MDSC). Laboratory UV resistance testing using a Xenon Arc[9] indicates that in comparison with polyester and acrylic fibres, PLA fabrics have superior strength retention than polyester and far superior resistance to discoloration than acrylics. The application of PLA fibre in automotive interior has also been discussed earlier and its properties are compared with polyester[10].

The comparison of flammability properties of PLA and polyethylene terephthalate (PET) are summarized[11,12]. It is reported that the fibre has low flammability (Oxygen Index also known as limiting oxygen index: 26+) and less smoke generation property than PET (oxygen index 21).

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In India, PET is a most popular fibre used in upholstery, apparel and work wear. It is either used as such or blended with various fibres especially cotton to manufacture these products. As PLA fibre has better strength retention[9] to UV light exposure and low flammability than PET, it was thought to test this fibre and its blends for flame retardant (FR) properties for the use in textiles such as upholstery, apparel and work wear. In the present study, various blends of the PLA fibre were developed and their FR performance was evaluated on the basis of end use.

2 Materials and Methods

PLA, Nylon 66, PET and Shankar 6 variety of cotton fibres were used for the study. These fibres properties are given in Table 1. Also, 20^s count yarn of PLA fibres and their blends (Table 2) were prepared

Table 1 —Fibre property

Parameter	PLA	Nylon 66	PET	Cotton
Length, mm (ASTM D 5867)	38	35.92	38.7	29.5
Specific gravity g/ cm^3	1.25	1.14	1.38	1.54
Denier (ASTM D-1577)	1.53	1.74	1.03	1.64
Tenacity, gpd (ASTM D -3822)	3.54	6.61	6.29	3.33
Elongation- at-break % (ASTM D -3822)	39.01	7.84	17.04	7.84
Moisture content, % (IS 199)	0.4	4.5	0.4	7.8
Melting point, °C	169	265	260	-
Tg, °C	64.9	50	69	-

Table 2—Blend percentage

Fibre	Code	Blend ratio, %
PLA	Pl	100
PLA: Cotton	(Pl:C)82	80:20
PLA: Cotton	(Pl: C)73	70:30
PLA: Cotton	(Pl: C)55	50:50
PLA: Nylon 66	(Pl:N)55	50: 50
PLA: Nylon 66: Cotton	(Pl:N:C)352	30:50:20
PLA: Nylon 66: Cotton	(Pl:N:C)154	10:50:40
PET: cotton	(PE:C)55	50:50
PET: cotton	(PE:C)82	80:20

Table 3—Weave particulars

Test parameter	Value	
	P-1*	R-2**
Ends per inch	76	92
Picks per inch	68	52
Warp count	20^s	20^s
Weft count	20^s	20^s
Weave	Plain (1 up 1 down)	Rip stop (Plain)

*P-1- Pl, (Pl:C)82, (Pl:C)73 and (PE:C)82.
**R-2- (Pl:C)55, (Pl:N)55, (Pl:N:C)352, (Pl:N:C)154 and (PE:C)55.

Using ring spinning system. Similar count of polyester and cotton blend was also developed for comparison purpose.

The developed yarns were converted into woven fabrics using rapier loom as per the requirement of end use. The two types of fabric developed are designated as P-1(Plain weave) and R-2 (Rip stop weave). The weave particulars and other mechanical properties are given in Table 3. Water soluble PVA was used as sizing agent.

These fabric samples were desized in hot water for the duration of 30 min followed by scouring. Finally, the fabric samples were evaluated using standard test methods for various properties including flame retardant properties as per the end use application.

3 Results and Discussion

3.1 Effect of Scouring

To remove natural and added impurities from the PLA and cotton blended fabric, it is required to scour this fabric before further processing. To observe the effect of scouring on PLA component, 100% PLA fabric is scoured in alkaline media. It is observed that after alkaline scouring (2.5% NaOH owf, 2% Na_2CO_3 owf, temperature 95°C, and duration 45 min), excessive weight loss (33%) occurs. Therefore, the cotton blended fabrics of PLA fibre are scoured using mild alkaline conditions with 2% Na_2CO_3 at 80^0C for 30 min before determining the various properties. With this mild scouring the weight loss is found only 1%. These fabric samples were not bleached as it has established[13] that bleaching of PLA fabric in hydrogen peroxide and sodium hydroxide solution, damages fabric severely.

Table 4—Mechanical properties (P1)

Test parameter	Pl	(Pl:C)82	(Pl:C)73	(PE:C)82
Ends/inch (IS 1963)	74	76	78	78
Picks/inch (IS 1963)	68	66	68	66
Tensile strength N (IS 1969)				
Warp	688.10	651.75	646.05	652.24
Weft	608.12	589.10	544.14	578.56
Tear strength, N (IS 6489)				
Warp	24.06	23.18	23.01	25.02
Weft	19.94	19.26	18.87	20.12
Weight, g/m^2 (IS 1964)	176	182	184	180

Table 5—Mechanical properties (R-2)

Test parameter	(Pl: C) 55	(Pl: N) 55	(Pl:N:C) 352	(Pl:N:C) 154	(PE:C) 55
Ends/inch (IS 1963)	92	94	92	94	94
Picks/inch (IS 1963)	54	54	54	52	52
Tensile strength, N (IS 1969)					
Warp	890	1120	1104	1050	901
Weft	580	683	670	630	612
Tear strength, N (IS 6489)					
Warp	29.12	35.86	30.97	29.98	30.20
Weft	39.02	45.08	42.28	40.06	40.23
Weight, g/m^2 (IS 1964)	202	204	206	210	208

3.2 Mechanical Property

PLA blended fabric samples (after scouring) were tested for mechanical properties. Tables 4 and 5 show the test results. From Table 4 (P1- Plain weave), it is clear that the tensile and tear strength of fabric decrease with the increase in percentage of cotton in the blend. This decrease in tensile and tear strength of the fabric samples is due to the lower tenacity of the cotton than PLA as well the

effect of scouring treatment on PLA fibre. It is also clear from this table that the tensile strength and tear strength properties of (PE:C)82 are higher than that of (Pl:C)82, because tenacity of PET fibre is higher than PLA. Table 5 shows the property of R-2 (Rip stop weave) fabric. It is clear from this table that with the increase in nylon 66 percentage in the blend, tensile and tear strength increase. The increase in tensile and tear strength is due to higher tenacity of nylon 66 than cotton and PLA fibres.

3.3 Flame Retardant Behavior

3.3.1 Oxygen Index

Oxygen index (OI) also known as limiting oxygen index (LOI) is the preliminary test to ascertain that fibre is flame retardant or not. The oxygen index (OI) is the minimum concentration of oxygen by percentage volume in a mixture of oxygen and nitrogen that will support combustion of a material under specified test conditions. Following equation is used to determine oxygen index value of a material.

$$OI = \frac{[O_2]\times 100}{[O_2]+[N_2]}$$

where $[O_2]$ is the volumetric flow of oxygen in cm^3/s; and $[N_2]$ the corresponding volumetric flow rate of nitrogen in cm^3/s.

All the fabric samples have been tested for OI value. The test results are given in the Table 6. The OI of the fabric made out of 100% PLA fibre is found to be 23. The OI values of PLA blended fabrics are found to be lower than the OI of the fabrics made out of 100% PLA fibre (Table 6). This drop in OI of the PLA blended fabric is expected, as the fibres used in the blends are cotton and nylon, having OI values[12] 18 and 21 respectively, which is lower than poly (lactic acid) fibre. As it is a well - known fact that if the combustible fibre like cotton (LOI around 17) is blended with inherently flame retardant Nomex fibre (LOI around 28), the average oxygen index of the blend will be lower than the inherently flame retardant fibre[14].

3.3.2 FR Tests for Upholstery Textiles

It has been observed that most of the fire accidents occur in public places due to the combustion behavior of upholstery textile material. Keeping in mind the higher OI value (23) of PLA fabric, it was thought to study its application in the manufacture of upholstery material such as seat covers, bed sheets and curtain cloth. The fabric manufactured using PLA and its blends were tested for various FR properties using standard test methods and specifications as given below:

Seat Covers

Test methods—For automotive seat cover SES N 3295 (horizontal flammability).

Criteria of passing—The maximum burn rate of the fabrics used for automotive seat covers should be 80 mm/min as per SES N 3295 standard.

Bed Sheets

Test methods—Ignition source used is Cigarette (BS 5852 Part I, Source 0) and match flame (BS 5852 Part I, Source 1).

Table 6—Fire retardant properties of PLA blended fabric

Test parameter	Sample particular								
	Pl	(Pl:C)82	(Pl:C)73	(Pl: C)55	(Pl: N)55	(Pl:N:C) 352	(Pl:N:C)154	(PE:C)82	(PE:C)55
Oxygen index, % (OI) (IS 13501)	23	20	19	19	21	20	19	19	18
Horizontal Flammability Test (SES N 3295)									
Burn rate, mm/min	346	113	120	130	110	90	79	120	140
Cigarette ignition (BS 5852-Pt 1 - Source 0)	Non ignition	Non ignition	Non ignition	Non ignition	Non ignition	Non ignition	Non ignition	Non ignition	Non ignition
Match flame (BS 5852-Pt 1 - Source 1)	Ignition	Ignition	Ignition	Ignition	Ignition	Ignition	Ignition	Ignition	Ignition
Vertical Flammability Test (BS5867 Part 2 Type B)									
Hole reached any edge	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	Yes
Flame reached any edge	Yes	Yes	Yes	Yes	Yes	No	No	Yes	Yes
Flaming debris	Yes	No	No	No	No	No	No	No	No

Criteria of passing—In the case of both the ignition sources, there should be no ignition in the sample.

Curtains

Test method — BS 5867 Part 2 Type B

Criteria of passing:

- No hole should reach any edge on any specimen.
- No flame should reach any edge on any specimen.
- No flaming debris should be observed.

The results of the various tests conducted on the fabrics are given in Table 6. It is observed that none of the samples, except (Pl: N: C) 154, meet the passing criterion of SES N 3295 (seat cover for automobile). Table 6 shows that with the decrease in PLA percentage in the blend of Pl:N:C, the burn rate decreases. The burn rates of (Pl:N)55, (Pl:N:C)352 and (Pl:N:C)154 are 110, 90 and 79 mm/min respectively. The higher burn rate of blends having higher percentage of PLA may be due to lower melting point of PLA than Nylon 66 (Table 2), which might be helping propagation of burning.

The fabric samples, tested as per BS 5852 Part I source 0(Cigarette), indicate that there is no ignition behaviour observed. However, in the case of 100% PLA fabrics, melting behaviour is observed, as PLA has lower melting point than nylon and polyester (Table 2). All the samples are failed to meet the passing criteria of BS 5852 Part I Source 1 (Match flame), as ignition is observed in the all samples. It means that all the fabric samples do not meet the passing criteria of flame retardant bed sheet. The reason of not meeting the requirement of passing criteria may be due to the fact that fibres used in the blends are not sufficiently flame retardant in nature.

To test PLA fibre suitability in curtain upholstery fabric, the developed samples were tested as per BS 5867 Part 2 type B. From Table 6, it is clear that all the samples, except (Pl: N: C) 154, failed to meet the passing criteria. As discussed earlier, higher PLA percentage in the blend might have helped in the propagation of burning. Only 10% PLA in the (Pl: N: C) 154 does not help in propagation of burning. Thus this sample meets the passing criteria.

3.3.3 FR Test for Apparel Textiles

Suitability of all the fabric samples for FR apparel textiles is studied using inclined flammability test as per ASTM D 4723 (D 1230).

Criteria of passing–For passing the performance criteria, three classes are given. As it is a plain surface fabric, Class 1 is recommended which is further categorized as (a) average burn time 3.5 s or more, (b) ignited but extinguished, and (c) did not ignite.

Table 7 shows that 100% PLA fabric (Pl), (Pl: N) 55, (Pl: N: C) 352, and (Pl: N: C) 154 does not ignite (Class-1 category - c); while other samples, which are ignited, fall in the Class-1 category- a.

3.3.4 FR Tests for Work Wear

There are two important methods to test limited flame spread of materials for use in garments to protect against heat and flames. These are ISO 15025 method A (Surface ignition) and ISO 15025 method B (Edge ignition).

Criteria of passing - Following are the performance criteria for passing a sample in accordance to the specification ISO 11612:

ISO 15025-Method A

- No specimen shall suffer flaming to the top or either side edge.
- No hole formation.
- No flaming or molten debris.
- The mean value of after flame and after glow time shall be $\leq$2 s.

ISO 15025-Method B

- No specimen shall suffer flaming to the top or either side edge.
- No flaming or molten debris.
- The mean value of after flame and after glow time shall be $\leq$2 s.

It is clear from Table 7 that only sample (Pl: N: C) 154 meets the passing requirement of ISO 11612 for test ISO 15025 Methods A and B. It can be observed from the table that with the increase in PLA fibre the flame retardant property of the samples starts decreasing. The PET and Cotton blended fabric also failed to meet the passing criteria. The reason of decreasing flame retardant property with increase in PLA percentage in blend may be attributed to its low melting point.

Table 7—Fire retardant properties of PLA blended fabric

Test parameter	Sample particular								
	Pl	(Pl:C)82	(Pl:C)73	(Pl: C)55	(Pl: N)55	(Pl:N:C)352	(Pl:N:C)154	(PE:C)82	(PE:C)55
Inclined flammability									
Burn rate, s (ASTM D 4723 - D 1230)	DNI*	11.5	10.34	9.8	DNI	DNI	DNI	12.3	13.2
Surface ignition (ISO 15025-Procedure A)									
Hole formation	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	Yes
Flaming to top or either side	Yes	Yes	Yes	Yes	Yes	No	No	Yes	Yes
After flame, s	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
After glow, s	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Melting or molten debris	Yes	No	No	No	No	No	No	Yes	Yes
Edge ignition (ISO 15025-Procedure B)									
Flaming to top or either side	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	Yes
After flame, s	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Yes	Yes
After glow, s	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Melting or molten debris	Yes	No	No	No	No	No	No	Yes	Yes

*Did not ignite.

4.Conclusion

4.1 Strong alkaline scouring (2.5% NaOH owf, 2% Na_2CO_3 owf, temperature 95°C, and for 45 min) severely damages PLA fibre, therefore it is recommended to use mild scouring conditions.

4.2 The oxygen index (OI) of 100% PLA fabric is found to be 23. With the decrease of PLA proportion in blends, the OI values of the blended fabric decreases. This drop in OI of the PLA blended fabric is in line with the expectations, because the OI values of the fibres used in the blends (cotton and nylon) are 18 and 21 respectively, lower than poly (lactic acid) fibre.

4.3 Only fabric sample (PL: N: C) 154 pass the criteria of horizontal flammability when tested in accordance with SES N 3295 (seat cover for automobile).

4.4 For bed sheet, 5852 Part I source 0(Cigarette) and BS 5852 Part I Source 1 (Match flame) are the important tests to analyze its flame retardant behaviour. All the fabric samples pass the performance criteria of BS 5852 Part I source 0(Cigarette) as there is no ignition behaviour observed in all the samples. However, all the samples failed to meet the passing criteria of BS 5852 Part I Source 1 (Match flame) as ignition behaviour was observed in the all samples. It means that all the fabric samples do not meet the passing criteria of flame retardant bed Sheet.

4.5 The FR property of curtain upholstery fabric is tested as per BS 5867 Part 2 type B. The results indicate that only one fabric (Pl: N: C) 154 meets the passing criteria.

4.6 It is observed that 100% PLA fabric (Pl), (Pl: N) 55, (Pl: N: C) 352, and (Pl: N: C) 154 do not ignite (Class-1 category - c), while other samples which are ignited fall in the Class-1 category- a. These samples meet the passing criteria.

Only sample (Pl: N: C) 154 meets the passing requirement of ISO 11612 for test ISO 15025 Methods A and B. It can be observed that with the increase in PLA fibre the flame retardant property of the samples starts decreasing.

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