An investigation on non-flammability properties of polypropylene knitted fabrics

DOI: 10.35530/IT.074.02.202178

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ABSTRACT – REZUMAT

An investigation on non-flammability properties of polypropylene knitted fabrics

The research performed in recent years regarding synthetic filament yarns is improving the structures and properties of such yarns, and diversifying their usage areas. Most of the current studies being carried out in this field are in the form of bringing functionality to filament yarns. And one of these functional properties is the production of flame-retardant yarns from different types of polymers. In this study, polypropylene (PP) raw material was mixed with organophosphonate based flame retardancy additive at rates in between 1% and 8% in the feed hopper of a laboratory-type melt spinning machine during PP filament yarn production and then knitted fabric structures were obtained from these yarns. Then the flame retardancy properties of these PP knitted fabrics were tried to be determined by vertical flammability tests and Limit Oxygen Index (LOI) tests. Test results indicated that after flame time and char length values had significantly decreased and values of LOI had increased in knitted fabrics by the increase in additive rate. In conclusion, it was observed that the increase in the rate of flame retardancy additive had significantly improved the non-flammability properties of the PP samples.

Key-words: *flame retardancy (FR), knitted fabric, mechanical properties, polypropylene filament yarn, vertical flammability and Limited Oxygen Index (LOI)*

O investigație asupra proprietăților de neinflamabilitate ale tricoturilor din polipropilenă

Cercetările efectuate în ultimii ani cu privire la firele filamentare sintetice îmbunătățesc structurile și proprietățile unor astfel de fire și le diversifică domeniile de utilizare. Cele mai multe dintre studiile actuale care se desfășoară în acest domeniu sunt sub forma aducerii în stadiul de funcționalitate a firelor filamentare. Și una dintre aceste proprietăți funcționale se referă la producerea de fire ignifuge din diferite tipuri de polimeri. În acest studiu, materia primă din polipropilenă (PP) a fost amestecată cu aditiv ignifug pe bază de organofosfonați în proporții cuprinse între 1% și 8% în buncărul de alimentare al unei mașini de filat din topitură de laborator, în timpul producției de fire filamentare PP și apoi au fost obținute structuri tricotate din aceste fire. Apoi, proprietățile de ignifugare ale acestor tricoturi PP au fost testate, prin teste de inflamabilitate verticală și teste cu Indice Limită de Oxigen (LOI). Rezultatele testelor au indicat că, după timpul de ardere și lungimea reziduului de carbon, valorile de ardere au scăzut semnificativ, iar valorile LOI au crescut în tricoturi prin creșterea proporției aditivilor. În concluzie, s-a observat că creșterea proporției aditivului de ignifugare a îmbunătățit semnificativ proprietățile de neinflamabilitate ale probelor PP.

Cuvinte-cheie*: ignifugare (FR), tricot, proprietăți mecanice, fire filamentare de polipropilenă, inflamabilitate verticală și Indice Limită de Oxigen (LOI)*

INTRODUCTION

Polypropylene (PP) based fibres are raw materials that are frequently preferred and used in the textile industry due to their outstanding properties such as low density, easy processability, a sufficient level of tenacity, high chemical resistance and low cost even if they vary. PP fibres, due to their above-listed advantageous properties, are mainly being used in the fields of carpet, upholstery, clothing, technical textile applications etc. In addition to these properties of fibres and of products being obtained from these fibres, bringing in additional properties to these products depending on their place and purpose of use is one of the accentuated issues. For instance, textile products must possess flame retardancy properties, especially in respect of safety. Thus, various studies are being performed in recent years to improve the flame retardancy property of PP fibres in addition to their already available properties. Bringing in flame retardancy property to textile products consisting of PP fibres is generally ensured by the addition of an additive having flame retardancy (FR) properties. Moreover, despite having various additives used for this purpose (phosphorus, boron, intumesant, SiO_2 etc.), the action mechanisms and effectiveness degrees of these additives differ from each other. Today, there are many scientific studies regarding the examination of the action mechanisms and synergistic actions of flame retardancy additives and the production of new types of additives [1–3]. When the results of these studies were reviewed, it was being observed that the flame retardancy property of PP could be improved by substances and that this property was generally being increased to the required

level. Also, it was seen that the textile materials gain a good level of flame retardancy property by the phosphorus-based flame retardancy additives which were based on in this study [1, 4–6]. Examining the permeability properties (air permeability, water vapour permeability, etc.) of knitted fabrics and determining the effects of these properties on their flammability are also important research topics [7–9]. This subject has been evaluated as another study.

This study aims to investigate the non-flammability properties of PP knitted fabrics in detail. Hence, the large control group (1–8% FR additived PP knitted structure) has been used. As a result, the effect of organophosphonate based FR additive on the flammability properties of PP knitted fabrics has been determined. In this study, the production of PP filament yarn by the addition of FR substance at rates of 1%, 2%, 3%, 4%, 5%, 6%, 7% and 8% was performed, and the structures and properties of these yarns were examined. The yarns produced afterwards were converted to the knitted fabric surface, and the effect of additive on flame retardancy properties was tried to be determined through vertical flammability and LOI tests. During production, all other production parameters were kept fixed, and only the rate of additive was determined as a variable factor. The originality of this study can be defined as a detailed examination of the burning properties of PP knitted fabrics which have been obtained by the addition of FR additive at a wide range changing from 1% to 8%.

MATERIAL AND METHODS

Polypropylene polymer chips (Sabic PP 518P) and particles of additive providing flame retardancy properties were used as raw materials in this study. Sabic PP 518P, an isotactic PP (IPP) chips with a melt flow index of 24 g/10 min. and 905 kg/m^3 density was used as a polymer substance. The properties of these PP chips are given in table 1.

Also, an organophosphonate based FR additive (CESA-Flam CFR1) was used in this study and spin-finish oil (Polymast-MKL) was used as an auxiliary chemical during the production of yarns to prevent

Table 1

PROPERTIES OF PP CHIPS USED IN THIS STUDY

Properties	Value	Standards
Resin properties		
Melt flow rate (MFI) (230°C&2.16 kg load density)	24 g/10 min. 905 kg/m^3	ASTM D 1238 ASTM D 792
Mechanical properties		
Tensile strength at yield	32 MPa	ASTM D 638
Tensile elongation at yield	12%	ASTM D 638
Flexural modulus (1% secant)	1550 MPa	ASTM D 790A
Notched izod impact strength at 23°C	30 J/m	ASTM D 256
Rockwell hardness, R-Scale	100	ASTM D 785
Thermal properties		
Vicat softening point	152°C	ASTM D 1525B
Heat deflection temperature at 455 KPa	118°C	ASTM D 648

fibre-metal and fibre-fibre frictions after the spinneret and to prevent adhesion of filaments to each other.

Yarns have been produced according to the melt spinning principle by using a laboratory-type filament yarn machine (figure 1). The PP polymer chips and FR additives were fed into the hopper consisting of a single-screw extrusion system. The heating along the screw was adjusted to obtain a temperature gradient from 220°C to 245°C and a volumetric pump regulated the injection of molten polymer towards the dies at a flow rate of 350 cm^3 per minute. Then, the filaments were air-cooled and a spin finish was applied on the filaments before their passage in the drawing godets. Then filaments were drawn using heated godets before being wound. The speeds of the two godets serve to adjust the drawing ratio: the first godet speed was set at 400 rpm, whereas the second one was set at 800 rpm, giving a drawing ratio of 2. Finally, the filament yarns were wound on a cheese

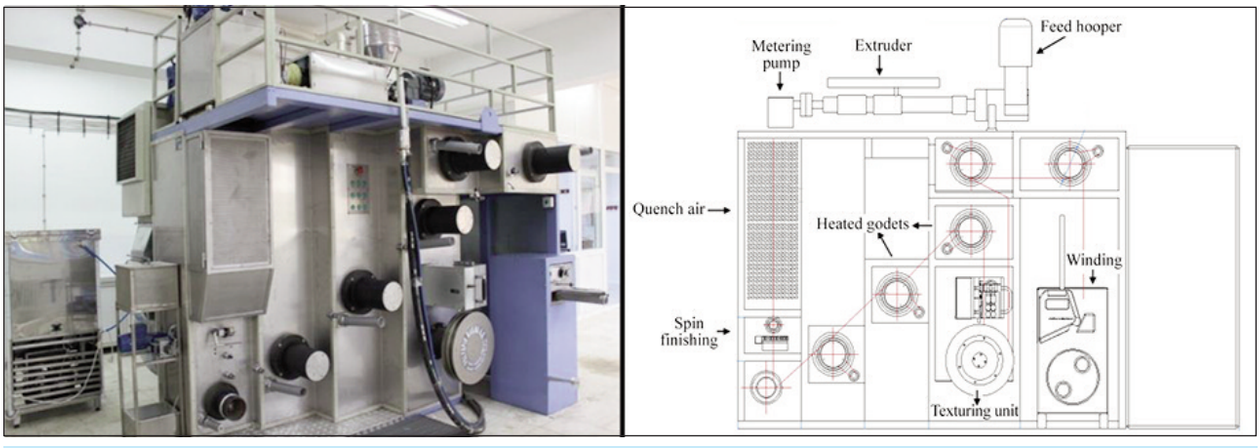


Fig. 1. Laboratory-type melt spinning machine used for yarn production [10]

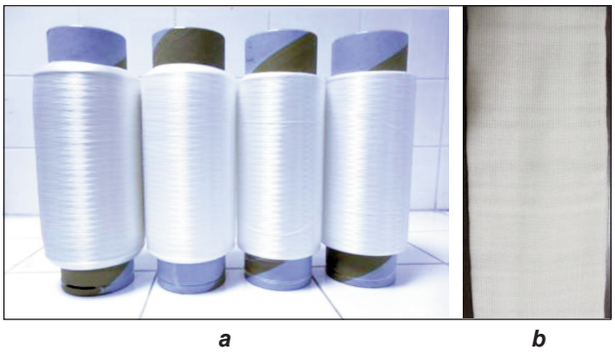
Table 2

PRODUCTION PARAMETERS OF PP FILAMENT YARNS			
Extruder parameters		**Drawing unit parameters**	
Zone-1 temperature	220°C	Godet-1 speed	390 rev/min.
Zone-2 temperature	240°C	Godet-2 speed	400 rev/min.
Zone-3 temperature	245°C	Godet-3 speed	800 rev/min.
Zone-4 temperature	245°C	Godet-4 speed	680 rev/min.
Extruder speed	143 rev/min.	Godet-5 speed	680 rev/min.
Extruder pressure	6.6 bar	Speed of textured drum	400 rev/min.
Pump speed	70 rev/min.	Winding speed	2050 m/min.

package. In addition, during the production process, all other parameters except for the factors whose effects were aimed to be investigated (ratio of additive material) were kept constant. Production parameters of PP filament yarns are given in table 2.

STRUCTURE AND PROPERTIES OF PP FILAMENT YARNS

PP polymer chips and FR additive were mixed at determined rates (99/1%, 98/2%, 97/3%, 96/4%, 95/5%, 94/6%, 93/7% and 92/8%) and fed to the system during production. First of all, 100% PP reference yarns have been produced and then, FR added PP filament yarns were produced. Also, these yarns have round cross-sectional shape and structure and the fineness of produced yarns were determined as "144f918denier" (figure 2).



a ***b***

Fig. 2. Produced materials in the scope of this study: *a* – PP filament yarns; *b* – structure of supreme knitted fabric [11]

To determine the structure and properties of the yarns, tenacity-breaking elongation, unevenness and intermingled tests were applied to the yarns. Filament yarn samples were conditioned for 24 hours at 20°C±2 temperature and 65±2% relative humidity, which are the standard atmospheric conditions before the tests. Tenacity-Elongation tests were carried out with a Uster Tensorapid-3 test device according to BS EN ISO 2062, 1995 test standard and unevenness tests were carried out with a Uster Unevenness Measurement device according to DIN 53817-1 test standard ("Using BS EN ISO 2062", 1995; "Using DIN 53817-1", 1983) [12, 13]. Also, the intermingling test was realized with the Rothschild Entanglement Tester R-2070 test device according to the "Internal Method". Additionally, the surface morphology of filament yarns was investigated with the help of a Scanning Electron Microscope-SEM (Jeol JSM-6390 LV) at a voltage of 20 kV. SEM images of PP filament yarns and test results are given in table 3.

As will be seen in table 3, while additive-free PP filament yarns have a smooth structure, granules caused by additives which had not been completely integrated into the structure are being observed on the surfaces of filament yarns produced by FR additive. Moreover, it was determined that the tenacity-breaking elongation and unevenness values of PP filament yarns do not change in a specific direction depending on the rate of additive in the structure. Intermingling numbers of filament yarns per meter were determined as 9–12 units [11].

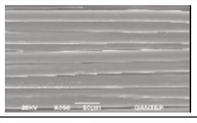
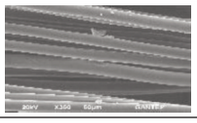
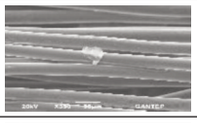
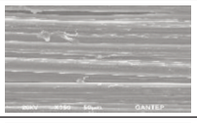
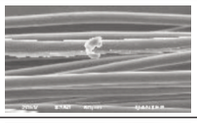
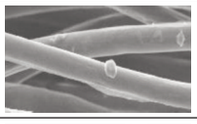
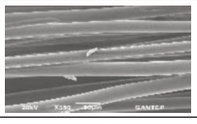
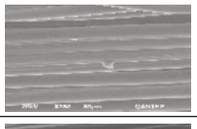
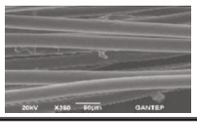
KNITTED FABRIC PRODUCTION FROM PP FILAMENT YARNS AND FLAME RETARDANCY TEST

PP filament yarns were converted to a knitted fabric structure (Supreme) to perform the flame retardancy tests. The knitting operation was performed at Lonati Goal-6715 brand knitting with a cylinder diameter of 3½ inches and by automatic yarn tension settings. As the result of the knitting operation performed by 54 needles and a machine speed of 160 rpm, yarns with supreme knitting structure were obtained. Supreme (Single jersey) is one of the basic knitted structures. Single jersey fabric is produced with the RL knitting technique. RL plain knitted fabrics have plain (R) loops on one side and only reverse (L) loops on the other. Supreme knitted fabrics are widely used in home textiles. As it is known, the flame retardancy property is desired from home textile products for safety. Therefore, a single jersey knitted structure was preferred in this study. The GSM of supreme knitted samples which are used in this study is 352 g/m^2. Also, the thickness of these knitted fabrics is 0.4 mm.

300×76 mm samples were prepared from the fabrics for vertical flammability test according to ASTM D 6413 testing standard and the relevant test was

Table 3

SEM (LONGITUDINAL) OBSERVATIONS AND MECHANICAL PROPERTIES OF PP FILAMENTS [11]

Sample	SEM Pictures	Tenacity (cN/tex)	Breaking elongation (%)	Uster (U%)	CVm (%)	Intermingling (number of nips per meter)
PP filament yarn		22.8	28.4	6.94	7.49	10.1
1% FR		23.6	28.4	5.29	6.63	10.2
2% FR		23.0	27.4	5.45	6.88	11.0
3% FR		22.5	26.7	5.60	6.95	8.9
4% FR		22.7	27.3	5.98	7.53	10.1
5% FR		22.8	26.2	5.95	7.41	9.7
6% FR		23.4	26.6	5.37	6.64	10.0
7% FR		23.7	28.7	5.97	7.50	12.0
8% FR		22.5	26.7	6.17	7.63	9.2

applied to Atlas Brand Fire Science Product-AGC Automic Gas Control device (“Using ASTM D 6413”, 2011) [14]. By the beginning of the test, the ignition source at the central lower point of knitted fabrics was lit up, and the fabrics were exposed to flame with a length of 19 mm for 12 seconds. In this test, the ignition source dies following a period of 12 seconds and the period of continuation of the burning of the fabric is measured by a chronometer (“Using ASTM D 6413”, 2011) [14]. Limit Oxygen Index (LOI) test was applied to knitted fabric samples as well. These samples were prepared with a dimension of 140×52 mm as per ASTM-D 2863 testing standard by using a Dynisco brand device (“Using ASTM D 2863-10”, 2009) [15]. For both tests, the samples were exposed to the same process 15 times, and the averages of the results were taken. Before tests, the knitted fabric samples were conditioned for 24 hours at 20°C±2 temperature and 65±2% relative humidity which are the standard atmospheric conditions. After testing, the effect of the rate of FR additive on the PP knitted fabrics was statistically analysed by using the “One-Way Variance Analysis (ANOVA)” method. The statistical study was realized at $\alpha = 0{,}05$ reliability level.

RESULTS AND DISCUSSION

Vertical flammability test-char length

Vertical flammability test is being used under controlled laboratory conditions to measure the reaction of textile materials against heat and flame (“Using ASTM D 6413”, 2011) [14]. This test is being applied in order to determine whether the fabric continues to burn or not after cutting the source of flame applied to the sample fabric. During the test, as the sample is directly exposed to flame from its central lower point, significant results are obtained by the end of the test in respect of determining the flame retardancy property of the samples. By this test, many burning

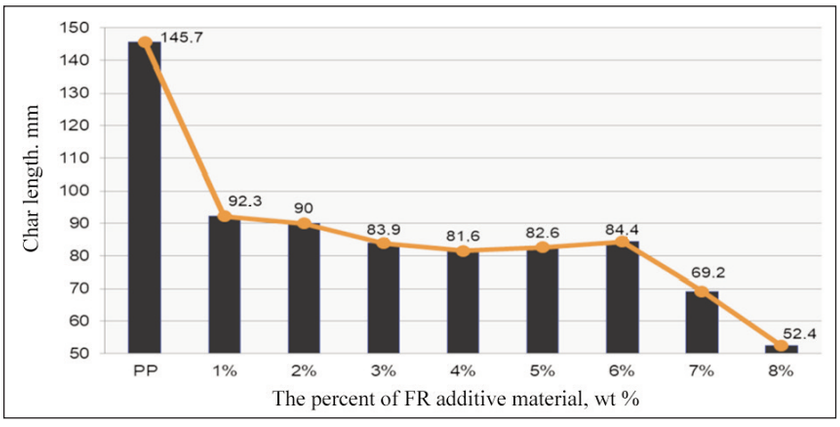


Fig. 3. Effect of flame-retardant additives on char length of PP knitted fabrics (Significant: 0.000)

properties of the fabrics such as "afterflame", "afterglow", "aftertime", dripping behaviour and burning behaviour are determined. This test in the subject is generally used to determine the flame retardancy property of textile materials such as apparel fabrics, upholstery, curtains etc.

During the study, the char length and after-flame time of the fabrics were determined by the vertical flammability test being applied to PP knitted fabrics. The change of char length values of PP knitted fabrics according to different rates of FR additive are presented in figure 3.

Descriptive statistical data table of the char length of FR added filament yarns is given in table 4.

ANOVA table of char length of FR added filament yarns is given in table 5.

From figure 3, it is seen that the increase in the amount of FR additive significantly decreases the vertical char length of the samples. From the graph (figure 3), it is seen that the PP knitted fabric which doesn't include FR additive has a char length of 145.7 mm and that the PP knitted fabric with additive has a char length much lower than this value. Especially, the 52.4 mm char length of the PP knitted fabric with 8% FR additive is remarkable. This result indicates that 8% significantly improves the flame retardancy property of knitted fabrics. And another remarkable result is the similarity among the char lengths of knitted fabrics including an additive in between 3–6%; not a significant decrease tendency but similarities were observed among these results. The change in the additive rate of 3–6% had not improved the FR property of fabrics. Consequently, this condition can be defined by similar flame retardancy properties of test samples including FR at rates of 3%, 4%, 5% and 6%. By the addition of additive at a rate of 7%, it was observed that the flame retardancy property of the fabrics significantly improves. Moreover, according to the

Table 4

DESCRIPTIVES								
Sample	**N**	**Mean**	**Std. Deviation**	**Std. Error**	**95% Confidence interval for mean**		**Minimum**	**Maximum**
					Lower bound	**Upper bound**		
100% PP	9	145.667	37.1113	12.3704	117.140	174.193	90.0	200.0
1% FR	9	92.333	11.5217	3.8406	83.477	101.190	79.0	118.0
2% FR	9	90.000	13.8203	4.6068	79.377	100.623	60.0	101.0
3% FR	9	83.889	8.1001	2.7000	77.663	90.115	73.0	100.0
4% FR	9	81.556	17.0449	5.6816	68.454	94.657	59.0	111.0
5% FR	9	82.556	14.9592	4.9864	71.057	94.054	60.0	105.0
6% FR	9	84.444	7.6012	2.5337	78.602	90.287	71.0	95.0
7% FR	9	69.222	19.7976	6.5992	54.004	84.440	30.0	89.0
8% FR	9	52.444	11.8544	3.9515	43.332	61.557	35.0	71.0
Total	81	86.901	29.2539	3.2504	80.433	93.370	30.0	200.0

Note: Char length (mm).

Table 5

ANOVA					
Description	**Sum of squares**	**df**	**Mean square**	**F**	**Sig.**
Between groups	45493.877	8	5686.735	17.826	0.000
Within groups	22969.333	72	319.019		
Total	68463.210	80			

Note: Char length (mm).

results of statistical analysis, as the significance value of test results of the char lengths of knitted fabrics was found to be 0.000 at $\alpha = 0,05$ reliability level, the effect of the amount of additive on the char lengths of knitted fabric samples was also statistically significant.

Vertical flammability test-after-flame time

Another data obtained as the result of the vertical flammability test applied on PP knitted fabric samples is the time in which the fabrics continue to burn following the duration of exposure to flame. Figure 4 indicates the effect of change in FR additive rate on after-flame time values of PP knitted fabrics.

Descriptive statistical data table of after flame time of FR added filament yarns is given in table 6. ANOVA table of after flame time of FR added filament yarns is given in table 7.

From figure 4, it is seen that against the increase in the amount of FR additive, after-flame time following 12 seconds of exposure to the ignition source significantly decreases. It was determined that the additive-free PP knitted fabric samples burn for about 46 seconds following 12 seconds of exposure to flame and that the fabrics produced by adding additive have much lower after-flame times than this value. The change in the direction of the increase-decrease of the after-flame time of knitted fabrics including FR additive in between 5% and 8% can be assessed as a remarkable result. This condition can be interpreted as reaching a specific flame retardancy level of knitted fabrics including 5% additive and as observing similar burning behaviours in samples even by the addition of a higher rate of additive in the yarn. Moreover, as the significance value of test results of the after-flame time values of PP knitted fabrics was found to

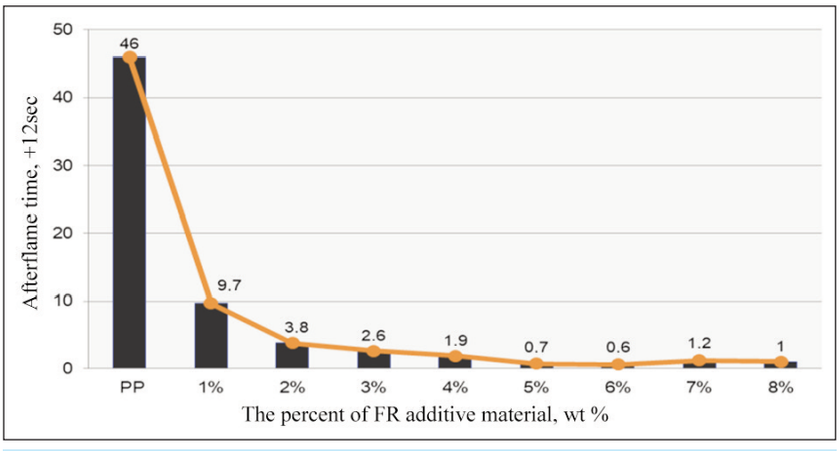


Fig. 4. Effect of flame-retardant additives on after-flame time of PP knitted fabric (Significant: 0.000)

Table 6

DESCRIPTIVES								
Sample	**N**	**Mean**	**Std. Deviation**	**Std. Error**	**95% Confidence interval for mean**		**Minimum**	**Maximum**
					Lower bound	**Upper bound**		
100% PP	9	46.067	20.6870	6.8957	30.165	61.968	27.9	91.5
1% FR	9	9.689	5.3471	1.7824	5.579	13.799	4.5	19.6
2% FR	9	3.822	2.7730	0.9243	1.691	5.954	0.0	8.0
3% FR	9	2.633	1.6793	0.5598	1.343	3.924	0.0	4.9
4% FR	9	1.911	1.0764	0.3588	1.084	2.738	0.6	3.7
5% FR	9	0.656	0.5341	0.1780	0.245	1.066	0.0	1.3
6% FR	9	0.578	0.9667	0.3222	-0.165	1.321	0.0	2.9
7% FR	9	1.167	0.9975	0.3325	0.400	1.933	0.0	2.6
8% FR	9	1.044	0.6598	0.2199	0.537	1.552	0.0	2.1
Total	81	7.507	15.5730	1.7303	4.064	10.951	0.0	91.5

Note: Afterflame time (+12 sec).

Table 7

ANOVA					
Description	**Sum of squares**	**df**	**Mean square**	**F**	**Sig.**
Between groups	15634.562	8	1954.320	37.355	0.000
Within groups	3766.893	72	52.318		
Total	19401.456	80			

Note: Afterflame time (+12 sec).

be 0.000 at α=0.05 reliability level, the effect of the amount of additive on the after-flame times of knitted fabric samples was also found to be statistically significant. Another result of the vertical flammability test is relevant to afterglow behaviours of knitted fabrics; along with the vertical flammability test applied on PP knitted fabrics with and without additive, afterglow had not been observed on any of the fabrics. And a significant dripping behaviour was also not observed on the samples during the tests, and dripping was observed only on additive-free PP and on a few knitted fabric test samples with 1% and 2% additive.

Limit Oxygen Index (LOI)

LOI test was also applied on knitted fabric samples produced from PP filament yarns to support the results of the vertical flammability test. By this test method, the reactions of materials against heat and flame under controlled conditions are measured and defined ("Using ASTM D 2863–10", 2009) [15]. By the end of the test, the minimum oxygen amount required to be available in the environment for the continuation of burning is being determined. The LOI test method provides a good measurement regarding the flammability properties of polymeric materials, and it can determine the flame retardancy characteristics of the materials. In these respects, this test was deemed to be significant and was used in this study. Figure 5 indicates the change in LOI values of fabrics as per the flame retardancy additive rate.

As the oxygen amount in the air is about 21%, any material with an LOI value lower than that value can easily burn in the current air condition. On the contrary, the materials with an LOI value over 21% slow down or stop burning when the ignition source is removed. Figure 5 shows that the LOI value of additive-free PP was calculated as 19% and LOI values of samples in which FR additive was added had significantly increased as per that reference value. It is known that the LOI value for PP polymeric materials is about 17–18%, and the LOI value of the PP sample fabric structure used in the study was higher. It can be interpreted as it had arisen from the condition that the linear density (918 denier) of continuous filament yarn was high [11]. It was also observed that the LOI values of PP knitted fabrics significantly increased by the addition of FR additive at a rate of 1%. According to the literature, it was determined that the samples including FR substances at rates of 1%, 2%, 3%, 4%, 5% and 6% are in the "Slowly Flammable" products category due to their LOI values of 25, 26, 27 and 28 respectively and that the samples including additive at a rate of 7% and 8% are in the "Flame Retardancy-Self-Extinguishing Material" class due to LOI value of 30 [16, 17]. When studies are included in the literature relevant to improving the flame retardancy properties of textile products, it is observed that the LOI test determining the resistance of materials against burning had been used in many scientific studies. In these studies, the LOI test had generally been used to support the other flame retardancy tests [2, 11, 18].

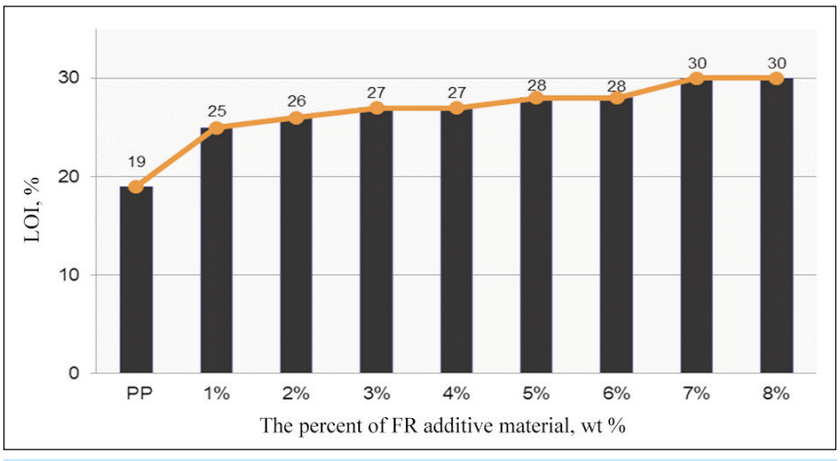


Fig. 5. Effect of flame-retardant additives on LOI value of PP knitted fabrics

CONCLUSION

Polypropylene fibre is significantly being used in the textile industry and especially in the field of technical textiles. However, it is a significant disadvantage that polypropylene fibre has lower resistance to flame. Thus, many research and application studies are being performed today to bring flame retardancy properties to PP textile products. In this study, knitted fabric samples were produced by additive-free and FR added PP filament yarns, and then Vertical Flammability and LOI tests were applied to these samples to examine their flame retardancy properties. When the test results were examined, it was observed that the increase in the additive rate of the samples significantly decreases both the after-flame times and char lengths of PP knitted fabrics. The average char length value of additive-free PP knitted fabrics was determined as 145.7 mm, and this value decreased to 52.4 mm with the increase in the additive rate. Again, the average after-flame time value for additive-free PP knitted fabrics was calculated as 46 seconds, and this value gradually decreased with the increase in additive rate, and it had decreased below 1 second by the addition of 5% additive.

Moreover, the results of the LOI test supported the vertical non-flammability test results. While the LOI value of additive-free PP knitted fabric was found as 19%, it was observed that this value significantly increases with the increase in the additive amount. LOI values of the samples with 7% and 8% FR were found as "30". The obtained result indicated that these samples have "Self-Extinguishing" properties. Consequently, this study showed us that the flame retardancy properties of knitted fabrics produced from PP filament yarns

can be improved significantly by the organophosphonate-based additive.

Investigation of the lifetime and performance of the functional properties (such as non-flammability) of fabrics is an important issue. Investigation of the flammability performance of fabrics after washing and abrasion was determined as the next study. In addition, the effect of permeability properties of knitted fabrics on non-flammability is another important research topic. Also, another future work is currently being prepared and the subject of this study is examining the burning behaviour of these fabrics. Images of samples before and after the vertical flame test will be given in this future work.

ACKNOWLEDGEMENTS

This work was financially supported by the "Turkey Ministry of Science, Industry and Technology" within the research program called SAN-TEZ, Project Number: 00428.STZ.2009-2.

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