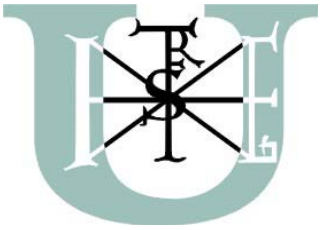

SZENT ISTVAN UNIVERSITY

THE ROLE OF OXYGEN INDEX IN THE FLAMMABILITY OF OXIDISED - AND CARBON FIBRES

Thesis of PhD work

dr. Zsuzsanna Kerekes

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Doctoral school
Denomination: **Mechanical Engineering PhD School**

Science: **Agricultural Engineering**

Leader: **Prof. Dr. Istvan Farkas**
Dr. of Technical Sciences
Faculty of Mechanical Engineering
Szent Istvan University, Gödöllő, Hungary

Supervisor: **Dr. Beda László, PhD**
Szent Istvan University Ybl Miklós Faculty
of Architecture and Civil Engineering Institute
of Fire Protection and Safety Engineering, Budapest,
Hungary

Co-supervisor : **Prof. Dr. István Barótfi, CSc**
Faculty of Mechanical Engineering
Institute of Environmental Systems
Szent Istvan University, Gödöllő, Hungary

…… ……

Affirmation of head of school **Affirmation of supervisor**

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1. INTRODUCTION

Carbon fibre based products, due to their special properties, are also used in applications where heat and fire resistance need to be ensured, e.g. in protective clothing including protective gloves, fire blankets and internal lining of vehicles. These carbon-based products and the carbon fibre itself are qualified, beside their mechanical and physical properties (fibre density, fibre diameter, tensile strength etc) by their oxygen index.The Fire Protection Testing Laboratory of the Institute of Fire Protection and Safety Engineering has quite a long time dealt with measuring the oxygen index of carbon fibres and oxidised fibres.The well-known natural carbon is burning well, but in the man-made structure it is inflammable in normal athmosphere. The carbon fiber (CF - carbon fiber), which contains more than 95% carbon and the oxidized fibers (PN, Pyron as a brand name) containing approx. 65% oxygen, were developed, with high carbon content that -in addition to the advantageous mechanical properties – to have combustion- and flame-resistance. In normal atmosphere they do not burn, so most of the standard fire tests are not suitable for the determination of their combustibility. This feature makes them to the most modern fire-proof materials. The carbon and the oxidized fibers have both a micro-structure and after the final processing a macro structure as well, which together determine the final quality of the products. The complex raw material production and manufacturing (through chemical and physical processes) change the micro-and macro-structure of the fibers.

Goals and tasks: I would like to give a literary overwiew of a reletively new area of materials science – less published from the point of view of flammability – structural materials, oxydised and carbon fibers, which are considered to be specific.

1. I perform my measurement in increased oxygen atmosphere which makes it possible to measure material while burning which is normally inflammabele in normal airy atmosphere. Further on it is possible to observe types of burning which would not be possible in normal airy atmosphere.
2. I am looking for explanations, reasons and coherence what causes and which parameters influence the flammability of oxydized carbon fiber material. Furter I examine what kind of mutual impact of micro and macro structures plays role in flammability.

3. My aim was to develop an burning classification for fabrics and fibres.

2. MATERIAL AND METHOD

2.1. Selection of test methods

The burning tests were made in standard equipment as described above, All the samples were tested before and after the combustion with infrared and Raman spectroscopy and elementary - analysis.

In my work the following test methods thought appropriate, and opted to set a target to reach those formulated:
- *Oxygen index tes*t, which I can characterize the combustible of the materials,
- *Construction materials testing* to find out what kind of microstructural parameters can be the influence factors of the combustibility,
- *Element analytical studies*, to the relationship between the combustibility and chemical composition.

2.1.1 Method of Measurement for Oxygen Index

There are several methods to describe the flammability of various materials, ignition tempetature, the flame propagation in different directions, smoke densities and fume temperatures. The flammability of materials can also be characterized by their minimal oxygen concentration required to maintain of burning.

The majority of flammable materials are capable of combustion at normal oxygen levels (21 vol %.), but there are materials that burn at lower or higher oxygen concentration in comparison. Oxygen index (LOI – Limited Oxygen Index) is not a commonly practiced laboratory measurement method, despite its being a significant parameter in the characterisation of flammable materials. This method has originally been developed to describe the flammability of plastics, but in principle it is applicable to all combustible solid materials. The LOI parameter is used for fire protection qualification of carbon fibres in the literature, oxidized fibres and materials made from those. The samples under investigation are to be produced locally, after solving initial problem.
The precise circumstances of the measurement are regulated by standards. (Plastics - Determination of flammability by oxigen index) and international standards (ISO 4589, ASTM 2863). By definition oxygen index is defined by the minimum oxygen content of the air, where the material is still capable of fire propagation or burning in a defined time interval

2.1.2 Infrared and Raman spectroscopy analysis.

Measurements for the structure investigations were considered important for two reasons. Measuring of the different spectra is a first step to determine the cyclisation, stabilization and carbonisation indices. On the other hand, the spectroscopic studies can follow the micro-level structural transformations in production steps identifying the most important groups (Table 1).

The most characteristic changes are:

1. The cyclisation of hangi g cyanide groups (-C = N) leads to ladder type structure
2. The dehydrogenation results in conjugated double bonds
3. During the oxidation a carboxyl group (C = O) is created, and the loosely bound hydrogen escapes.
4. The (-C ≡ N) and C = O / C-O groups which are hanging on C-H chain are being transformed accompanied by the decrease in absorbance at C-H locations (at approximately 2900 cm -1), due to the dehydrogenation.
5. The C ≡ N binding peak at 2240 cm -1 decrease as a sign that the linear cyanide groups transform to cyclic structure.

In fact a heterocyclic structure is formed with C = N, C = O bonds, besides the remaining C-H bonds.

Testing Groups

Group 1: polyacrylic-nitrile (PAN) as a raw material, various forms depending on ascending treatment temperature. Identification as: PAN, OX1, OX2, PN, LT, HT, CF.

Group 2 : PN 37 TW oxidized fiber (fiber density: 1.37 g/cm3, LOI: 44.5), which were combusted under following oxygen content: 21% (baseline), 30%, 40%, 50%, 60%, 70% and 80%. Number of samples: 7.

Group 3: CF fibre, with 98% carbon content, LOI over 90.

These samples were considered as the closest reference to pure graphite.

Table 1 Absorption maxima of some most importand bonds and functional groups

- C≡N linear cyanide	C=N cyclical cyanide	C-N amine	-C=O linear	-C=O cyclic	-CH alkane	C-C aromatic	C=C aromatic	C–O carboxyl
2240	1590	1340-1020	1700	1780 és 1840	2960-2850	1580	1600	1000-1300

2.1.3 Elementary Analysis

The tests were performed on fiber cables grouped by lower and higher density. They were combusted in different oxygen concentrations. The impact of combustion parameters on fiber structure, mass loss and elementary composition was investigated. The selected elements of the composition were the following: carbon, nitrogen, hydrogen.

Sample groups and their properties:
PN 1: fiber density of 1.37 g/cm3, LOI 35,
PN 2: fiber density of 1.41 g/cm3, LOI 61,
As reference
CF: fiber density of 1.78 g/cm3, LOI above 90 were used.
The elementary analysis measurements were performed in Central Chemical Research Institute on Elementar Vario EL III CHNOS Analyzer.

2.2 Testing Groups

According to their macrostructure the samples can be classified in to following groups:

1.Samples of **cables**, ie. **bundles** of fibres
2.**Vlies** or felted materials (non-woven, felted textiles). Their usual thickness is 5-8 mm.
3. Woven textile samples, a./ pure oxidized fibers (100% PN tissue), notation PW (plain weave canvas fabric) b/. mixed samples are fiber blends with (PVC, PA, PES).

2.2.1 Characterization of the Cable Samples

Cables consist of fiber bundles. Cables are the starting material for more complex products (vlies, fabric, composite). A fiber diameter varies in range of 7-12 microns. The microstructure of fibers is determined by their chemical composition, crystal structure and surface morphology.

In my work for qualifers fiber density (g/cm3) , and the linear mass density (dtex) were used. For the structural characteristics of fibres are futher used c*arbonisation index, a ciklisation index, and stabilsation index.*

Following the steps in the production technology the test samples undergo changes in carbon content beginning with the raw material PAN up to the theoretical maximum at CF (with

graphite-like structures). Table 2. shows the specific properties of these samples and the measured oxygen index

Table 2 Changes in the important properties of cable samples depending from the treatment temperature

Sample	***Treatment temperature*** ***(°C)***	***Carbon content*** ***(%)***	***Fibre density*** ***(g/cm^3)***	***Fibres thickness*** ***(mikron)***	***Measured oxygen index (LOI)***
PAN precursor	20	44	1,17	12	**18,3**
Ox1	220-2240		1,218	12	**18,7**
Ox2	240-250		1,285	12	**26**
Ox3	250-270	cc. 60	1,35	12	**33**
LT (Low Temp)	600-900	cc. 70	1,776		**68±2**
Intermedier	600-900	cc. 70	1,784		
HT (Hight Temp)	1100-1400		1,774	6	**83**
Carbon fibre (CF)	1300-1700	(95 % C)	1,82	6	**86,5**

2.2.2 Not woven textiles (felt, vlies)

In in case of non-woven textiles only the macrostructure should be considered. As characterization parameter the areal weight (g/m2) can be used. The measured samples have values from 80 to 500 g/m2. The thickness of nonwoven materials in the production process is typically 5-6 mm. In case of low-weight, loose samples variations in thickness and color changes may occur.

2.2.3 Fabrics, woven fabrics

Similar to the non-woven fabrics, tissues have also their macrostructure. The investigation should take into account the direction of the weave, the density of warp and weft. The final product is a pure oxidized fibre or a blend with synthetic fiber or glass fiber (PVC, PA, PES), The woven fabrics were characterized by areal weight (g/m^2) and yarn density.

The testing samples and their parameters are summarized in Table 3.

The notations used in the Table refer to the type of construction, the numbers refer to the groups of parallel samples. The notations: PW - plain wave, SW - satin wave, TW - twill her-ing bone, AS - textile from Japan, KF - Knitted Fabric . The samples were made from 100 %

oxidized PAN fibre. In the present paper the overall effects of the textile parameters on the oxygen index, including the type of construction, are examined.

Table 3. The testing samples and their parameters

Type of fabric	**Sample identifity**	**Type of constuction**	**Areal weight g/m^2**	**Fibre, dtex**	**Fibre density g/cm^3**	**LOI, %**
Woven fabric	PW01	plain	190	1.7	1.35	29.9
	PW02	plain	360	1.7	1.37	30.2
	PW03	plain		1.7	1.35	32
	PW04	plain	340	1.7	1.4	50
	SW01	Satin Weave	460	1.4	1.37	31
	SW02	Satin Weave	470	1.7	1.39	35.4
	TWH	Twill hering bone	420	1.7	1.395	43.3
	TW*	Twill hering bone	980	1.7	1.4	58
	TWC	Twill	410	1.7	1.4	53
	AS	plain	400	1.7	1.42	54
Knitted fabric	KF01	Double Inter-lock	340	1.7	1.35	31
	KF02	Double Inter-lock	340	2.2	1.37	32.1
	KF03	Double Inter-lock		1.7	1.37	33.5

3. RESULTS

3.1 The Combustion Properties of Samples

According to my observations, the combustion characteristics are influenced by:
- The consistency of the sample (the examined macrostructure)
- Measurement conditions,
- The combined effect of the obtained microstructures and macrostructures during the production process of fibers and final products.

The common property of the samples with different structures (cable, vlies, cut thread) is that their surface is not smooth, but fuzzy. In the case of normal measurements, the sample vertically secured in a U shaped sample-holder burns downwards when ignited. Especially these fibres projecting outwards make measurement problematic. These surface fuzzy piles ignite significantly easier than the interior of the material itself. The burning instantaneously runs down on both sides of the sample, whilst the interior of the sample remains unchanged. The appearance of this so called surface flame is dependent on a certain level of oxygen concentration. By reaching another determined oxygen content, this surface flame is able to ignite the sample from below resulting in the upward spread of burning.

This is not according to the standards but nevertheless it is uniquely characteristic of the sample. The speed of upward spread of combustion is considerably greater than downward spread of burning to be expected, therefore before we could take measurements with true value, the sample has already burnt. In the case of such samples it is especially important to take care of securing the sample to be properly tensioned and that the length of the sample would be stretched to the bottom of the frame serving as a flame stopper. The majority of carbon fibre based samples can be characterised by several oxygen concentration, including LOI as well. Ie. the various flame phenomena happen at different oxygen concentrations determined by material and structural characteristics. Surface fuzziness is the most problematic in case of vlies but especially in the case of tread, frequently inhibiting correct measurements.

When selecting samples, it is an important to have samples of equal thickness and evenness. In the case of vlies samples - even if they are made of the same fibre – selection is required to be based on thickness due to technological limitations which do not ensure even thickness distribution. Beyond thickness consideration needs to be given to density and looseness. These char-

acteristics can be judged by eye, however, differences in 1-2 LOI can still occur. By paying attention to these, we can achieve good reproducibility.

Vlies samples show most sensitively the degree of dependency on structure by LOI. The danger of re-ignition is most pronounced in these samples, because the presence of fuzzy piles is inherent to the material and it can't be influenced by mounting. Surface flame can appear in cases when the top of the sample cannot be ignited and therefore usual flame spreading cannot be observed. If the material is rather loosely structured (tread), even the surface flame is capable of burning through the whole thickness of the material.

As with all measurements, reproducibility is the most important requirement here as well, where evenness of samples plays a significant role. The difficulty of the selection within the same sample is partly due to the selection of structurally similar parts (sample taking).

In the case of composite materials, structural inhomogeneity, indicated by colour differences as well, means further dispersal during measurements. Naturally this means qualitative differences in the material.

In the case of bundles we can ignore the inhomogeneity of the fibres. Fibres protruding from the surface can be minimised by the tight mounting of the cable. Burning can be best evaluated in

3.2 Parameters influencing the oxygen index of cable samles

The oxygen index (LOI) is primarily determined by the fiber density, but there are other factors, such as:

1. Fussiness of the surface,
2. yarn density,
3. Effect of sample size,
4. fiber orientation,
5 fiber length density (dtex)
6 drawing speed,
7 surface treatment.

Fibre density is the easiest and the mostly measured parameter during the production Figure 1 shows the oxygen index (LOI) of more than twenty samples with different densities

There is a clear correlation between the fibre density and combustibility. Nearly a hundred measurement points recorded in the range from 1.34 to 1.50 g/cm^3 show approximately a linear relationship. Fibers with density above 1.4 g/cm^3 show only a glow like flameless burning.

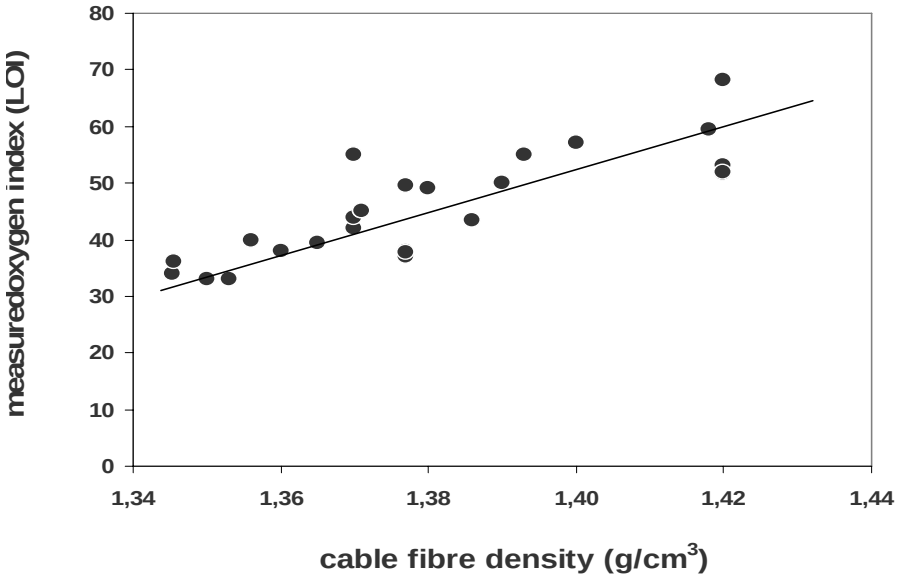


Fig. 1. Effect of the fibre density on LOI

3.3 Parameters influencing oxygen index of Non-woven fabrics

In this sample group, the influence of macrostructure on the flammability parameters was examined. The question was whether the oxygen index of fibres bundled in cables changes if they are organised into another macrostructural form. The characteristic parameter of non-woven textiles (felt) patterns is the surface density (g/m^2), i.e. is how loose or closed is the non-woven sample.

The following influences could be determined:

The combustion properties and the oxygen index values are determined by the macrostructural properties, especially from the surface density. The loose and closed felts have different fussiness of the surface. Like in case of cable samples, the fussiness plays dominant role in the combustion.characteristics.

The LOI value is linearly related to the surface density. If the oxygen index of samples is greater than 50 the surface shows only flameless glowing. This can be reached at surface density of over 500 g/cm^2. A surface flame could not be detected even above 50% oxygen content.

Additional measurements show also that there is large spread of 7 in LOI values at high fiber

(1.38 to 1.39 g/cm^3), In the case of small density values (below 1.37 g/cm^3) the spread on the oxygen index is much smaller. This confirms the assumption that oxygen index of macrostructures made of PYRON fibers is strongly dependent on the structure

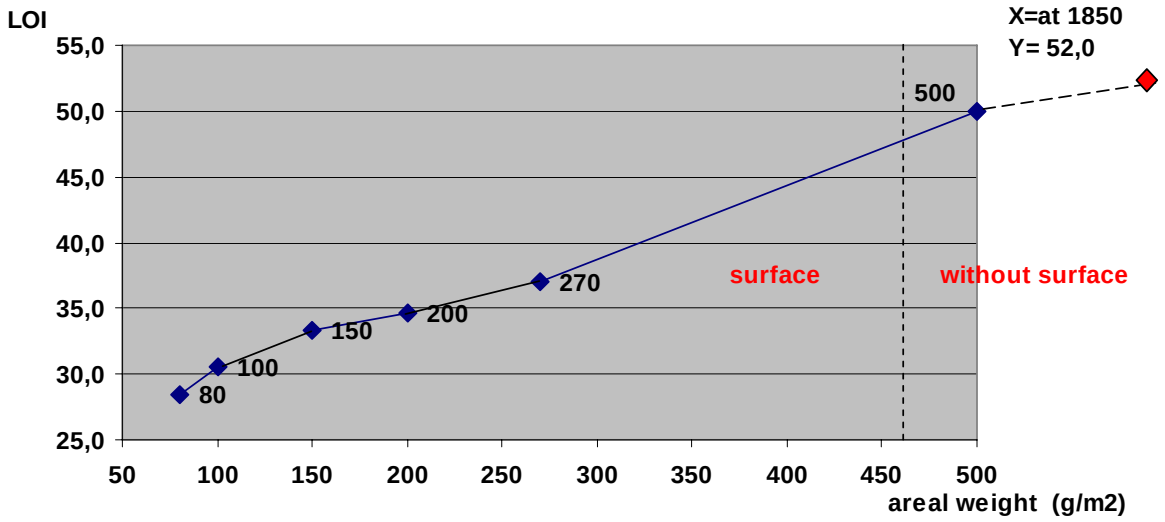


Fig.2. Areal weight parameters influencing oxygen index of Non-woven fabrics

3.4 Parameters influencing oxygen index of woven fabrics

In the function of the textile area weight two LOI ranges can be separated (Figure 3).

The first range appears in low area weight, under 360 g/m^2, to which belongs LOI about 30. In this range the fibre density of the basic fibre of the textile is also low, it is under 1.37 g/cm^3, and the oxygen index as a function of the area weight is constant. In the range where the fibre density is higher then 1.4 g/m^3 the LOI of the product made from this fibre grows significantly. The fibre density looks to be determining. This statement will be confirmed, if two textiles with the same fibre density are compared (1.37 g/cm^3), one is the SW01 which area weight is 460 g/m^2 and the other is the PW02 which area weight is 360 g/m^2, both LOI value is nearly 30 (Table 1).

The second range appears in the range of area weight more than 370 g/m^2, to which belongs LOI more than 50. In this range the common parameter is that the fibre density is 1.4 g/cm^3.

In case of the TWH samples with high fibre density it was observed as well, that at the edge of the textile where the areal weight is higher because of the technology, the value of the oxygen index is higher (LOI = 52), while in the middle part of the sample a lower value (LOI = 43.3) could be measured. The difference is significant, 8-9 units. The edge of the sample is not flammable in 43.3% oxygen content, while the middle of the sample is flammable.

Difference could be observed between the low and high area weight textiles regarding their burning phenomena. The samples with LOI < 29-30 were carbonised after burning and fragile frame remained, while the dense textiles even kept their woven pattern and in some cases they kept their tear resistance too. This is also true for knitted fabrics, for which the oxygen index in elastic (weft) direction is LOI = 33.0, in inelastic (warp) direction is LOI = 33.3. 2-3 mm flame length is typical for their burning. They keep also their elasticity after burning despite their thickness.

Summarized: the changing of oxygen index is not linear with the macro structure parameters. In woven macro structures, a sharp step can be observed in LOI value at areal weights 370 g/m^2 at closed textile structures. This means by the view of flammability, that textiles with low area weight can burn easier than textiles with higher area weight. Regarding the flammability the closed woven textiles are well resistant. At low woven density a micro structure parameter, the fibre density will be dominant.

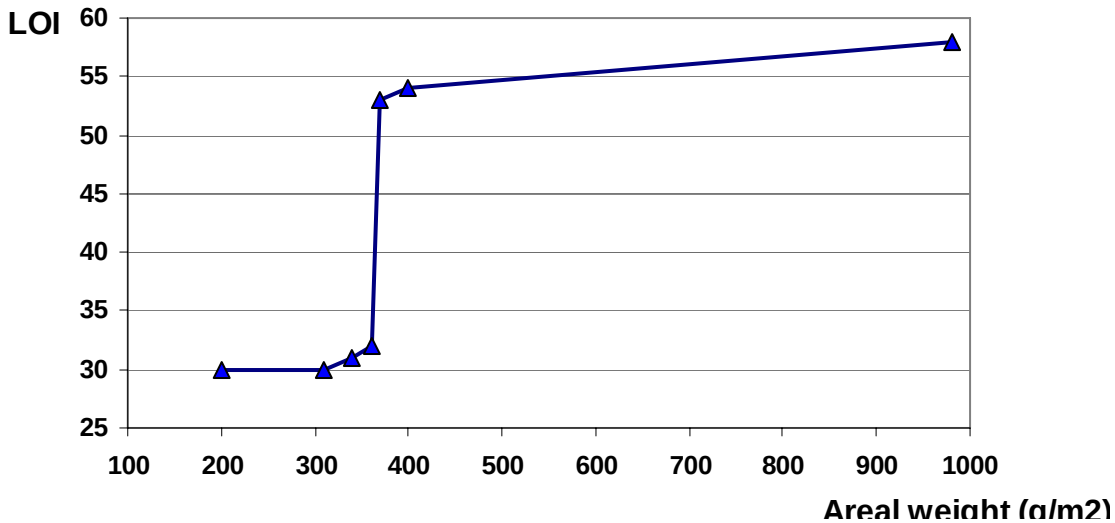


Fig. 3. Effect of area weight on the oxygen index

3.5. Influence of changes in elementar composition

At 50% oxigen concentration starts to the structure change drastically and a parallel increase in weight loss. The oxygen content of 50% over the incineration of hydrogen changes the steepest decline, PN1 samples, 2.38% to 1.70%, and PN2 sample. 3.05 % to 1.35% in change. The remainder is most likely that the moisture content recorded afterwards. It shows to 50% oxygen in the dehydrogenation is complete incineration, and combustion occurs against a more stable structure (Fig. 4 and 5.).

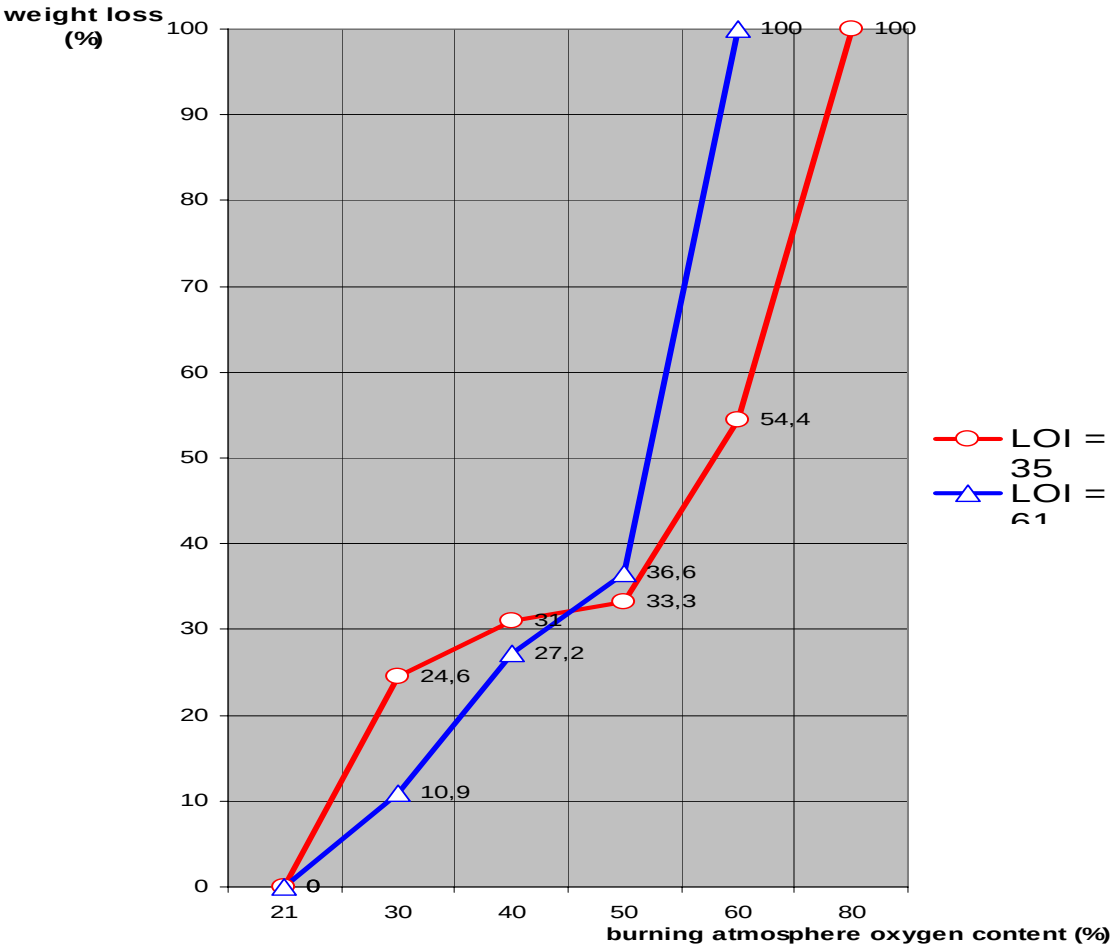


Fig.4. Mass loss changes of cable fibres /PN1(LOI=35) and PN2 (LO=I61)/ after burning

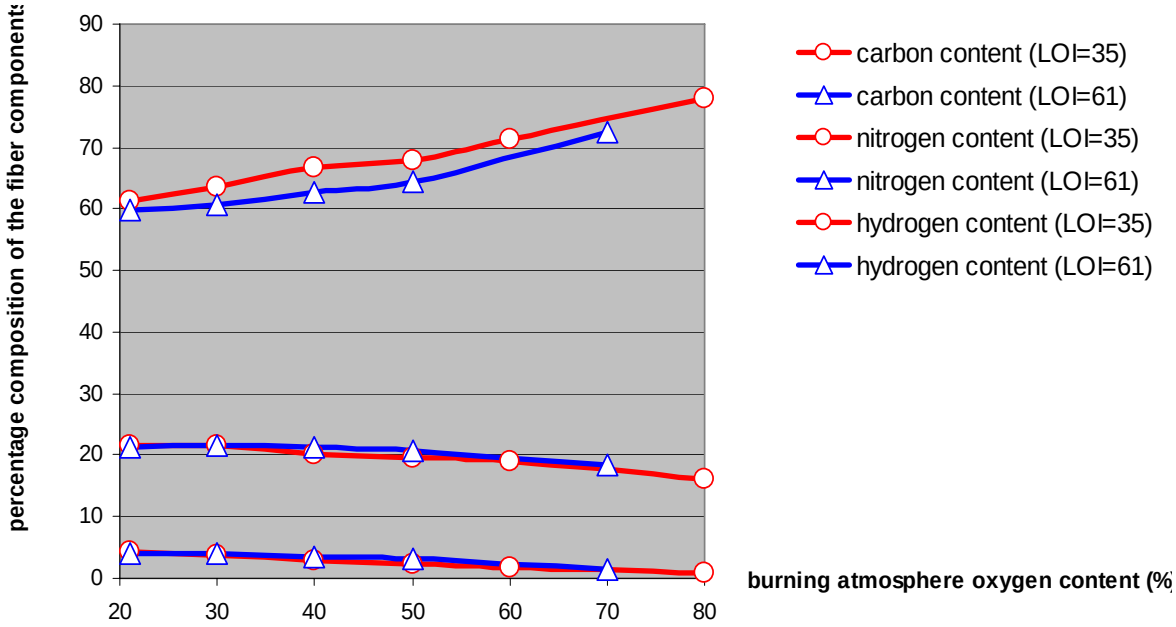


Fig.5. Composition changes of cable fibres /PN1(LOI=35) and PN2 (LO=I61)/ after burning

3.6 Results of measurements by infra and Raman spectroscopic

Structure studies are based on studies of the stabilization index (CI), a qualifying parameter based on spectroscopic investigations. The best way to follow the conversion rate (the PAN fibre transformation to oxidized fibre) is to measure the decrease in linear cyanides (C ≡ N at 2240 cm^{-1}) and the increase in related cyclic cyanides (C = N 1590 cm^{-1}). The absorption signal of the linear and cyclic cyanides shows the conversion rate, and indicates the oxidized fibre quality as well. To interpret the observed results so far, one has to know which structural changes occur in the process It is well known that the PAN forming linear cyanides (C ≡ N) in the stabilization step are being transformed into cyclic cyanides (-C = N-). This study shows that the transformation is not continuous, but staggered like the carbon content or the density increase.

Following the steps in the production technology the test samples undergo changes in carbon content beginning with the raw material PAN up to the theoretical maximum at CF (with graphite-like structures). Considering the microstructural parameters which are responsible for the increasing oxygen indices three distinct areas can be determined (Figure 7) The three

domains are also indicated by the appearance (-C = N-) or disappearing of the functional groups (C ≡ N).

Range1.: LOI 21-30 , The cyclization is ending and the number of linear cyanides (-C = N) reach its maximum value.
Range.2: LOI: 30-60, The remaining cyclic cyanides disappear, while the density increases. In this range the structure becomes thermodynamically stable against the oxidation processes, which is confirmed by LOI values above 50.
Range.3: LOI: 60-90 The graphitisation begins with a slight shrinkage of the fibres and a minimal decrease in fibre density could be also observed. This occurs when the cyano groups permanently disappear.

Raman spectra however are suitable for the monitoring of graphitisation. When it starts, the characteristic 1580 cm-1 first order and 1340 cm-1 second order peaks of the pure crystalline graphite appears.

The structural transformations of the PAN raw material are indicated by the quantitative change of the most common functional groups (C ≡ N groups decrease, C = N increase), the *graphitisation* rate (Raman peak of graphite at 1580 cm^{-1}), and changes of the fibre density and oxygen index. Note that the linear nitrile groups are changing gradually, while the cyclic nitrile groups are still present at quite high oxygen index values. The *graphitisation* starts only than when the nitrogen content is reduced or completely destroyed. This observation is consistent with the trends outlined in Figure 8. Very high LOI values can be related to pure graphite structures only.

The parameters carbon content, density and LOI are structure-dependent, their values change as the consequence of the drastic structural change due to heat treatment.

The structural parameters have influence on combustibility and on each other as well. There is a correlation between the oxygen index, the fiber density, the carbonization index, the stabilization index and also between the carbon content. Measurements have shown that the level of stability is well characterized by LOI. The structural transformations are followed by sudden changes of LOI values

The structural transformations are not linear but abrupt. Considering the temperature treatment ranges which are responsible for the increasing oxygen indices three distinct areas can be determined:

Range.1: LOI 21-30 , end of cyclisation

Range.2: LOI: 30-60, the structure becomes thermodynamically stable

Range.3: LOI: 60-90, the graphitisation process takes place

It has been confirmed by measurements that LOI can express the grade of stability. Based on spectroscopic measurements it has been also confirmed that the oxidized fibers dramatically decompose when burning in enriched air with more than 50% oxygen content. Materials with LOI> 50 possess the necessary thermodynamic stability against combustion. These materials are characterized by slow, flameless burning in solid-phase as the easily removable functional groups on surface no longer exist.

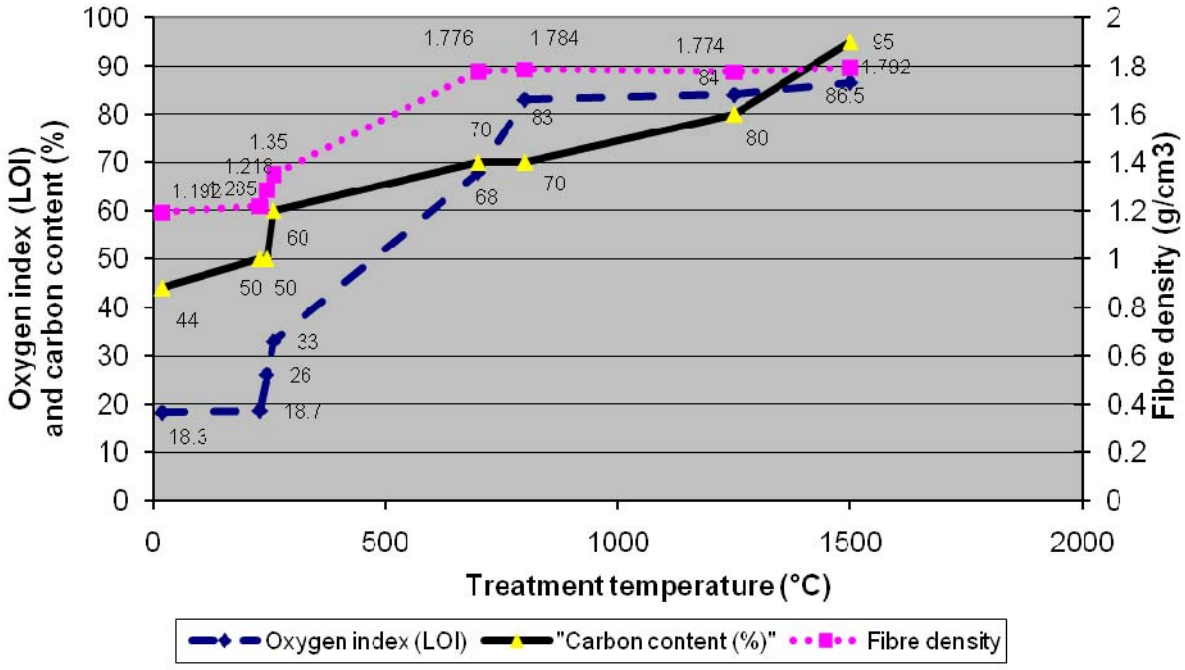
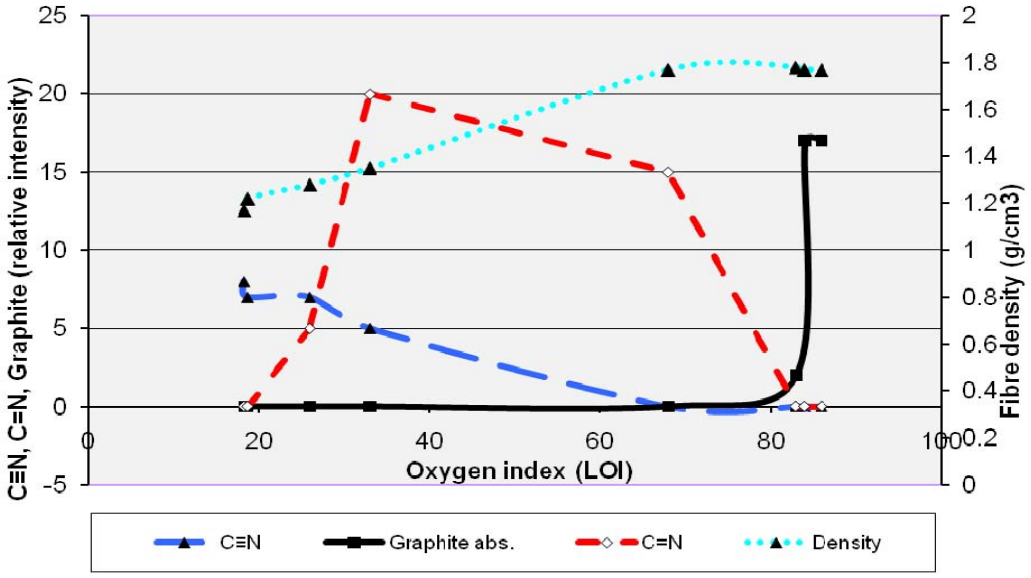


Figure 6. Changes in carbon contenet, fibre density and oxygen index as a function of the treatment temparature



Figur 7 Effect the grade of grafitisation, cyclic cyanides, linear cyanides on the oxygen index

4. NEW SCIENTIFIC RESULTS

1. Measurements have confirmed that to the various combustion phenomena separate LOI values could be assigned to which new flammability parameters can be created :
LOI (1), LOI (2), LOI (3).
The parameters are defined as follows:
LOI (1): the oxygen content, at which surface flame appears at the moment of ignition, and it spreads down on the surface swiftly while the sample inside remains unchanged
LOI (2): the oxygen content, at which surface flame appears at the moment of ignition, and it spreads down on the surface and the sample will be ignited from bottom side
LOI (3):): the oxygen content, at which surface flame appears at the moment of ignition, and it spreads down on the surface swiftly while the sample inside remains unchanged the sample can be ignited again at a higher oxygen content. This LOI(3) value is 6-8 higher than LOI (1) .

2. Found out that all the microstructural parameters affect the combustibility and these parameters are related to each other as well. A correlation can be found between oxygen index, fiber density, carbonisation index, stabilization index and the carbon content. The performed measurements confirm that the LOI values are not a linear function of the structural transformations, but an abrupt change can be detected.

3. It has been proved by measurements that can be sensitively measured as LOI to follow the microstructural changes in oxidized fibers, the method can be applied instead of other parameters, in technological process to monitor in-process changes. To follow the technological process, the following ranges of the oxygen index values can be defined:
I. initial range, the cyclization is complete: 20 ° - 250 ° C, LOI from 21 to 30,
II. the structure becomes thermodynamically stable: 250 ° - 800 ° C, LOI from 30 to 60,
III. the graphitisation process takes place at 800 °C, 1300 ° C, LOI from 60 to 90.

It could be shown that the flammability range is dependent from decrease or increase of linear (-C ≡ N) and cyclic (-C = N-) groups.

4. It has been confirmed by measurements that LOI can express the grade of stability. Based on spectroscopic measurements it has been also confirmed that the oxidized fibers dramatically decompose when burning in enriched air with more than 50% oxygen content. Materials with LOI> 50 possess the necessary thermodynamic stability against combustion. These materials are characterized by slow, flameless burning in solid-phase as the easily removable functional groups on surface no longer exist.

5. Infrared spectroscopy measurements proved that the surface of flames appear in the presence of C-H and O-H groups. The surface flames appear in range of the small LOI (LOI = 27-34) Measurements also showed that the appearance of the surface flame is most likely in case of loose macrostructures i.e. at small area weight values.

6. It was shown that the flammability of a fabric is determined by both the oxidized fibre as raw material (micro structure) and the woven structure of the knitted textiles (macro structure) made from them. The oxygen index of the flammability of the fibre will always less if this fibre is processed.

7 The changing of oxygen index is not linear with the macro structure parameters. In the function of the textile area weight two LOI ranges can be separated. The first range appears in low area weight, under 360 g/m^2, to which belongs LOI round 30. The fibre density looks to be determining. In case of felts show a stable non-combustibility when the surface density is over 500 g/m^2 where the LOI = 50.

8. As a result of measurements is could be shown that with the oxygen index values (between minimum and maximum) the macrostructural inhomogenity can be characterized.

5. CONCLUSIONS AND SUGGESTIONS

Both the spectral and elemental analysis studies lead to the result that the LOI = 50 value is a borderline separating the combustibility properties of the cables and the both structured forms (non-woven and woven fabrics).

When in the production process the fibers have reached the LOI value of 50the combustion tests prove that structure does no longer changes any more. Oxidized fibers with oxygen index above 50 are becoming more stable due to the increased graphite-like structures in them. This means that only air enriched with over 50% oxygen will be able to disrupt the structure. The test samples will hardly have hetero atoms and no surface flame could be observed in combustion tests due to disappearance of functional groups as C-H and C-O.

As a result of my investigations, it can be stated that the combustibility of end products are determined by the structure:

1. The microstructure of micro-structural properties of the fibers carry the cable. The fiber microstructure during heat treatment, forming polymer chains are increasingly interlinked by losing oxygen, hydrogen and nitrogen content. This process results in the consequence of causing a relative increase in carbon content. The increase in resistance to oxygen, or reaction to fire is reduced, the consequence of structural changes.

2. The macrostructure level is represented by the structure of the carbon fibre fabrics. Fibre type, direction, spacing and volume fraction can be varied to meet the mechanical or thermal requirements of each unique application. Varying weave constructions produces different physical characteristics, which may be beneficial for a particular usage. The structure of the fabrics depends on the applied weaving techniques and has a strong affect on the of the material.

LOI measurements can be a sensitive tool to follow the microstructural changes in oxidized fibers. With this measurements the in-process controls in technological process can be made cheaper and easier.

Proposals

1.Introduce new certification parameters for qualifying the products (LOI (1), LOI (2), LOI (3) as with these the combustibility can be characterized more accurately./

2. The in-process controls for determining the conversion with usual expensive and lengthy instrumental methods in oxidized and carbon fiber production process can be made cheaper and easier determining the oxygen index (LOI).

6. SUMMARY

The structure and flammability of a material registered amongst the most up to date once was investigated. The carbon fibers and oxidized fibers have excellent mechanical properties, in addition to the increasingly stringent fire protection requirements must be met.

As the end uses of these fibers are in the open market products (woven non woven textiles), their qualification from the flammability point of view was the subject of this paper too.

In the first part of my investigations the burning phenomena of the samples were tested. The test parameters varied from 21-95% oxygen content that was produced in a standard equipment for oxygen index measurement. The investigations were performed at the Szent Istvan University Ybl Miklps Faculty of Architecture and Civil Engineering Institute of Fire Protection and Safety Engineering.

The oxidized fibers are inflammable in normal air, but they show characteristic burning phenomena in a high oxygen containing atmosphere: spread of burning adequate with standards and surface flame propagation downward or upward. Each burning phenomena is in close correlation to its oxygen index, thus their flammability can be characterized by their LOI range too.

My further goal was to find relationships between the flammability and micro and macro structure. It was confirmed that the LOI can be the degree of stability.

My research area is not considered closed, because the effect of the macro-structure of oxidized fibers on the combustion in oxygen-rich atmosphere is not yet fully explored.

7. PUBLICATIONS RELATED TO THE RESEARCH

Referred articles in English:

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2. **Kerekes Zs**. (2003): The oxygen index of oxidised fibres and influencing factors**,** Annuel News, Vol.1. pp.50-54.

3. **Kerekes Zs.**, Szakál B., Tatár A..(2003): Risk Assessment Procedures of Major Industrial Accidents, Annuel News, Vol.1. pp.42-49

4. **Kerekes Zs**..Beda L.(2005): Alterration in fire protection caracteristics of oils in the mineralwool matrix, Annuel News, Vol. I. pp. 94-97.

5. **Kerekes Zs**., Beda L. (2007): Characteristics Influencing the Limited Oxygen Index (LOI) of Carbon Fibers I, Annuel News, Vol 7(1). pp. 133-137.

6. **Kerekes Zs**., Beda L.(2012): Effect of the macro-structure on the flammability of the oxidized PAN fibre based woven textiles, TEXTIL (accepted 2012, juni)

Referred articles in Hungarian::

7. **Kerekes Zs.**: Műanyagok befolyása a szénszál alapú textíliák éghetőségére, SZIE Tudományos közlemények, I.évf., 1.sz., 28-31.o.

8. **Kerekes Zs.**, Tálos Jánosné (2006): A pyron oxidált szál a műszaki textíliák egyik fontos alapanyaga, Magyar Textíltechnika, LIX..évf., 1.sz., 9-11.o.

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