

Degradation of Polyethylene Terephthalate in Natural Conditions

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Resistance of biaxial oriented polyethylene terephthalate foil to outdoor natural weathering factors was investigated in two different climatic areas. Physical, mechanical and optical properties of polyethylene terephthalate before, during and after two years of exposure were determined. Factors affecting natural weathering of this material were described. It was concluded that this material could be used for up to 18 months in outdoor static conditions.

Key words: polymers, thermoplastics, polyethylene terephthalate, natural weathering, polymer degradation, physical properties, mechanical properties, optical properties.

Introduction

POLYMERIC materials, among which polyethylene terephthalate, are used in almost all areas of human endeavour, owing to a unique set of different characteristics. In the second half of the century, the total worldwide production of synthetic plastics and fibres has grown so much it now exceeds that of steel [1]. More than half of polymeric materials based products are used in outdoor conditions.

Results of investigation of natural weathering factors on the degradation process of polyethylene terephthalate foil in the open in two different climatic areas are described.

Degradation of polymeric materials denotes changes in physical, mechanical, optical, thermal and other characteristics caused by chemical reactions involving bond scission in the backbone and weakening of secondary intermolecular bonds. In linear polymers, among which polyethylene terephthalate, primarily, changes in the mentioned properties are caused by bond rupture in the backbone, while the chemical reactions in pendant groups and side chains have smaller influence [2].

Depending on the cause of change of the above mentioned properties, there are different modes of polymer degradation: chemical, thermal, biological, mechanical, light and radiation. Regardless of the mode of degradation involved, apart from bond scission in the backbone of polymers, there is also weakness of secondary bonds and concurrently cross-linking of chains and essential changes of initial polymer's properties.

Chemical degradation, refers, in its strict sense, exclusively to the processes which are induced under the influence of chemicals (e.g. acids, bases, solvents, reactive gases, etc.) brought into contact with polymers. However in many such cases, a significant conversion is observed only at elevated temperatures because the activation energy for these processes is high.

Thermal degradation refers to the case where the polymer, at elevated temperatures, starts to undergo chemical changes without the simultaneous involvement of

other factors. Often, it is rather difficult to distinguish between thermal and thermo-chemical degradation. Polymeric materials are only rarely chemically "pure". Impurities or present additives might react with the polymeric matrix, if the temperature is high enough.

Biologically induced degradation is also strongly related to chemical degradation as far as microbial attack is concerned. Micro-organisms produce a great variety of enzymes which are capable of reacting with natural and synthetic polymers. Enzymes of micro organisms attack polymers over a rather wide range of temperatures, but it is considered that significant changes in materials appear in the range from 60°C to 70°C.

Mechanical initiated degradation generally refers to macroscopic effects brought about under the influence of shear forces. It should be pointed out that stress-induced processes in polymeric materials are frequently accompanied by bond ruptures in the polymer main-chains.

Light-induced polymer degradation or photodegradation, concerns the physical and chemical changes caused by irradiation of polymers with ultraviolet or visible light. In order for the photodegradation to occur, light must be absorbed by the material. Thus, existence of chromophoric (light absorbing) groups in macromolecules (or in additives) is a prerequisite for the initiation of photochemical reactions. Photodegradation is of great importance due to the fact that different polymeric materials absorb ultraviolet portions inducing chemical processes, which in their turn lead to dramatic changes of physical, mechanical and other characteristics.

Radiation degradation entails high energy radiation such as electromagnetic radiation (*X*-rays, γ -rays) or particle radiation (α -rays, fast electrons, neutrons, nuclear fission products). Unlike the light degradation, the existence of chromophoric groups is not a prerequisite, since all parts of molecule are capable of interacting with radiation. The extent and character of chemical and physical changes depend on the chemical composition of the irradiated material and on the nature of the radiation.

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Practically, it is very rarely that only one mode of degradation causes changes in material properties. Among different kinds of degradation there are often strong interrelations. Depending of circumstances, different modes of degradation occur.

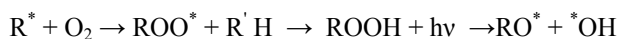
Climate represents a group of atmospheric states in an area over a specified time. Changeable composition of atmosphere, different states in air and phenomena happening in it influence, it is stated, all living things and objects. Meteorological elements are: temperature, moisture, air pressure, illumination, cloudiness, falls, wind, visibility, ground evaporation, sun light properties, etc. Main weather components i.e. main meteorological factors, which can cause degradation of materials in the open, are sunlight, temperature, water, wind and pollutants [3].

Sun is the main energetic source in nature. The original solar spectrum is modified while passing through the atmosphere so the composition of radiation reaching the Earth's surface consists of ultraviolet (about 5 % of overall radiation), visible (45 %) and infrared light (50 %). Infrared region causes molecular vibrations and causes heating of materials without chemical damage. There is a wide region of the spectra from ultraviolet through visible to infrared, which can excite electrons in molecules, causing weakening of bonds, but without chemical damage. The ultraviolet radiation has enough energy and is capable of breaking chemical bonds. When material absorbs an appropriate quantity of this energy, the scission of bonds in the polymer main-chains occurs [4].

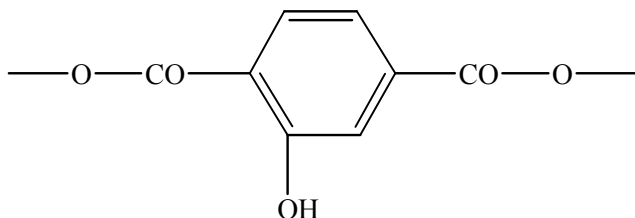
Solar radiation is an important factor causing degradation of polyethylene terephthalate. The influence of ultraviolet radiation on the characteristics of polyethylene terephthalate is quite clear: occurring of yellow surfaces, loss of elasticity, cracks on the surface and material breaks easily. Radiation from 300 nm to 380 nm causes deterioration of materials and damages are mainly concentrated on the surface [4].

When a material is simultaneously exposed to sunlight and oxygen, scissions of bonds in the backbone of polymers is predominant and change of colour also occurs.

Photodegradation process involves peroxil radicals and hydroperoxides [4]



Monohydroxy radical, formed in this way can cause substitution on the phenylene ring and forming of monohydroxy compounds. The change of material's colour is caused by formation of the monohydroxy compound, whose structure can be



The process of backbone chain cleavage yields products with carboxy- and vinyl-end groups. Oxidation of aromatic rings produces 2-hydroxyterephthalate and 2,5-dihydroxyterephthalate. Besides these two processes, there is a possibility of chain branching via reactions with aromatic rings and aryl radicals, produced in photo-oxidation processes [5].

Determination of characteristic groups content in nonstabilized 3 mm thick outdoor exposed copolyester indicates that oxidative process has inductive character. After only 19 days of exposure, hydroxylated products appeared and their content kept rising constantly, indicating that there was no auto-acceleration in the oxidative process. It is of special interest that the examined plaque does not break after 19, but after 28 days of outdoor exposure, which corresponds the finding that oxidative process producing the OH groups appears to start after only 19 days of exposure [5].

Examination of characteristic groups content and molecular weight (M_n) of 3 mm overall thickness plaque, during and after 70 days outdoor exposure by infrared and gel permeation chromatography, indicates that changes i.e. degradation takes place only in the surface layer thickness up to 0,250 mm [5]. At the depths bigger than 0,250 mm, the material has the same characteristics as before exposure. The obtained results also, indicate that in the surface layer thickness up to 0,250 mm branching reaction takes place. A logical explanation would be that phenyl radicals, produced in the chain cleavage process, react with terephthalate rings of the neighbouring chain, producing biphenyltri carboxylic acid moieties [5].

An examination of individual influences of hydrolytic, oxidative and thermal degradations of polyethylene terephthalate properties revealed that the hydrolytic process takes place much faster than the other two [4]. It is possible to cause excitation of the carbonyl chromophore group, which can then react with water in the photohydrolytic process. Excited groups can take part in processes which lead to coloured species formation i.e. yellow colour appears. If there is a small quantity of water, the predominant reaction would be the one leading to colour formation. If, however, there is a higher concentration of water, a photohydrolysis takes place and colourless products appear [6].

Recent investigations confirm that polyethylene terephthalate may be affected by ultraviolet radiation, chemical agents and hydrolysis. Absorption of ultraviolet radiation leads to oxidative reactions, but polyethylene terephthalate is more sensitive to chemical agents and especially to hydrolysis. Hydrolysis occurs only in the amorphous phase (i.e. permeable to water) and causes cut off of ester bridges, which leads to decline in both the degree of polydispersion and the average molar mass number [7].

During simultaneous action of water and high temperatures, it has been confirmed, that besides simple diffusion process, there are (a) hydrolysis of the macromolecular chains, which leads to the formation and migration of low-molecular-weight chains, (b) hydrophilisation, which tends to increase the equilibrium water intake through the development of local or overall swelling and plasticisation (or softening) of the polymer and (c) interfacial decohesion, which induces a degradation of the material [8].

Experiment

Investigations and measurements in this paper are planned so as to examine the direct influence of natural weathering factors on polyethylene terephthalate properties in continental (location Nikinci) and marine environments (location Kumbor).

Samples and experimental methods

The material is stabilized biaxial oriented polyethylene terephthalate foil, of trade name YUBORLEN, produced by polyester foil plant, Bor. Polyethylene terephthalate specimens i.e. 0,14 mm thick foil, are placed between two metal frames. Frames with foils were fixed for carrier equipment, which is 45° angle facing ground in open at two climatically different areas (Nikinci and Kumbor).

Polyethylene terephthalate specimens were exposed to natural weathering during 24 months i.e. 2 years. Based on the changes of the obtained polyethylene terephthalate properties, vs. original level, the resistance of this material in natural weathering conditions was estimated.

From polyethylene terephthalate foil, before, during and after natural weathering, specimens for determining the following properties: colour, thickness, tensile strength in extrusion direction, σ_{ml} i.e. longitudinal tensile strength (further: lon. ten. strength), tensile strength perpendicular to extrusion direction, σ_{mp} i.e. perpendicular tensile strength (further: per. ten. strength), tensile strain at break in extrusion direction, ϵ_{ml} i.e. longitudinal tensile strain at break (further: lon. ten. strain), tensile strain at break perpendicular to extrusion direction, ϵ_{mp} i.e. perpendicular tensile strain at break (further: per. ten. strain), tear resistance in extrusion direction, TR_l i.e. longitudinal tear resistance (further: lon. tear resistance), tear resistance perpendicular to extrusion direction, TR_p i.e. perpendicular tear resistance (further: per. tear resistance), flexibility at low temperature (-30°C) and integral light transparency have been cut.

Colour was determined visually. Thickness was determined by CARL FRANK thickness meter using 100 mm x 100 mm specimen and procedure described in JUS G.S2.733, method A [10]. Tensile strength and tensile strain were examined on ZWICK dynamometer at 100 mm/min crosshead speed using 200 mm long and 25 mm wide specimen with 50 mm gauge length, according to JUS G.S2.734 [11]. Tear resistance was determined on ZWICK dynamometer at 100 mm/min crosshead speed using trapezoid shape specimen and procedure described in JUS G.S2.707, method B [12]. Trapezoid shape specimen is 50 mm wide, while longer and shorter parallel sides are 120 mm and 95 mm, respectively. In the middle of the short side a 25 mm long cut was made. Flexibility at low temperature was examined on 100 mm x 10 mm specimen in VOETSCH chamber by MEHNERT method. Integral light transparency was determined on 100 mm x 100 mm specimen on spectrophotometer BECKMAN UV 5240 by optical method.

Results and discussion

Physical, mechanical and optical properties of polyethylene terephthalate foil before, during and after two years of exposure are shown in Tables 1 and 2. Table 1 contains tensile strength, tensile strains and tear resistance (in both directions) of polyethylene terephthalate foil. Table 2 contains colour, thickness, flexibility at low temperature and integral light transparency of polyethylene terephthalate foil.

Tensile strength and tensile strains of polyethylene terephthalate in both directions before exposure are arithmetic mean values for 35 specimens. Arithmetic mean values for tensile strength and tensile strain, in both directions during exposure for the specified periods of time are determined on the bases of 8 single specimens. Results

of lon. and per. tear resistance before, during and after the specified periods of exposure are arithmetic mean values for 8 single specimens. Enclosed results of colour, thickness, flexibility at low temperature and integral light transparency before, during and after the specified periods of exposure are arithmetic mean values for 3 single specimens.

Statistical analysis of tensile strength, tensile strains and tear resistance of polyethylene terephthalate foil in both directions was done. Arithmetic mean values (\bar{X}) and standard deviations (s) were calculated. Small values of standard deviations of arithmetic mean values indicate an equality of single results expressed numerically.

When determining material properties, whether results are expressed in numbers or words, it was observed that specimens have the same behaviour. Bearing in mind the mentioned fact that at 3 mm thick plaque degradation takes place only in the surface layer thickness up to 0,250 mm, results of small standard deviations and observation of behaviour of 0,14 mm thick polyethylene terephthalate foil specimens, show that the degradation process takes place throughout the volume of the investigated material.

Table 1: Tensile strength, tensile strain and tear resistance polyethylene terephthalate of foil before, during and after two years of exposure to natural weathering

Exposure time (months)	σ_{ml} (MPa)		σ_{mp} (MPa)		ϵ_{11} (%)		ϵ_{1p} (%)		TR_l (daN/cm)		TR_p (daN/cm)	
	\bar{X}	s	\bar{X}	s	\bar{X}	s	\bar{X}	s	\bar{X}	s	\bar{X}	s
0	128	16	183	9.1	154	14	97	7	375	17	380	18
Continental climate (Nikinci)												
6	59.9	5.6	88.1	3.0	4.9	0.4	4.2	0.4	196	10	256	17
12	57.1	7.8	72.8	3.9	3.8	0.8	3.2	0.8	7	0.9	9	1.0
18	41.9	4.8	64.6	2.0	2.7	0.5	2.9	0.5	5	0.4	6	0.5
24	22.3	4.8	24.9	3.5	2.8	0.7	2.3	0.5	4	0.3	4	0.3
Continental climate (Kumbor)												
6	52.6	9.8	67.9	9.1	3.0	0.7	2.9	0.4	62	9.6	105	9.3
12	51.2	7.8	61.6	9.7	3.5	0.3	2.9	0.3	7	0.8	7	0.6
18	39.6	2.4	61.1	3.5	2.8	0.4	2.5	0.3	5	0.5	5	0.4
24	20.7	4.1	21.6	5.6	2.6	0.4	2.2	0.3	4	0.3	4	0.3

Table 2: Colour, thickness, flexibility at low temperature and integral light transparency of polyethylene terephthalate foil before, during and after two years of exposure to natural weathering

Exposure time (months)	Colour	Thickness (mm)	Flexibility at low temperature (-30 °C)	Integral light transparency (%)
0	No colour	0,14	No break	89
Continental climate (Nikinci)				
6	No colour	0,14	No break	89
12	No colour	0,14	No break	89
18	No colour	0,14	No break	87
24	No colour	0,14	Break	87
Continental climate (Kumbor)				
6	No colour	0,14	No break	87
12	No colour	0,14	No break	87
18	No colour	0,14	No break	86
24	No colour	0,14	Break	86

Changes of tensile strength and tensile strain in the specified terms of natural weathering, expressed as percents of initial value before exposure, are shown in Fig.1 (Nikinci) and 2 (Kumbor).

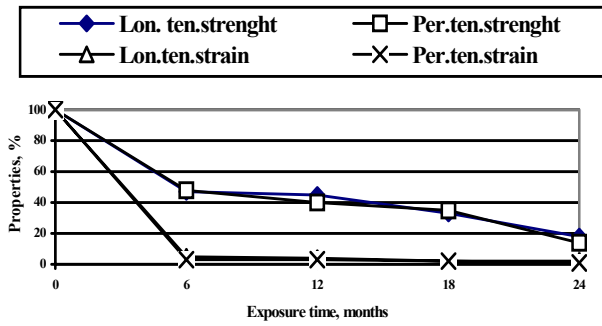


Figure 1. Changes of tensile strength and tensile strain in longitudinal and perpendicular directions in continental area (Nikinci)

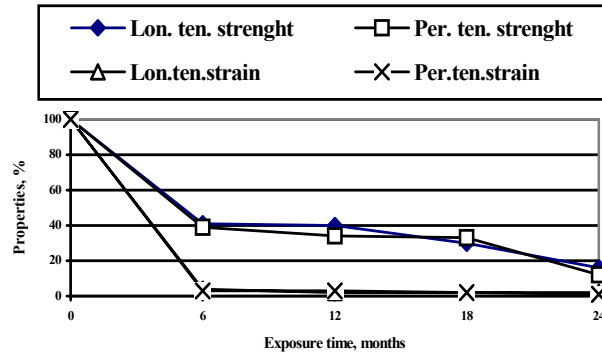


Figure 2. Changes of tensile strength and tensile strain in longitudinal and perpendicular directions in marine area (Kumbor)

Changes of tear resistance in longitudinal and perpendicular directions and integral light transparency in the specified terms of natural weathering, expressed as percents of initial values before exposure, are shown in Fig.3 (Nikinci) and Fig.4 (Kumbor).

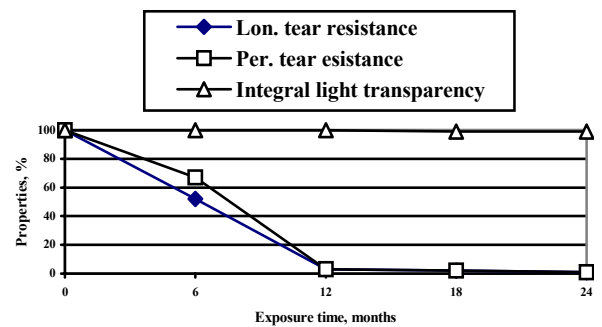


Figure 3. Changes of tear resistance in longitudinal and perpendicular directions and integral light transparency as a function of exposure time in continental area (Nikinci)

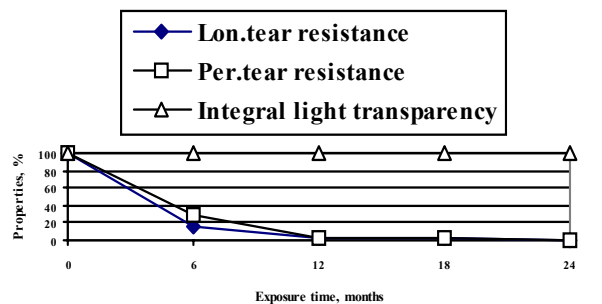


Figure 4. Changes of tear resistance in longitudinal and perpendicular directions and integral light transparency as a function of exposure time in marine area (Kumbor)

Changes of tensile strength in longitudinal and perpendicular directions vs. exposure time in continental

(Nikinci) and marine areas (Kumbor) are shown in Figures 5 and 6, respectively.

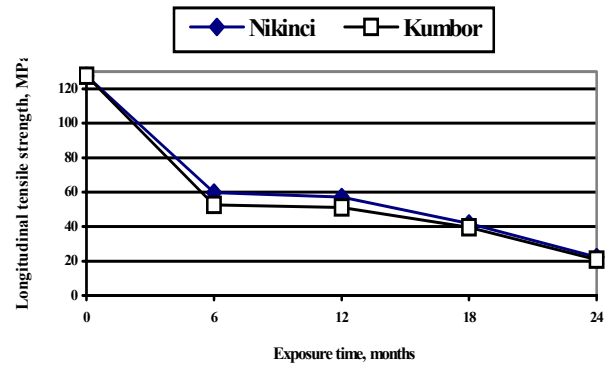


Figure 5. Changes of longitudinal tensile strength vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

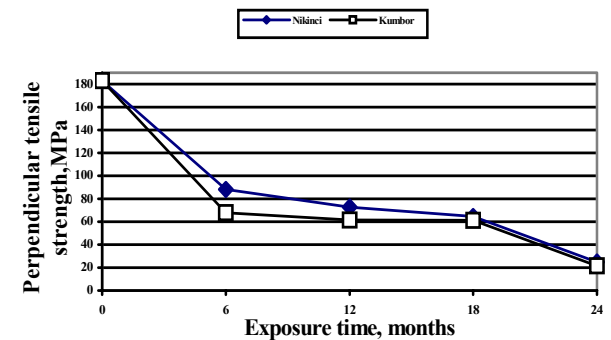


Figure 6. Changes of perpendicular tensile strength vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

Dependence of tensile strain in longitudinal and perpendicular directions vs. exposure time in continental (Nikinci) and marine areas (Kumbor) is shown in Figures 7 and 8, respectively.

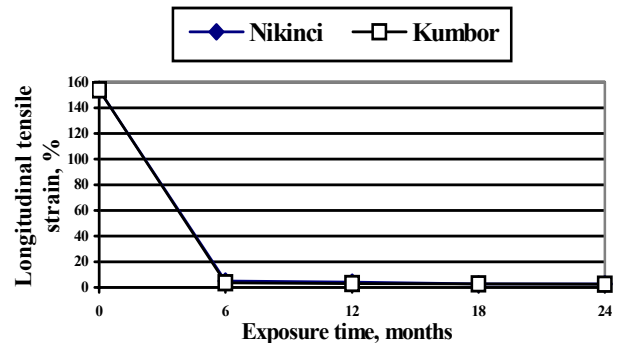


Figure 7. Changes of longitudinal tensile strain vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

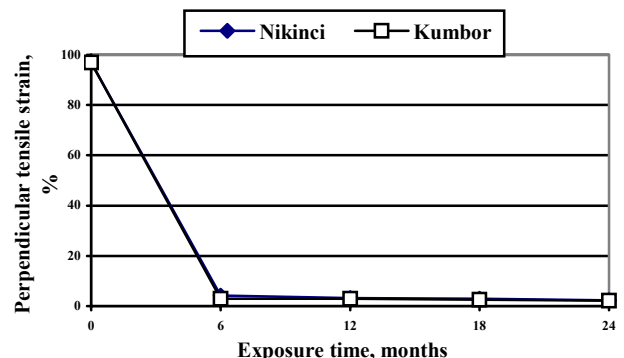


Figure 8. Changes of perpendicular tensile strain vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

Dependence of tear resistance in longitudinal and perpendicular directions vs. exposure time in continental (Nikinci) and marine areas (Kumbor) is shown in Figures 9 and 10, respectively.

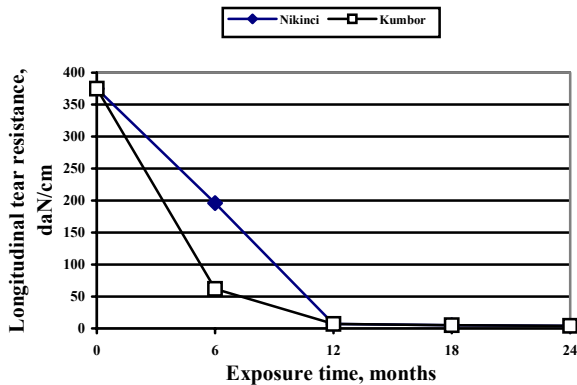


Figure 9. Changes of longitudinal tear resistance vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

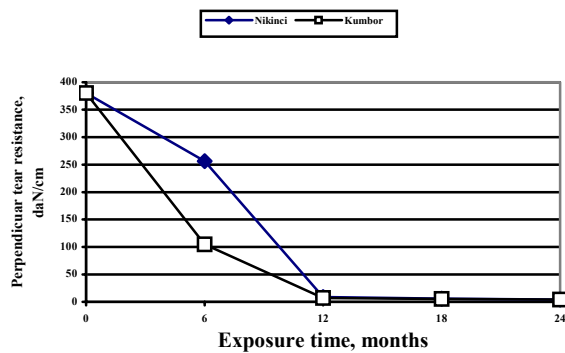


Figure 10. Changes of perpendicular tear resistance vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

Dependence of integral light transparency vs. exposure time in continental (Nikinci) and marine areas (Kumbor) is shown in Fig. 11.

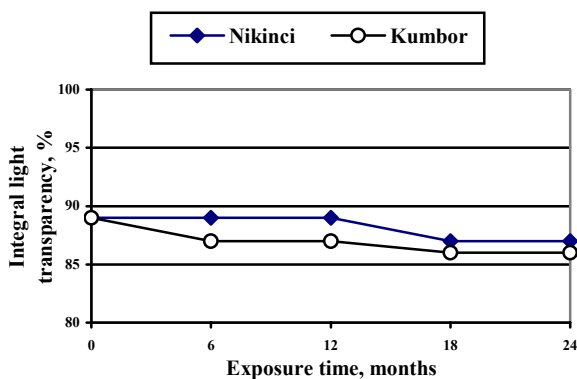


Figure 11. Changes of integral light transparency vs. exposure time in continental (Nikinci) and marine areas (Kumbor)

Results of determining physical, mechanical and optical properties of polyethylene terephthalate foil confirmed a drop of tensile strength, tensile strain and tear resistance, changes of flexibility at low temperature and also that there are no changes in colour, thickness and integral light transparency. After only 6 months of exposure, besides quantitative i.e. numerical changes of tensile strength and tensile strain values, qualitative change was observed at

tensile examination. While determining the initial values of tensile strength and tensile strain i.e. during tensile tension, the specimen elongates in a visible and gradual manner and initial width of the specimen keeps reducing, characteristic "neck" appears and specimen breaks at the end of the examination period. While determining tensile strength and strain of specimens exposed in the outdoors for six (or more) months, the described behaviour was not observed and specimens would break at a certain point over their initial width. Analyzing the data obtained through examination of polyethylene terephthalate foils before and after 6, 12, 18 and 24 months of outdoor natural weathering, it can be concluded that the degradation process started from the moment of exposure.

On specimens, which were exposed for six months on both locations, the following was observed:

- drastic drop of longitudinal and perpendicular tensile strain of 95 %,
- reduction of longitudinal and perpendicular tensile strength of 55 % to 60%,
- qualitative change of behaviour of specimens during tensile tension,
- drop of longitudinal tear resistance from 50 % (Nikinci) to 80 % (Kumbor) i.e. perpendicular tear resistance from 35 % (Nikinci) to 70 % (Kumbor),
- flexibility at -30°C was satisfactory i.e. the specimen does not break,
- there are no changes in colour, thickness and integral light transparency.

After 12 months of natural weathering, the following was observed:

- further diminution of longitudinal and perpendicular tensile strength to level 60 % (Nikinci) and 65 % (Kumbor),
 - values of longitudinal and perpendicular tensile strain were similar to those after 6 months of exposure,
 - severe drop of longitudinal and perpendicular tear resistance of 97 %,
 - there were no changes in flexibility at low temperatures (-30°C), colour, thickness and integral light transparency.
- Results of testing specimens after 18 months of exposure demonstrated:

- drop of longitudinal and perpendicular tensile strength to 65 % (Nikinci) and 70 % (Kumbor),
 - values of other properties were similar to those after 12 months of exposure.
- Period of 24 months of exposure is characterized by:
- drop of longitudinal and perpendicular tensile strength of 80 % (Nikinci) and 85 % (Kumbor),
 - qualitative change of specimen flexibility behaviour at low temperatures (-30°C) i.e. specimens broke for the first time,
 - very low values of tear resistance in both directions.
 - there were no changes in colour, thickness and integral light transparency.

The most important phenomena of gradual degradation process of polyethylene terephthalate foil during 24 months of exposure to outdoor natural weathering are:

- very large drop of tensile strain and qualitatively different behaviour during tensile tension after six months
- significant reduction of tear resistance after 12 months of exposure,
- drop of tensile strength to level of 33 % of initial values after 18 months of exposure,

- breaking of specimen for the first time during low temperature flexibility testing, after 24 months of exposure.

The observed tendency of initial properties' reduction, especially a drastic drop of tensile strain and tear resistance, qualitative change of behaviour of the specimen during tensile tension and breaking of specimen during low temperature flexibility testing indicated that the following occurred in polyethylene terephthalate foil:

- hydrolysis, which causes bond scission of the macromolecular chains,
- absorption of water in amorphous part; this phenomenon induces stress at the amorphous/crystalline interface, followed by microcavity formation [9],
- absorption of sunlight, more precisely ultraviolet part of the spectrum, which causes chemical reaction i.e. bond scission in the polymer backbone,
- interfacial decohesion, which leads to decline of mechanical characteristics.

There were no observations, which pointed out that the hydrophilisation process i.e. softening of polyethylene terephthalate, had happened.

The fact that, after 24 months of exposure, yellow colour was observed at neither specimens from Nikinci nor specimens from Kumbor, confirms that specimens were mostly in humid environment at continental and marine locations.

The above given results speak in favour of the fact that the changes of specimen properties after natural weathering at Nikinci and at Kumbor is the same. But, degradation process of polyethylene terephthalate foil is faster in marine than in continental environment, as shown in Figures 5 - 11.

Conclusion

Based on the results obtained for polyethylene terephthalate foil and their analysis, it can be concluded that:

Constant reduction of longitudinal and perpendicular tensile strength, tensile strain and tear resistance actually starts from the moment of exposure to the outdoor conditions at continental (Nikinci) and marine (Kumbor) environments.

There are no changes, regarding the initial quality in colour, thickness and integral light transparency, after 24 months of exposure.

The trend of decreasing i.e. retaining the mentioned properties is observed during the whole period of weathering, in all but flexibility; after 24 months of exposure, specimens break, for the first time, when testing flexibility at low temperatures (-30°C).

The drop of tensile strength of about 80 %, of tensile strain and tear resistance of about 95 %, compared to the initial quality, and breaking of specimens for the first time during low temperature flexibility testing, proved that there is no rational point in exploring natural weathering process in the open after 24 months of exposure.

A severe drop of tensile strain after only 6 months of exposure indicates that this material can not be used outdoor in dynamic conditions.

The obtained results of testing confirmed that biaxial oriented polyethylene terephthalate foil can be used outdoors in static conditions up to 18 months.

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Degradacija polietilentereftalata u prirodnim uslovima

Ispitivana je otpornost biaksijalno orijentisane folije polietilentereftalata prema dejstvu faktora prirodnog starenja na otvorenom prostoru u dva različita klimatska područja. Određivana su fizička, mehanička i optička svojstva polietilentereftalne folije pre, tokom i posle dvogodišnjeg izlaganja. Opisani su faktori koji utiču na prirodno starenje ovog materijala.

Zaključeno je da se ovaj materijal može koristiti do 18 meseci na otvorenom prostoru u uslovima statičkog opterećenja.

Ključne reči: polimeri, termoplasti, polietilentereftalat, prirodno starenje, degradacija polimera, fizičke osobine, mehaničke karakteristike, optičke karakteristike.

Дегградация полиэтилентерепфталата в натуральных условиях

В настоящей работе испытывана прочность двоаксиально ориентированной фольги полиэтилентерепфталата к влиянию фактора естественного старения на открытом пространстве в двух различных климатических областях. Здесь определялись физические, механические и оптические характеристики фольги полиэтилентерепфталата перед, в течении и после двухлетнего внешнего воздействия. Здесь тоже описаны и факторы влияющие на естественное старение этого материала. В конце сделан вывод, что этим материалом возможно пользоваться до 18 месяцев на открытом пространстве в условиях статической нагрузки.

Ключевые слова: полимеры, термопласты, полиэтилентерепфталат, естественное старение, дегградация полимера, физические свойства, механические характеристики, оптические характеристики.

Dégradation du polyéthylène téréphtalate dans les conditions naturelles

On a examiné la résistance d'une feuille en polyéthylène téréphtalate biaxialement orientée contre l'action du facteur de vieillissement naturel en plein air dans deux différentes régions climatiques. On a déterminé les propriétés physiques, mécaniques et optiques de la feuille en polyéthylène téréphtalate avant, au cours et après l'exposition de deux ans. Les facteurs qui influencent au vieillissement naturel de ce matériel ont été décrit dans cet article. On a conclu que ce matériel peut être utilisé jusqu'à 18 mois en plein air dans les conditions de charge statique.

Mots clés: polymères, thermoplastiques, polyéthylène téréphtalate, vieillissement naturel, dégradation des polymères, propriétés physiques, caractéristiques mécaniques, caractéristiques optiques