

Investigation of Natural Weathering of Medium and High Molecular Weight Polycarbonate at Continental and Marine Location for Period of 96 Months

Jovan Radulović¹⁾

Two thermoplastic polycarbonate bisphenol A types were exposed to the influence of natural weathering at continental location and at marine location for a period of 8 years (96 months). Every six months the specimens of medium molecular weight polycarbonate and high molecular weight polycarbonate were taken from mentioned locations and tested. Seven characteristics were chosen for determination of quality before exposition and during the process of degradation of two mentioned polycarbonate types (tensile strength, elongation to break, Shore hardness, Vicat softening point, water absorption, density and Charpy impact resistance). Changes of these seven properties at continental location are similar to the changes of mentioned characteristics at marine location during specified period of time. Data obtained by testing specimens of medium molecular weight polycarbonate pointed out that there were two important changes of quality regarding elongation to break and Charpy impact resistance (first after 36 months and second after 54 months of exposure). After 96 months of natural weathering of the above-mentioned material at both locations meaningful drop of tensile strength, moderate change of water absorption and negligible changes of Shore hardness, Vicat softening point and density were recorded. The influence of molecular weight is very apparent because high molecular weight polycarbonate exhibited higher resistance to the degradation process in natural conditions. Two very important characteristics of this polycarbonate material (tensile strength and Charpy impact resistance) practically did not change during 96 months of exposition in an open air, while drop of elongation to break is meaningful, diminution of water absorption is moderate and changes of Shore hardness, Vicat softening point and density are very small.

Key words: Thermoplastic polycarbonate, degradation, natural conditions, tensile strength, elongation to break, Shore hardness, Vicat softening point, water absorption, density, Charpy impact resistance, natural weathering influence, continental location, marine location, molecular weight mass influence, results of investigations, tables, diagrams.

Introduction

POLYMERIC materials are one of four basic engineering materials (other three are metals, ceramics and composites). Plastic materials and elastomeric materials are two main groups of polymeric materials and the first mentioned material can be divided into thermoplastic and thermosetting ones. There are few hundreds of thermoplastic materials and each of them possesses unique properties which determine their usage. Thermoplastic engineering materials are used for production of parts which are exposed to a high level of stress in practice and very often they replace metals in load bearing applications. Polycarbonates, beside polyamides, polyacetals, polyphenylene oxides, polysulfones and the thermoplastic polyesters, are thermoplastic engineering polymers [1].

People associated with polymeric materials are sure that the second half of the twentieth century, first two and future decades of ongoing century are the age of a new class of materials. Owing to a set of specific characteristics, these rapidly developed and presented materials are widening the areas of usage.

Generally speaking, no matter where these materials are used (inside or outside), degradation (or deterioration) of product depends on the nature and time of interactions with surroundings. Although there are no precision data where the polymeric material products are used, one real estimation is that about one half of produced parts are used outdoors.

The main problem of polymeric parts used in outdoor environment is ageing or weathering. Sunlight, temperature, moisture, wind, dust and pollutants are the most important components of weathering, which cause degradation of polymeric material products.

Maybe the most important property of polymeric materials used in external environment is photostability. This characteristic is influenced by impurities (catalyst residues and trace metals) and structural irregularities of polymers. There are few methods of improving stability, i.e. in eliminating or reducing mentioned entities.

The first method is a modification of polymeric structure by changing the fabrication process. This procedure is rarely used because it is highly expensive and can change properties which are important for practical conditions.

In outdoor environment, photostability of polymers is disrupted when this material absorbs a photon, which produced activated species, followed by photophysical and chemical conversions of the mentioned species. *The second* method of improving a photostability is adding an absorber or a reflector to the polymer. This treatment is useful when polymer is irradiated but the quantity of received energy is not enough to initiate damaging chemical processes. *The third* method is used when polymer absorbed enough quantity of radiation and activated species are formed. This method consists of employing additives whose role is to quench the

¹⁾ Jovan Radulović, 11000 Belgrade, SERBIA
Correspondence to: Jovan Radulović, e-mail: jovan.r.radulovic@gmail.com

excited chromophores and, after that, to dissipate the absorbed energy in harmless way. Another (*fourth*) method in improving the photostability is to hinder and subdue the chemical reactants, which make a degradation process. In this step a transformation of nonstable intermediates (for example hydroperoxide) into stable compounds and prevention of their photodecomposition and formation of radicals occurred. But if the radicals are even formed the use of scavengers can reduce the degradation process [2].

This paper is dealing with polycarbonate exposed to the degradation process in natural environment.

Polycarbonate

Thermoplastic and thermosetting polycarbonate

Polycarbonate was discovered at the end of the nineteenth century (precisely in 1898) when Alfred Einhorn from the University of Munich was trying to obtain cyclic carbonates using hydroquinone with phosgene. He observed the formation of an insoluble, infusible solid, but after 30 years' laboratory research, this class of materials was abandoned without commercialization [3, 4].

Few years after A. Einhorn (precisely in 1902) two chemists (Bischoff and Hedenström) obtained similar cross-linked, high-molecular-weight polycarbonate. Famous W.H. Carothers extended the work on the product but it remained at the laboratory scale.

Intensive research of this material, done at the Bayer company in Uerdingen, ended in 1953 when Hermann Schnell patented the first linear polycarbonate.

Daniel Fox from the General Electric, New York, independently synthesized a branched polycarbonate in the same year (1953) but only one week after the invention at Bayer.

Bayer, Germany, has produced polycarbonate under a trade name "Makrolon" since 1958 and General Electric, USA, has started with fabrication of their polycarbonate (brand name "Lexan") in 1960 [3-8].

Polycarbonate (chemical formula $C_{15}H_{16}O_2$) got its name from the carbonate groups in its backbone chain and a basic unit of this macromolecule is shown in Fig.1 [9, 10].

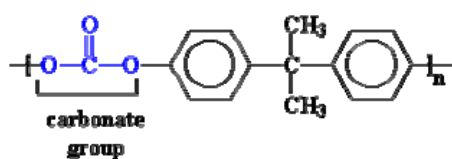


Figure 1. Basic unit of polycarbonate [10]

Precisely speaking, the main polycarbonate material is named polycarbonate of bisphenol A because two main compounds in synthesizing are bisphenol A and phosgene.

In the first reaction, bisphenol A reacts with sodium hydroxide and the sodium salt of bisphenol A is obtained [10]:

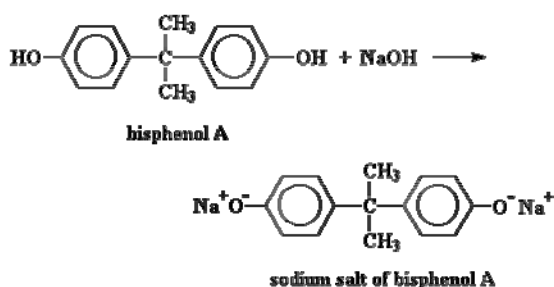


Figure 2. Reaction of bisphenol A with sodium hydroxide and producing of sodium salt of bisphenol A [10]

In the second reaction, sodium salt of bisphenol A reacts with phosgene to produce the polycarbonate [10]:

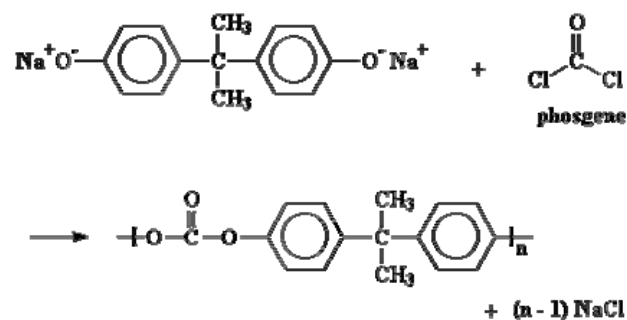


Figure 3. Reaction of sodium salt of bisphenol A with phosgene and producing of polycarbonate of bisphenol A [10]

Speaking about polycarbonate, it is important to say that, beside thermoplastic polycarbonate of bisphenol A, there is a thermoset polycarbonate. This material is produced starting from the monomer presented in Fig.4 [10]:

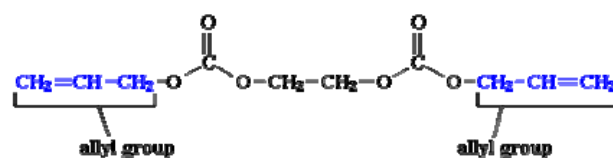


Figure 4. Monomer-starting compound for producing the thermoset polycarbonate [10]

On the ends of this monomer, there are two allyl groups which have carbon-carbon double bonds in them. This enables them to polymerize by free radical vinyl polymerization. One allyl group at one end of the monomer becomes a part of one chain and the second allyl group at the other end of the monomer becomes a part of the second chain. In this reaction, all the chains will become tied together to form a crosslinked material, which is shown in Fig.5 [10]:

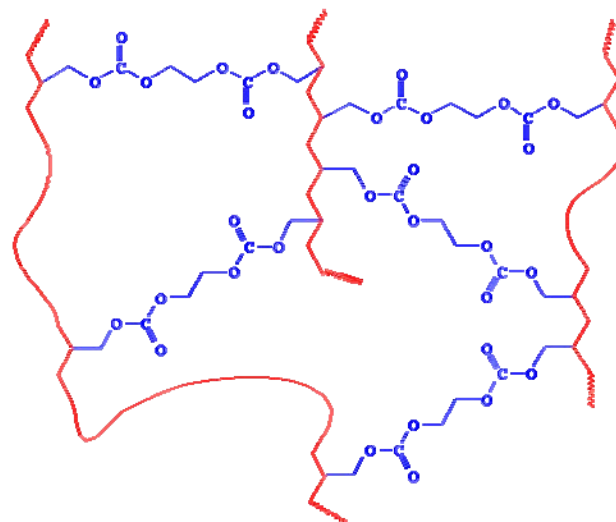


Figure 5. Cross linked polycarbonate [10]

As it can be seen in Fig.5, the carbonate-containing groups (shown in blue) crosslink between the polymer chains (shown in red). This crosslinking is making the material very strong with high refractive index. Based on this property, this kind of polycarbonate is used almost exclusively in optical industry for making ultra-light eyeglass lenses. Thermosets do not melt, and they cannot be remolded.

Based on the molecular arrangement, the thermoplastic polymers can be divided into two types: amorphous polymer and crystalline polymer.

Molecular structure of amorphous polymers has no organized pattern, whereas the crystalline polymers have an ordered molecular structure, as shown in Fig.6 [11]:

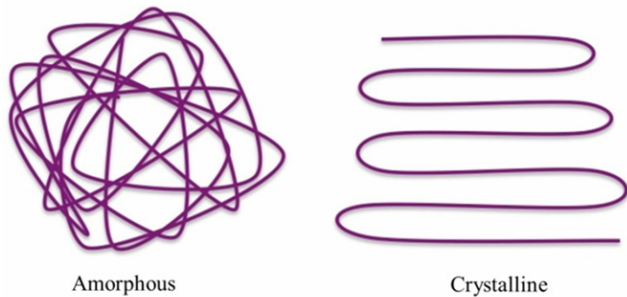


Figure 6. Molecular structure of amorphous and crystalline polymers [11]

Polycarbonate of bisphenol A is one of the most commonly used amorphous polymers in engineering. In amorphous structure of thermoplastic polycarbonate, molecular chains are associated with intermolecular attractive forces. When the temperature of polycarbonate increases, mentioned forces weaken rapidly and the solid material turns into a viscous liquid. Thus, polycarbonate of bisphenol A can be reshaped

by heating it and it can be processed by various processing techniques (injection molding, compression molding, calendaring and extrusion) [12, 13].

Degradation of polycarbonate

Photo-oxidation and photo-Fries mechanism are two main reactions in photodegradation of polycarbonate of bisphenol A. The photo-Fries mechanism is initiated with wavelengths under 310 nm. Photo-oxidation reactions take place if the wavelengths are in the 290 nm – 350 nm range, but only in the presence of oxygen [14].

The first reaction in the photo-Fries mechanism is a scission of the carbonate linkage in polycarbonate and two primary free radicals are formed [15]. Rearrangement according to the Fries type and further crosslinking of the free radicals results in the formation of different compounds (phenyl salicylates, dihydroxybenzophenones, dihydroxybiphenyl and hydroxydiphenyl ether). In the further "rearrangement" reactions of the mentioned compounds are formed substances which absorb in the blue range (ortho-dihydroxybenzophenone or diphenoquinone,), and this is the reason for the yellowing observed in the photodegraded samples [16-18].

The main reactions in the photo-Fries mechanism are presented in Fig.7 [15]:

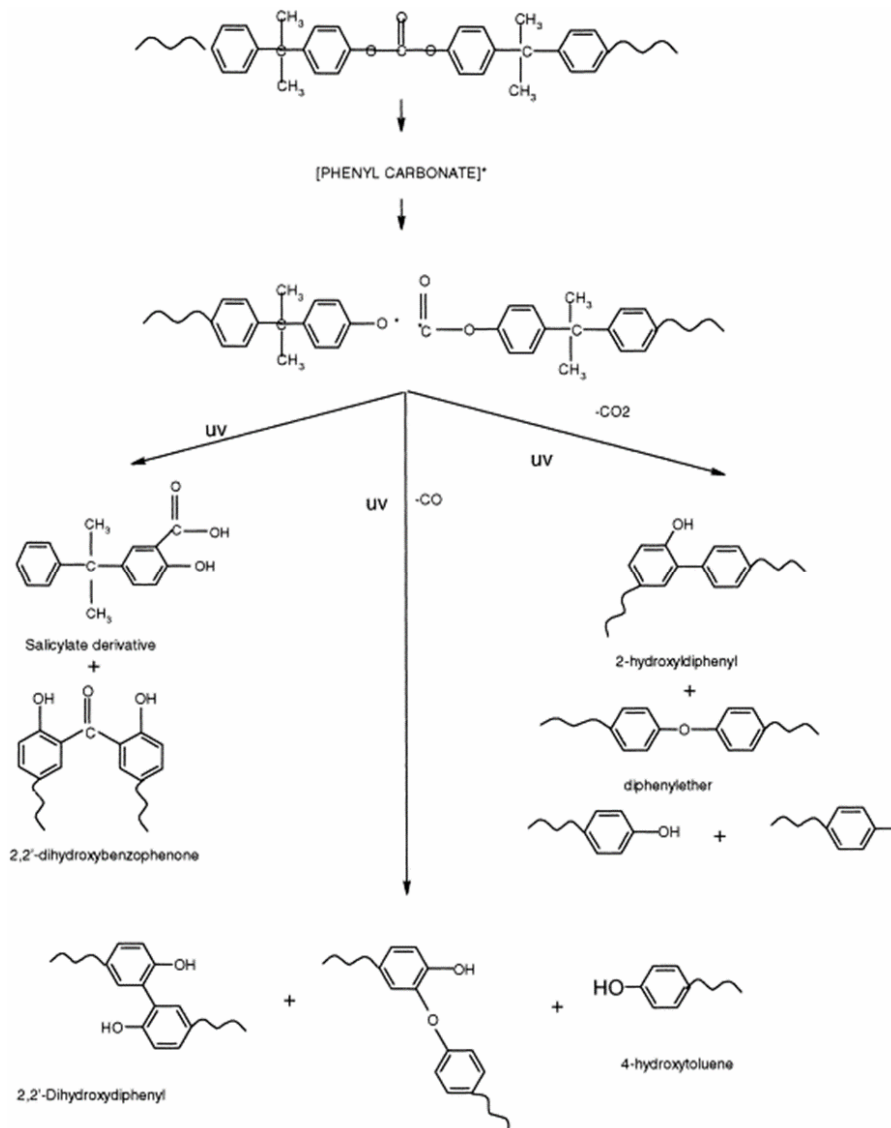


Figure 7. Main reactions in the photo-Fries mechanism [15]

The photo-oxidation mechanism is a complex one and it is realized through three reactions: side chain oxidation, ring oxidation and ring attack. The side chain attack is best known route. Polycarbonate polymer absorbs UV radiation, and, as a consequence of the energy excitation, hydrogen atoms are abstracted from the methyl groups. In this way a free radical is formed, which abstracts hydrogen from neighboring molecules yielding new free radicals. The methyl side chains are photo-oxidized into hydroperoxide intermediates, and the reaction can either propagate or terminate yielding tertiary

alcohols and ketones, but only in the presence of oxygen. The free radical reaction is self-propagating but it stops if there are no more active sites or oxygen is not present [19].

Polyconjugated species are the products of photo-oxidation reaction of the aromatic rings in the presence of oxygen (in conditions of artificial and natural weathering) and are considered the contribution to the yellowing of polycarbonates [20].

The main reactions in the photo-oxidation mechanism are presented in Fig.8 [19]:

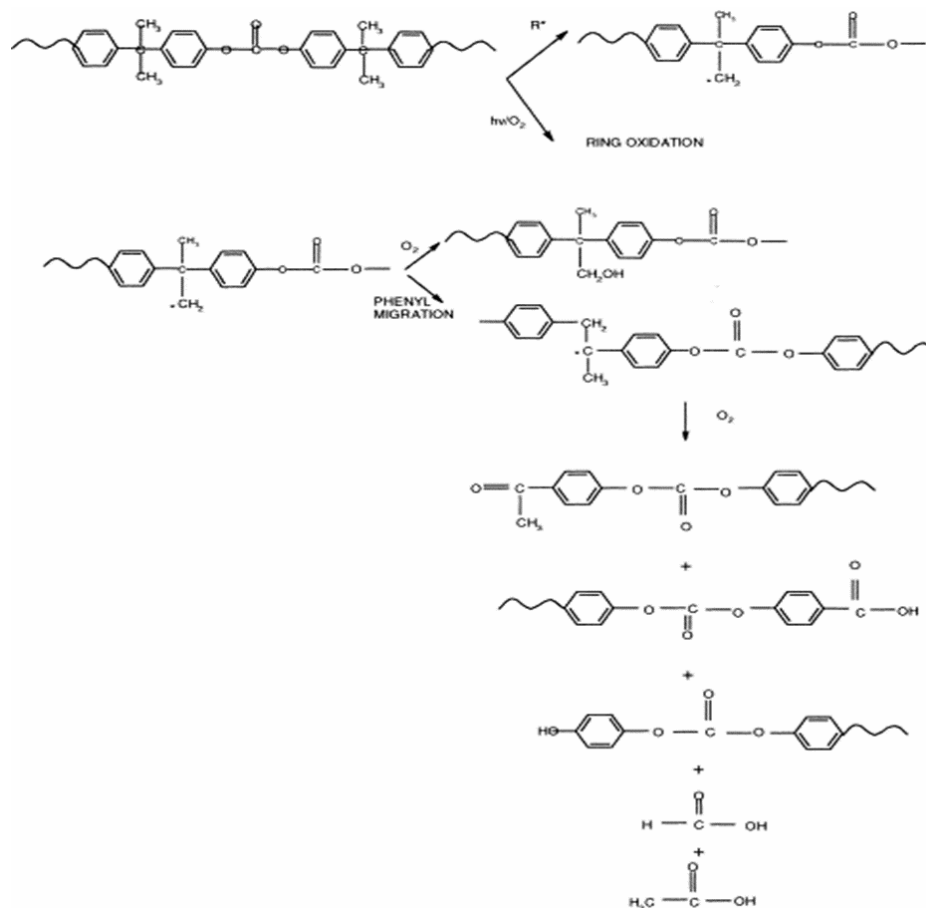


Figure 8. The main reactions in the photo-oxidation mechanism [19]

The planned and realized investigations of two polycarbonate bisphenol A types, described in this paper, have two goals:

- the first is to establish the influence of natural weathering in outdoor conditions at continental location and at marine location for a period of 96 months and
- the second is to determine the influence of molecular weight on changes of the characteristics of two described polycarbonates types during exposition in an open air for the mentioned period of time at both locations.

In this paper a process of natural weathering of two polycarbonate bisphenol A types at continental location and at marine location for a period of 8 years (96 months) is described. Tensile strength, elongation to break, Shore hardness, Vicat softening point, water absorption, density and Charpy impact resistance of high molecular weight polycarbonate and medium molecular weight polycarbonate were tested before and during the mentioned process of expositions at both locations. The influence of natural weathering time for both investigated polycarbonate types is established. Obtained results pointed out that the changes of mentioned characteristics of both polycarbonate types are similar at continental location and at marine location. The

influence of molecular weight is evident because changes of the above-described seven characteristics of high molecular weight polycarbonate are meaningfully smaller than changes of described seven properties of medium molecular weight polycarbonate after the same conditions of overall exposition (time and locations).

S. Colin and others studied the influence of light irradiation on characteristics of polycarbonate specimen, including mechanical properties, molecular weight and structural changes. Changes in molecular weight were determined using contemporary equipment (AFM nanoscale thermal analyses). Some chemical changes, that reflect reactions in the macromolecular structure, were determined by infrared spectroscopy. The absorption process was studied by UV-Visible microspectroscopy and degradation changes by infrared microspectroscopy. They proposed a new reaction pathway (that reflects the cross-linking reaction route) to the existing mechanism of the photo-oxidation process [21].

Gao Weibin et al. investigated the effects of hydrothermal aging on the properties and structure of Bisphenol A polycarbonate. Mechanical testing showed that there is an elongation at break of tested samples before the influence of mentioned aging and no elongation at break after exposure.

Photographs of the fracture surfaces after hydrothermal effect, obtained by scanning electron microscopy, showed that there are many fragments which are linked to the fracture surface and that there is no the length of deformation at break. Based on the obtained data, they concluded that bending strength of Bisphenol A polycarbonate is increased after aging. The glass transition temperature (T_g) of the tested samples, determined by differential scanning calorimetry analysis, increases after hydrothermal treatment. Analyses of Bisphenol A polycarbonate samples by the Fourier transform infrared spectroscopy shows that hydrolytic degradation happened after hydrothermal aging [22].

Tjandraatmadja G. F. and others investigated the effects of humidity in photodegradation process of bisphenol A polycarbonate in laboratory conditions. This thermoplastic material was tested in QUV Weathering Accelerated Tester for a period of up to 3000 h while the range of relative humidity was from 0 % to 42 %. They identified that photo-Fries mechanism happened at the beginning and photo-oxidation degradation happened in later period of exposure time. Their first conclusion was that humidity alone has no damaging effect on the properties of the tested material at the ambient temperature. Combination of UV radiation and humidity has negative effects on optical characteristics. When the level of humidity is low, degradation of optical properties and reduction of the molecular weight of the polycarbonate are faster. At a lower humidity, the degradation reaction is slow and UV radiation penetrates deeper into the polymer. The consequence of this reaction is that the thickness of layer of degraded material is significantly higher. When the level of humidity is higher, a thin photo-oxidized layer is formed very fast and protects the remaining polymer from negative effects of UV rays. The rugged surface of polycarbonate and degraded layers induce the scattering of light on the polymer surface, which causes the reduction of gloss and increase of haze of the material. The increase in haze, in combination with two other degradation effects (yellowing causing the absorption of visible light and the formation of light-scattering agents at different thickness inside the sample), decreases the total amount of light transmitted through the polymer. The final conclusion is that coupled influence of humidity and UV radiation has negative effects on the optical properties of polycarbonate (reduced light transmission, loss of gloss, increased haze and discoloration) [14].

The time of useful functioning of polycarbonate parts changes with the different service environments. Dequan Wu and others constructed a back propagation artificial neural networks (BP-ANNs) model in order to estimate the degradation of polycarbonate in atmospheric environment. The model is based on datasets obtained after the exposure tests in 13 chosen places in China for a long period of time. The verification of mentioned BP-ANNs model has been done with datasets obtained in atmospheric weathering conditions in additional three locations. Using environment parameters of 804 cites in China, the model was used to predict the degradation of polycarbonate. It is concluded that the highest degradation changes of polycarbonate are in the tropical monsoon climate area and plateau climate area. Another statement is that slight degradation of polycarbonate is in the temperate monsoon climate area in the northeastern, and subtropical monsoon climate area in the southwestern basin of China. This developed model will contribute to the fast design, choosing and estimation of polycarbonate parts, predicting to be used in atmospheric service environments [23].

In order to establish how the mechanical properties of few polymers changed with outdoor weathering conditions, Kim Sunwoo and others, at the beginning, realized four-year

lasting investigation. They exposed samples of four thermoplastic materials (polycarbonate, polypropylene, polybutylene terephthalate and high-density polyethylene) to the total UV irradiation of 1020 MJ/m^2 at 315 nm-400 nm wavelength range. After every 60 MJ/m^2 interval, they tested five specimens of each material and determined tensile characteristics (tensile modulus, tensile-yield strength, yield strain and fracture strain). The first three mentioned mechanical characteristics did not change significantly with the exposure time for all four polymers, but the fracture strain underwent a significant reduction (the % fracture strain retention was 17 % for polycarbonate, 20 % for high-density polyethylene, 27 % for polybutylene terephthalate and 47 % for polypropylene). Therefore, this property was chosen for the further analysis and a fracture strain retention function was developed using logistic regression analysis for each polymer. This function was then applied to the true stress-strain curve and the level of degraded mechanical characteristics for weathering was determined by FE analysis. Double chamber channel, made of injection-grade polycarbonate, exposed to specific conditions (impact loading at intermittent UV radiation) was an example for FE analysis. Obtained data pointed out that UV exposure has influence to the fracture initiation and propagation energies upon the impact loading. They concluded that the fracture initiation energy was reduced up to 90 % when the fracture strain retention was 24 %. Another observation was that mentioned initiation parameter was more affected than the propagation energy by about 20 % [24].

Redjala S. and others investigated the influence of UV radiation on the properties of polycarbonate. They irradiated this thermoplastic polymer with a wavelength range from 200 nm to 280 nm, at different times. Using optical microscopy and UV-visible spectrophotometry, they observed a decreasing of transparency and occurrence of yellow color. Measurement of microhardness through the thickness proved a gradual deterioration in the depth of the specimen. Uniaxial tension tests were performed on the irradiated and non-irradiated specimens and lowering of this property was observed. FTIR analyses revealed the breaking of vulnerable bonds and the consequence of this process is a rearrangement of the molecular chains and a change in the size of the crystallites. The mentioned phenomenon is suggested by XRD investigations. The main conclusion of this paper is that UV radiations cause breaking of vulnerable chemical bonds, which allow a new rearrangement of the molecular chain's network. This process causes a decrease in tested physical and mechanical properties and change of optical characteristics of polycarbonate [25].

Gandhi K. and others exposed bisphenol A polycarbonate to blue light emitting diodes (LED) using irradiation of 360 nm as a tool for yellowing. A linear Arrhenius plots with activation energies of 30 kJ/mol – 48 kJ/mol was determined as an effect of temperature in the range from $90 \text{ }^\circ\text{C}$ to $120 \text{ }^\circ\text{C}$. The presence of stabilizers has influence on obtained results. Beside the mentioned experiment with LED blue light, an experiment with other source of light was done (xenon arc irradiation equipped by UV filters). Based on both experiments it was concluded that the occurrence of yellow color in specimens is proportional to the sample thickness up to at least 6 mm. The main conclusion is that the activation energies and thickness dependence for specimens tested in laboratory apparatus are very different from these parameters regarding specimens in external weathering tests [26].

Picket E.J. et al. wanted to establish the influence of some parameters (UV irradiance, UV wavelength, temperature and

relative humidity) on specific characteristics (yellowing and gloss loss) of four thermoplastic materials. They exposed specimens of polycarbonate (PC), poly(butylene terephthalate) (PBT), a PC/PBT blend, and poly(styrene-co-acrylonitrile) (SAN), all containing 3% TiO₂ (by mass) to the Simulated Photodegradation via High Energy Radiant Exposure. There was no effect of irradiance, but activation energy for yellowing for PBT was 16 kJ/mol while this parameter for other three tested materials (PC, PC/PBT blend, and SAN) is almost the same (about 20 kJ/mol). Gloss loss process has the activation energy in range 9 kJ/mol – 16 kJ/mol. They estimated that if the temperature is increased for 10 °C the increase in degradation rate is about a 20 % – 30 %. Relative humidity has no consistent influence on yellowing or gloss loss of polycarbonate or PC/PBT blend. Relative humidity greater than 10 % has little effect on SAN, which degraded in dry conditions. In dry conditions, PBT gloss loss process is slow and relative humidity has no influence on yellowing of this polymer. Blend PC/PBT is more sensitive to shorter UV wavelength than PC or PBT [27].

Experimental part

Two types of thermoplastic polycarbonate bisphenol A trade name Makrolon, produced by Bayer company, Germany, are chosen for investigation in natural environment.

The first type is *medium viscosity polycarbonate*, UV stabilized, unreinforced, flame-retardant injection molding grade (in further text: Makrolon 2803).

The second type is *high viscosity polycarbonate*, UV stabilized, unreinforced, flame-retardant injection molding grade (in further text: Makrolon 3203).

The viscosity of polymer solutions in organic solvents gives a value that is directly related to the molecular mass of the polymer. The molecular mass or molecular weight of polymer is related to its molecular size, which means that polymers with a larger molecular size have a higher molecular weight. In other words, the higher the viscosity, the higher the molecular weight.

Having those mentioned facts in mind, it can be stated that Makrolon 2803 is a polymer with medium molecular weight and Makrolon 3203 is a thermoplastic material with high molecular weight [28].

Makrolon 2803 and Makrolon 3203 specimens are produced by injection molding technology.

In order to establish the resistance of polycarbonate bisphenol A Makrolon 2803 and Makrolon 3203 to a direct influence of external conditions in open, the specimens of two mentioned polycarbonate bisphenol A types were placed in two different climatic areas, i.e. in continental location and marine location. The specimens of Makrolon 2803 and specimens of Makrolon 3203 were exposed to the outdoor natural weathering at the mentioned locations during 8 years, i.e. 96 months.

The specimens of two mentioned polycarbonate bisphenol A types, exposed to the outdoor natural weathering at the mentioned locations, were mounted on the carrier equipment.

Mentioned equipment is at an angle of 45° facing the Equator for two reasons: one is that this angle is an optimum angle of exposure in temperate latitude for the whole year and the other is based on the statement that the angle of exposure of specimens to natural weathering factors should be equal to the latitude of the site where specimens are exposed (continental location 44°51' N and marine location 42°26' N) [1].

The specimens of Makrolon 2803 and Makrolon 3203 were taken from mentioned carrier equipment every six months and tested.

Results and analysis

Tensile strength, elongation to break, Charpy impact resistance, Shore hardness, Vicat softening temperature, density and water absorption are properties chosen for determining the initial quality and following the degradation of the specimens of Makrolon 2803 and Makrolon 3203. SRPS EN ISO 527-2:2013 standard is used for determination of tensile strength and elongation to break [29]. Impact resistance without notch is determined in compliance with SRPS EN ISO 179-1:2011 [30]. Hardness is tested according to SRPS EN ISO 868:2015 [31]. SRPS EN ISO 306:2015 standard is used for testing the Vicat softening temperature [32]. Density is tested by immersion method according to SRPS EN ISO 1183-1:2019 [33]. Water absorption for seven days is determined in a line with SRPS EN ISO 62:2012 [34].

Initial value of tensile strength, value of elongation to break and value of Charpy impact resistance were based on fourteen single specimens. For the determination of Shore hardness, Vicat softening temperature, density and water absorption before weathering in outdoor environment seven single specimens were used.

Seven single specimens were tested for obtaining a value of tensile strength, value of elongation to break and value of Charpy impact resistance, after the specified periods of exposure. Three single specimens were used for the determination of Shore hardness, Vicat softening temperature, density and water absorption during mentioned periods of investigation in external conditions.

Statistical analysis of tensile strength, elongation to break and Charpy impact resistance of Makrolon 2803 and Makrolon 3203 before and during natural weathering at both locations has been done and arithmetic mean values and standard deviation have been calculated.

Table 1 contains the results obtained when the initial quality of Makrolon 2803 was determined (tensile strength, elongation to break, Charpy impact resistance, Shore hardness, Vicat softening temperature, density and water absorption) and the results which represent changes of chosen characteristics of mentioned polycarbonate bisphenol A type during the exposure to weathering in natural conditions at continental location and at marine location for a period of 96 months.

Table 1. Data for Makrolon 2803 before and during natural weathering in external conditions

Exposure time, (months)	Tensile strength, MPa	Elongation to break, %	Charpy impact resistance, kJ/m ²	Shore hardness, H Sh D	Vicat softening temp. °C	Density, g/cm ³	Water absorption, mg/100 cm ²
Continental location							
0	61.0±2.8	120.1±5.6	No break	81	167	1.20	53
6	60.2±2.6	120.0±5.7	No break	83	166	1.20	49
12	60.5±2.7	118.9±5.3	No break	83	163	1.20	48
18	52.3±2.2	114.1±5.1	No break	84	163	1.20	47
24	51.8±2.1	113.4±5.8	No break	83	161	1.20	47
30	47.1±2.7	108.3±4.9	No break	81	160	1.20	46
36	46.3±2.0	46.3±1.6	108.4±4.9	82	160	1.20	45
42	40.7±2.4	40.3±1.8	84.6±4.1	80	160	1.20	45
48	39.5±1.6	32.1±1.4	36.8±2.9	81	159	1.20	44
54	40.6±1.7	24.5±1.1	6.1±0.3	83	160	1.20	44
60	35.0±1.6	21.2±1.6	5.8±0.2	82	161	1.20	42
66	34.2±1.8	18.6±1.4	4.0±0.2	83	160	1.20	41
72	32.0±1.4	16.7±1.1	3.9±0.2	83	159	1.20	41

78	28.3±1.7	14.2±0.9	2.3±0.1	82	158	1.20	40
84	24.8±1.0	13.8±0.8	1.3±0.1	83	158	1.20	39
90	23.4±1.4	13.1±0.9	1.1±0.1	81	157	1.20	38
96	24.1±1.8	12.6±1.0	1.0±0.08	80	158	1.20	38
Marine location							
0	61.0±2.8	120.1±5.6	No break	81	167	1.20	53
6	60.6±2.7	119.4±5.5	No break	83	166	1.20	48
12	60.2±2.6	118.7±5.2	No break	83	163	1.20	47
18	53.8±2.4	115.7±5.1	No break	82	162	1.20	47
24	51.2±2.8	114.6±4.9	No break	83	160	1.20	46
30	48.3±2.4	107.3±5.4	No break	82	160	1.20	45
36	47.2±2.2	47.1±1.8	96.8±4.2	81	159	1.20	44
42	39.6±1.8	39.6±1.9	82.4±3.9	81	158	1.20	43
48	38.8±1.7	31.0±1.4	34.0±2.6	83	159	1.20	43
54	38.2±2.1	25.3±1.2	4.1±0.3	83	158	1.20	42
60	34.1±1.6	20.4±1.0	4.0±0.2	82	159	1.20	42
66	33.7±1.5	18.1±0.8	3.7±0.2	83	158	1.20	44
72	33.1±1.5	15.4±0.6	2.1±0.1	83	159	1.20	40
78	27.4±2.0	14.2±0.5	1.3±0.1	82	158	1.20	39
84	24.1±1.7	13.6±0.6	1.2±0.1	81	157	1.20	38
90	22.9±1.4	12.9±0.5	1.1±0.1	81	157	1.20	41
96	21.9±1.6	12.4±0.6	1.0±0.09	80	157	1.20	39

Data for tensile strength, elongation to break, Charpy impact resistance, Shore hardness, Vicat softening temperature, density and water absorption obtained by investigation of Makrolon 3203 before and during natural weathering in external conditions at two mentioned locations for a period of 96 months are presented in Table 2.

Table 2. Data for Makrolon 3203 before and during natural weathering in external conditions

Exposure time, (months)	Tensile strength, MPa	Elongation to break, %	Charpy impact resistance, kJ/m ²	Shore hardness, H Sh D	Vicat softening temperature, °C	Density, g/cm ³	Water absorption, mg/100 cm ²
Continental location							
0	65.0±3.1	118.1±5.8	No break	84	167	1.20	52
6	64.2±3.0	115.2±5.7	No break	83	166	1.20	47
12	63.1±2.9	110.9±5.3	No break	83	163	1.20	46
18	63.0±2.9	109.4±5.4	No break	84	162	1.20	46
24	62.8±2.8	108.8±4.6	No break	84	161	1.20	45
30	62.1±2.7	102.6±4.8	No break	82	160	1.20	46
36	62.5±2.7	65.2±2.9	No break	82	159	1.20	44
42	62.3±2.6	64.8±2.9	No break	80	160	1.20	42
48	62.1±2.5	60.4±2.8	No break	82	160	1.20	41
54	62.4±2.5	54.5±3.1	No break	81	159	1.20	41
60	62.8±2.6	50.1±2.4	No break	82	158	1.20	42
66	62.0±2.4	48.3±2.2	No break	82	160	1.20	42
72	62.2±2.5	47.2±2.7	No break	81	159	1.20	41
78	61.8±2.4	46.2±2.2	No break	82	160	1.20	39
84	61.1±2.3	44.3±3.0	No break	81	159	1.20	39
90	60.8±2.3	42.1±1.9	No break	80	159	1.20	39
96	60.6±2.2	41.8±1.8	No break	80	159	1.20	38

Marine location							
0	65.0±3.1	118.1±5.8	No break	84	167	1.20	52
6	64.4±3.0	114.2±5.6	No break	83	165	1.20	48
12	64.2±2.4	112.4±5.0	No break	83	165	1.20	47
18	63.6±2.6	110.1±4.9	No break	82	164	1.20	45
24	63.1±3.5	107.5±4.9	No break	80	163	1.20	46
30	62.9±2.4	101.8±5.2	No break	81	162	1.20	44
36	63.1±2.7	63.1±3.0	No break	80	160	1.20	43
42	62.8±2.5	64.1±3.1	No break	80	161	1.20	43
48	62.6±2.9	62.3±4.0	No break	81	161	1.20	42
54	62.7±2.5	56.7±2.5	No break	81	160	1.20	41
60	62.4±2.6	51.2±2.4	No break	82	161	1.20	42
66	62.2±3.1	49.6±3.3	No break	81	159	1.20	41
72	61.8±2.0	48.3±2.2	No break	81	160	1.20	41
78	61.2±2.1	47.0±2.8	No break	81	160	1.20	39
84	61.0±2.9	44.8±3.0	No break	80	159	1.20	38
90	60.4±2.0	42.3±1.9	No break	80	159	1.20	37
96	60.1±2.4	42.0±2.8	No break	80	160	1.20	38

In order to realize the first goal of this paper, i.e. to establish the influence of natural weathering in outdoor conditions at continental location and at marine location for a period of 96 months, data from Table 1 and Table 2 are presented in Diagrams.

In diagrams 1-4, where the changes of tested characteristics during mentioned exposure times are expressed regarding initial quality, number (1) denotes tensile strength, number (2) elongation to break, number (3) Shore hardness, number (4) Vicat softening point, number (5) water absorption and number (6) density.

Diagram 1 presents data for six mentioned characteristics of Makrolon 2803 (expressed in percentage of the initial value before exposure) during the weathering in natural conditions at continental location for a period of 96 months.

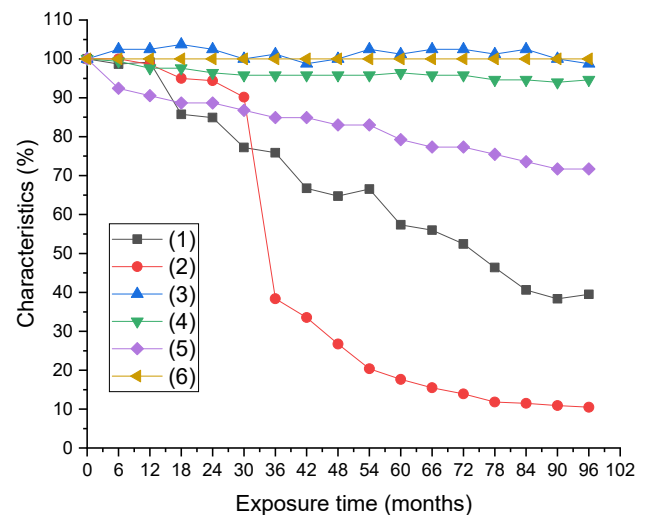


Diagram 1. Tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6) of Makrolon 2803 during the exposure in natural conditions at continental location

Changes of tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6) of Makrolon 2803 in the course of weathering for 96 months in natural conditions at marine location are shown in diagram 2. Data for mentioned six characteristics are presented as percentage of initial quality before natural weathering.

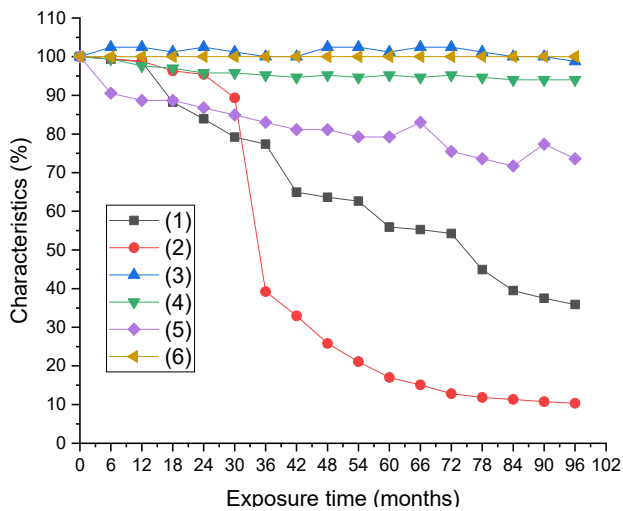


Diagram 2. Changes of tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6) of Makrolon 2803 in the course of weathering in natural conditions at *marine location*

From the first view at diagrams 1 and 2 it can be seen that the changes of presented characteristics of Makrolon 2803 for a mentioned period of exposure in natural conditions, can be divided into three groups:

- small changes (Shore hardness, Vicat softening temperature, and density),
- moderate change (water absorption) and
- high changes (tensile strength and elongation to break).

In diagram 3 the initial quality and changes of mentioned six characteristics of Makrolon 3203 caused by outdoor weathering at continental location during 96 months are shown.

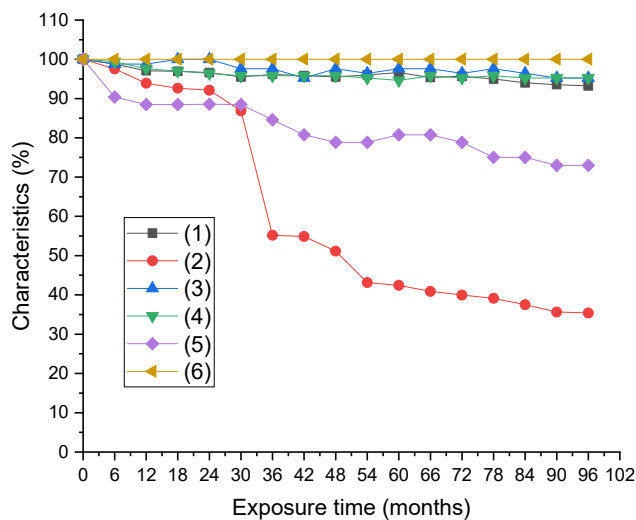


Diagram 3. Initial quality and changes of mentioned six characteristics [tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6)] of Makrolon 3203 caused by outdoor weathering at *continental location*

Data for tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6) of Makrolon 3203 during weathering for a period of 96 months in outdoor conditions at marine location, as percentage of starting values, are shown in diagram 4.

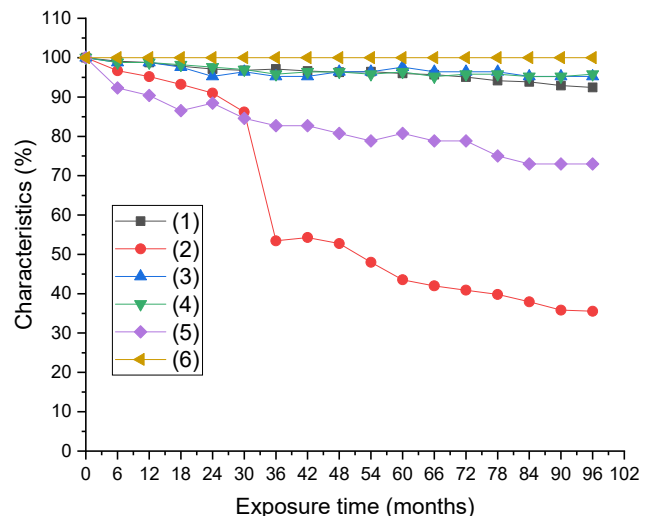


Diagram 4. Data for tensile strength (1), elongation to break (2), Shore hardness (3), Vicat softening point (4), water absorption (5) and density (6) of Makrolon 3203 during weathering in outdoor conditions at *marine location*

Roughly speaking, based on data presented in diagrams 3 and 4, there are three groups of changes of characteristics of Makrolon 3203 after expositions to natural conditions for a period of 96 months at mentioned two locations:

- small changes (tensile strength, Shore hardness, Vicat softening temperature and density),
- moderate change (water absorption) and
- high change (elongation to break).

Products made of thermoplastic bisphenol A polycarbonate trade name Makrolon are mostly used in conditions where three following characteristics are important: tensile strength, elongation to break and impact resistance.

Changes of tensile strength of Makrolon 2803 during natural weathering for a period of 96 months at continental location and marine location are shown in diagram 5.

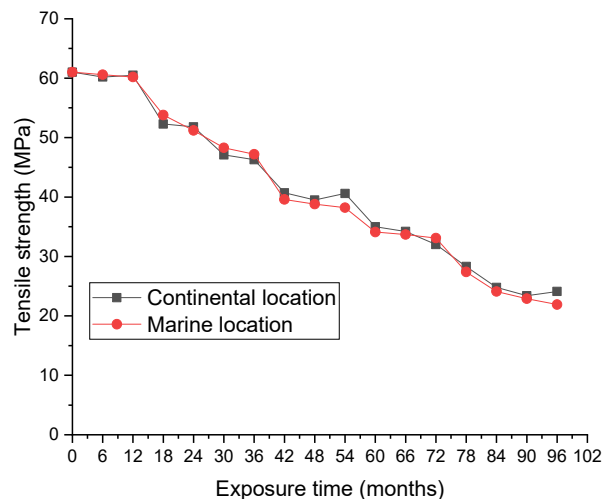


Diagram 5. Changes of tensile strength of Makrolon 2803 during natural weathering for a period of 96 months at continental location and marine location

From the above presented diagram, it can be seen that the changes of tensile strength of Makrolon 2803 at continental location are similar to the changes of mentioned characteristic at marine location.

Diagram 6 presents data from which it can be seen how tensile strength of Makrolon 3203 are changed for a period of 96 months at two mentioned locations.

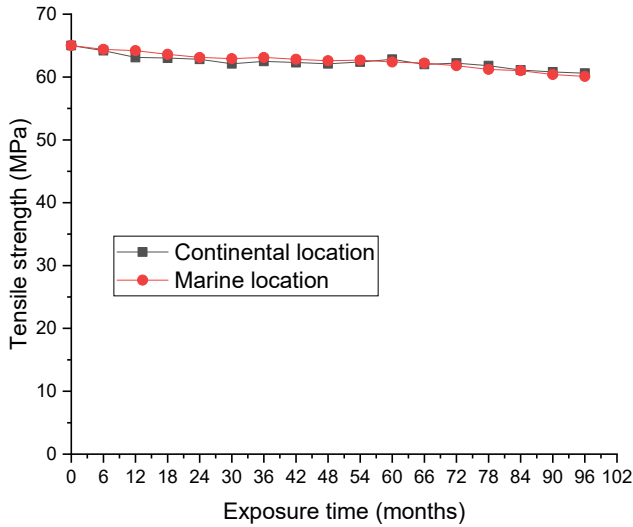


Diagram 6. Data of tensile strength of Makrolon 3203 at continental location and marine location in the course of weathering for 96 months

Data presented in diagram 6 pointed out that changes of tensile strength of Makrolon 3203 at continental location and at marine location for 96 months are almost the same.

Elongation to break of Makrolon 2803 before exposure and during natural weathering in the period of 96 months at continental location and marine location are presented in diagram 7.

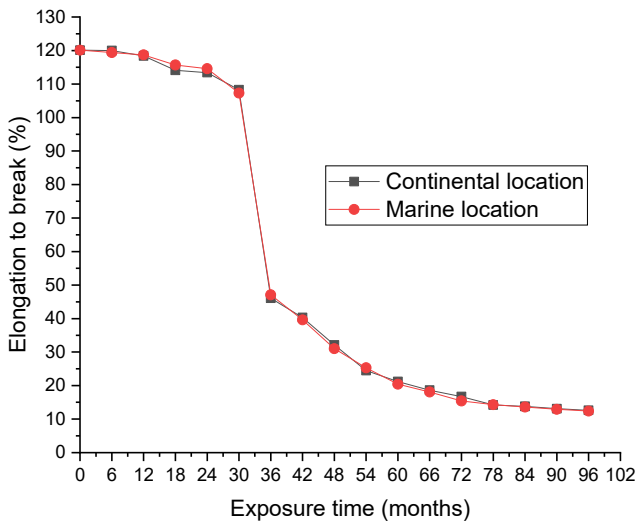


Diagram 7. Elongation to break of Makrolon 2803 for a period of 96 months at continental location and marine location

From diagram 7 it can be seen that there is very similar way of changes of elongation to break of Makrolon 2803 at two mentioned locations.

Diagram 8 shows the changes of elongation to break of Makrolon 3203 at continental location and marine location for 96 months of natural weathering.

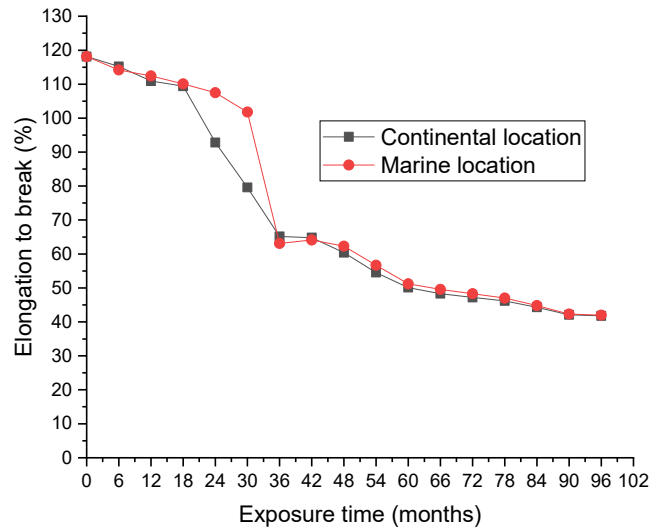


Diagram 8. Changes of elongation to break of Makrolon 3203 at continental location and marine location for 96 months of natural weathering

Changes of elongation to break of Makrolon 3203 at continental location and marine location is very similar in the initial period (for the first 18 months) and for a period from 36 months to the end of outdoor exposure (96 months). Changes of the mentioned property of Makrolon 3203 in the period from 18 months to 36 months at marine location are a little bit different than at continental location.

Data for Charpy impact resistance for Makrolon 3203, presented in Table 2, pointed out that there are no changes of this important characteristic for a period of 96 months of weathering at outdoor conditions regardless of the locations.

Data from Table 1 indicate that the situation with Makrolon 2803 during weathering in the same conditions (period of exposure and locations) are very different concerning Charpy impact resistance. Data for Charpy impact resistance of Makrolon 2803 at continental location and at marine location for mentioned time of natural weathering are shown in diagram 9.

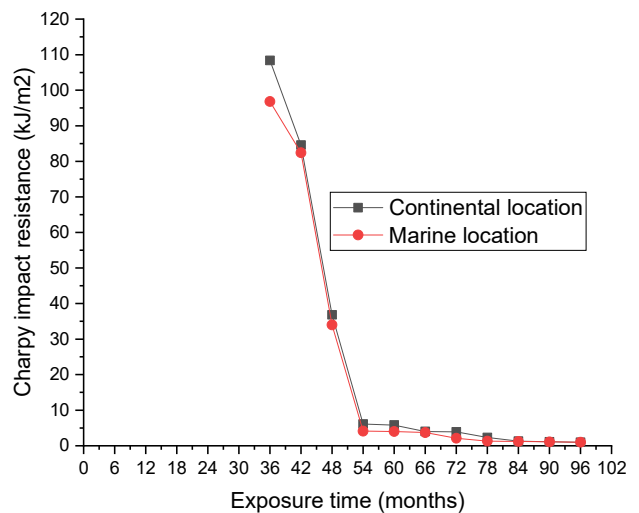


Diagram 9. Data for Charpy impact resistance of Makrolon 2803 at continental location and marine location for 96 months of natural weathering

For the first 36 months of outdoor exposure specimens of Makrolon 2803 from both locations were not broken during testing. After this period, a very important change happened. Charpy impact resistance specimens of Makrolon 2803 were broken for the first-time during testing regardless continental location and marine location. Drop of Charpy impact resistance of Makrolon 2803 in the period of exposure, 36 months - 54 months, at both locations is rapid. After 54 months of weathering in natural conditions it can be told that the values of this characteristic of Makrolon 2803 are negligible at both locations.

Detailed analysis of data concerning Makrolon 2803 during natural weathering in continental location and at marine location for 96 months (shown in Table 1 and in diagrams 1, 2, 5, 7 and 9) indicate that following statements are possible:

- the changes of Shore hardness, Vicat softening temperature, and density were negligible (less than 10 % of the initial values) at continental location and at marine location,
- the change of water absorption was worth mentioning (about 25 % of the initial value) at both locations,
- changes of tensile strength and elongation to break were very meaningful (higher than 50 % of the initial values),
- changes of tensile strength at continental location is almost the same as the changes of this property at marine location, the drop of mentioned characteristic is gradual during whole period of exposure at both locations reaching about 40 % of the initial values at the end of investigation period,
- the trend of reduction of elongation to break at continental location is mostly similar to the change of this characteristic at marine location, the reduction of this property in the period of initial time -30 months is from 100 % to 90 % of the initial value and in the period 36 months - 96 months it is from 40 % to 10 % of the initial value, enormous drop of elongation to break of Makrolon 2803 after 36 months of exposure (from 90 % to 40 %) is an indication that the first significant change of quality occurred,
- a fact that the specimen of Makrolon 2803 was broken for the first time during testing Charpy impact resistance after 36 months of exposure to the natural weathering is a direct proof that an important change of quality happened, the first numerous value of Charpy impact resistance is about 100 kJ/m^2 (after 36 months of exposure), after 54 months of exposure value of mentioned characteristic is negligible (only 4 kJ/m^2), it can be observed that this is the second important change of quality of mentioned thermoplastic material.

Presented data for Makrolon 3203 (in Table 2 and in diagrams 3, 4, 6 and 8) obtained during the investigation of natural weathering in continental location and at marine location for 96 months pointed out following possible statements:

- drop of one of the most important characteristics - tensile strength - is less than 10 % of the initial value at continental location and at marine location,
- there were no changes of one of the most important properties-Charpy impact resistance - at both locations,
- the changes of other three properties (Shore hardness, Vicat softening temperature, and density) were also negligible (less than 10 % of the initial values) at continental location and at marine location,
- the change of water absorption was worth mentioning (about 25 % of the initial value) at both locations,
- change of elongation to break was very meaningful (higher than 50 % of the initial values), the way of changing this

property at continental location is similar to the changes at marine location, the reduction of these characteristics in the period 0 months - 30 months is very small (from 100 % to 90 % of the initial value) and in the period 36 months - 96 months it is obvious (from 55 % to 35 % of the initial value), a diminution of elongation to break of Makrolon 3203 after 36 months of exposure (from 90 % to 55 %) is meaningful as an indication of significant change of quality.

The second goal of this paper is to determine the influence of molecular weight on changes of characteristics of two described polycarbonates types during exposition in an open air for the mentioned period of time at both locations.

Changes of tensile strength of Makrolon 3203 and Makrolon 2803 at continental location during outdoor weathering for 96 months are presented in diagram 10.

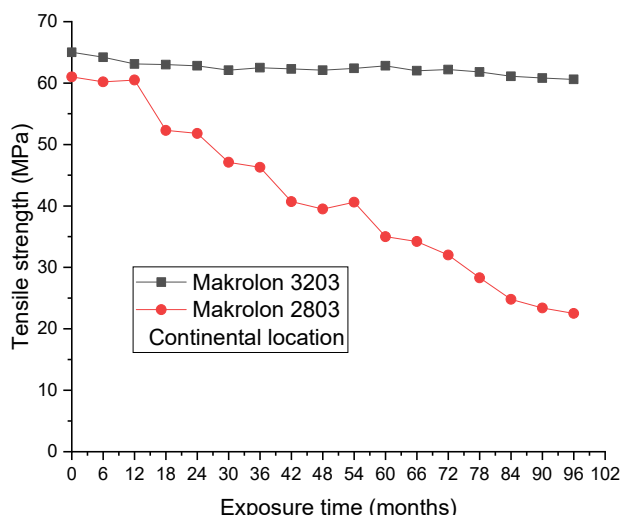


Diagram 10. Changes of tensile strength of Makrolon 3203 and Makrolon 2803 at continental location during outdoor weathering for 96 months

Diagram 11 contents changes of tensile strength of Makrolon 3203 and Makrolon 2803 at marine location during weathering in natural conditions for 96 months.

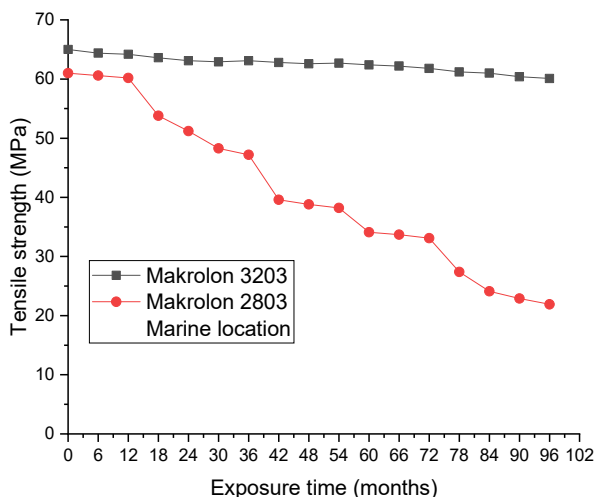


Diagram 11. Changes of tensile strength of Makrolon 3203 and Makrolon 2803 at marine location during weathering in natural conditions for 96 months

From diagrams 10 and 11 it can be seen that the changes of tensile strength of Makrolon 3203 after 96 months of exposure in natural environment are very small no matter locations, while the change of these characteristics of Makrolon 2803 are very meaningful for the both locations and for the same weathering time.

The important differences concerning the way of changing tensile strength of Makrolon 3203 and Makrolon 2803 during natural weathering for a period of 96 months, as a percentage of the initial values, are presented in diagram 12 (continental location) and diagram 13 (marine location).

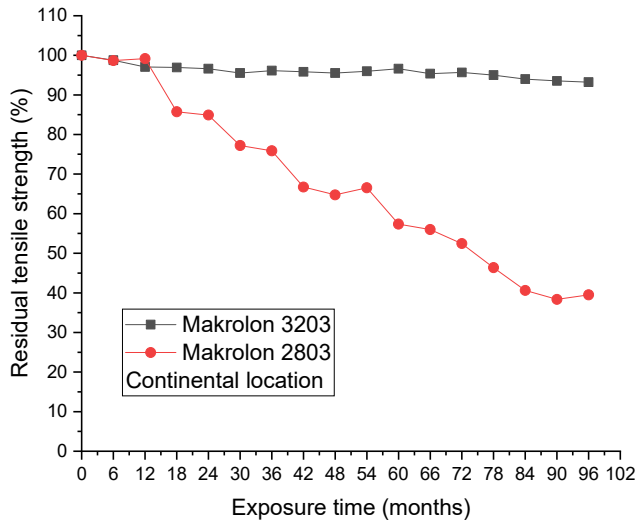


Diagram 12. Difference concerning way of changing tensile strength of Makrolon 2803 and of Makrolon 3203 at *continental location* as a percentage of the initial values

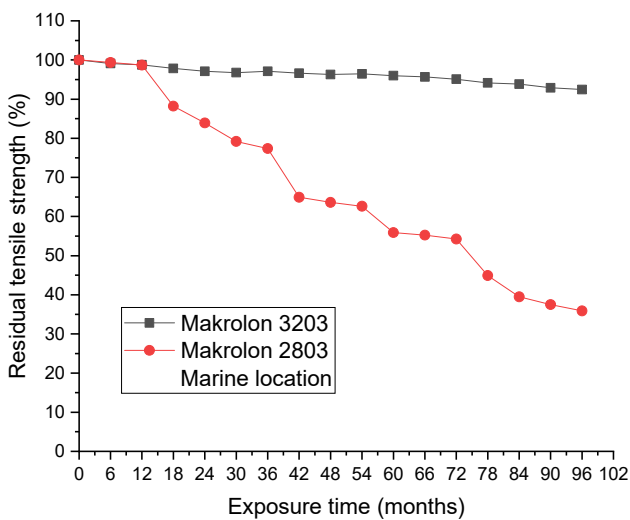


Diagram 13. Difference concerning way of changing tensile strength of Makrolon 2803 and of Makrolon 3203 at *marine location* as a percentage of the initial values

From the above-presented diagrams it is obvious that residual tensile strength of Makrolon 3202 is very high (92 % of the initial value), while this characteristic concerning Makrolon 2803 is considerably lower (only 40 % of value before weathering) after the entire period of exposition at both locations.

How the values of elongation to break of Makrolon 3203 and Makrolon 2803 at continental location are changed in the course of natural weathering for 96 months is presented in diagram 14.

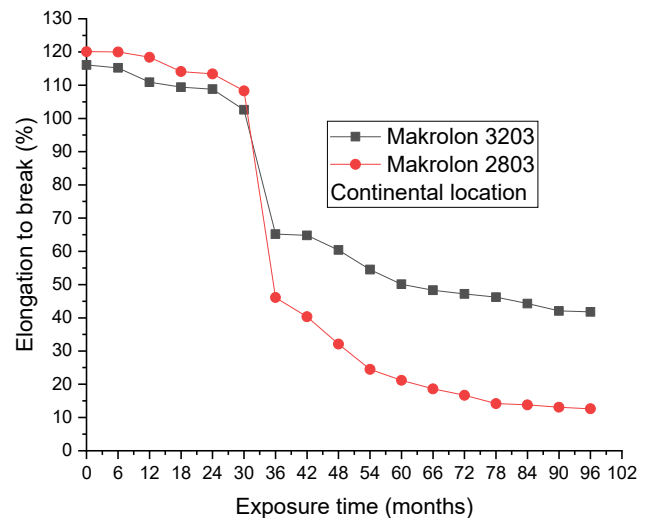


Diagram 14. Changes of elongation to break of Makrolon 3203 and Makrolon 2803 at *continental location* during outdoor weathering for 96 months

Changes of elongation to break of Makrolon 3203 and Makrolon 2803 at marine location during outdoor weathering for 96 months are shown in diagram 15.

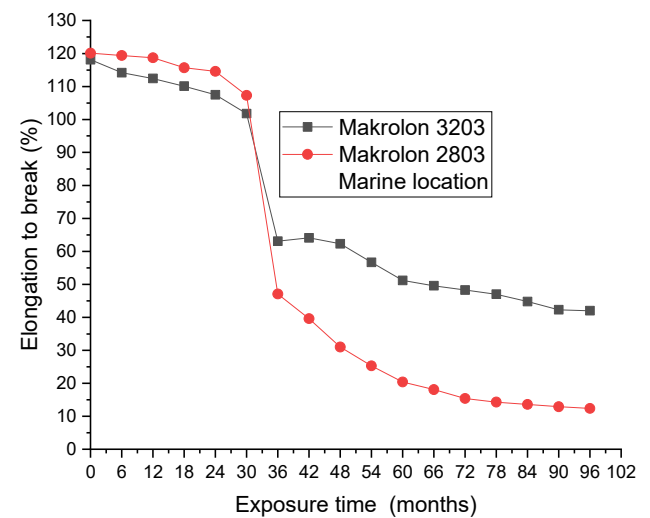


Diagram 15. Changes of elongation to break of Makrolon 3203 and Makrolon 2803 at *marine location* during outdoor weathering for 96 months

Two above-presented diagrams (14 and 15) pointed out that the elongation to break of Makrolon 2803 and Makrolon 3202 showed similar trend of change during weathering for a period of 96 months in outdoor conditions at both locations, but the specimens of Makrolon 2803 showed a higher drop of these characteristics. It is very important to observe that after 36 months of natural exposure a large drop of the elongation to break happened at both mentioned polycarbonate bisphenol A types at continental location and at marine location, but the reduction of mentioned property is much higher at Makrolon 2803 than at Makrolon 3203. This difference of changing of elongation to break at Makrolon 2803 and at Makrolon 3203 during weathering in outdoor conditions for 96 months at continental location (and at marine location) is much clearly visible in diagram 16 (and in diagram 17) where the changes of mentioned property of two above-stated thermoplastic materials are presented as a percentage of the initial values.

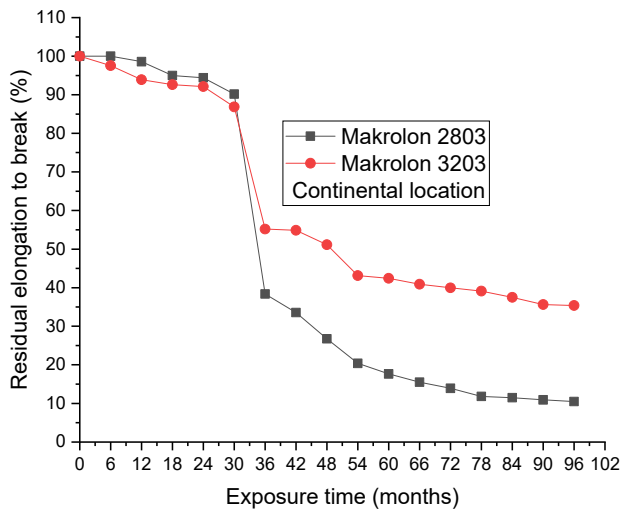


Diagram 16. Difference of changing of elongation to break of Makrolon 2803 and of Makrolon 3203 at *continental location* as a percentage of the initial values

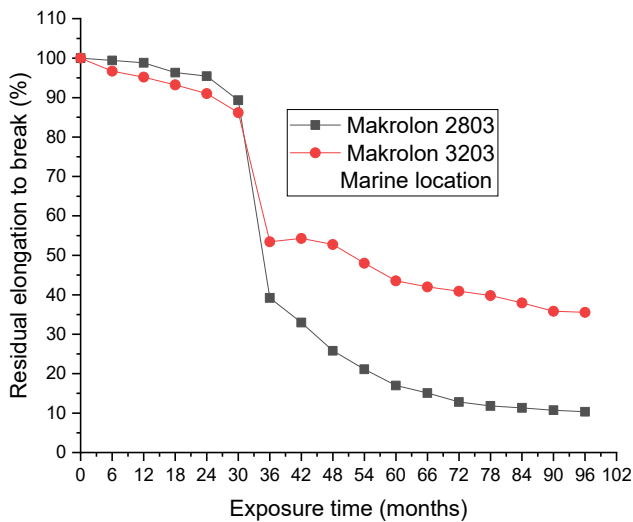


Diagram 17. Difference of changing of elongation to break of Makrolon 2803 and of Makrolon 3203 at *marine location* as a percentage of the initial values

Based on data presented in diagrams 16 and 17 it can be seen that after 96 months of exposure to natural weathering at continental location and marine location the value of residual elongation to break of Makrolon 3203 is significantly lower than the initial value (only 35 % of the value before exposition), but the drop of this property at Makrolon 2803 for the same period of time and the same locations is much higher (insignificant 10 % of the initial value).

Conclusions

Based on the realized investigations and data presented in Tables 1 - 2 and diagrams 1 - 17, it can be concluded:

- Two types of thermoplastic polycarbonate bisphenol A materials, named Makrolon 2803 and Makrolon 3203, were exposed to the natural weathering conditions at continental location and marine location for a period of 96 months.
- Changes of tested seven characteristics of Makrolon 2803 and Makrolon 3203 (tensile strength, elongation to break, Shore hardness, Vicat softening point, water absorption, density and Charpy impact resistance) at continental location are similar to the changes of

mentioned properties at marine location for the same period of exposition in an open air.

- There were two important changes of quality of Makrolon 2803, primarily concerning elasticity, during mentioned period of weathering in natural conditions:
 - the first is after 36 months of exposure when enormous drop of elongation to break and first breakage of Charpy impact resistance specimen were recorded and
 - the second is after 54 months of exposure when a value of Charpy impact resistance of only 4 kJ/m² was recorded, while after 36 months it was about 100 kJ/m².
- Regarding other five tested properties of Makrolon 2803, a meaningful drop of tensile strength (about 60 % of the initial value), moderate change of water absorption (drop of about 25 %) and negligible changes of Shore hardness, Vicat softening point and density (about 10 %) were established after 96 months of natural weathering.
- Two of three very important characteristics of Makrolon 3203 did not change after 96 months of exposure in an open air at both locations (change of tensile strength is only about 8 % of the initial values and any of Charpy impact resistance specimens did not break).
- Drop of elongation to break of Makrolon 3203 is meaningful (about 65 % of the initial value), diminution of water absorption is moderate (about 25 % of the initial value) while changes of remained three characteristics (Shore hardness, Vicat softening point and density) are less than 10 % of the initial values after natural weathering for 96 months.
- The influence of molecular weight is very evident because high molecular weight polycarbonate Makrolon 3203 exhibited much better resistance to the influence of natural weathering conditions than medium molecular weight polycarbonate Makrolon 2803 during the exposure period of 96 months at both locations.
- Practically there were no changes of tensile strength and Charpy impact resistance of high molecular weight polycarbonate Makrolon 3203 while there were very important changes of two mentioned properties of medium molecular weight polycarbonate Makrolon 2803 (drop of tensile strength up to 60 % of the initial values and first break of Charpy impact resistance specimen after 36 months and after 96 months value of 1 kJ/m², which is not worth being mentioned).
- The residual tensile strength of Makrolon 3202 is very high (92 % of value before weathering) but the same characteristic of Makrolon 2803 is considerably lower (only 40 % of the initial value) after the same conditions of overall exposition (time and locations).
- The residual value of elongation to break of medium molecular weight polycarbonate Makrolon 2803 is meaningfully smaller (about 10 % of the initial value) than the value of this characteristics of high molecular weight polycarbonate Makrolon 3203 after 96 months of exposure (about 35 % of the initial value) at both locations.

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Ispitivanje prirodnog starenja polikarbonata srednje i visoke molekulske mase na kontinentalnoj i morskoj lokaciji za period od 96 meseci

Dva tipa termoplastičnog polikarbonata bisfenola A bila su izložena prirodnom starenju na kontinentalnoj lokaciji i morskoj lokaciji za period od 8 godina (96 meseci). Svaki šest meseci epruvete polikarbonata velike molekulske mase i srednje molekulske mase su uzimane sa pomenutih lokacija i ispitivane. Za definisanje kvaliteta pre izlaganja i za vreme procesa degradacije dva pomenuta tipa polikarbonata izabrano je sedam karakteristika (zatezna čvrstoća, prekidno izduženje, tvrdoća po Šoru, tačka omekšavanja po Vikatu, apsorpcija vode, gustina i udarna žilavost po Šarpiju). Promene ovih sedam osobina na kontinentalnoj lokaciji su slične promenama pomenutih karakteristika na morskoj lokaciji za vreme specifičnog perioda vremena. Podaci dobijeni ispitivanjem epruveta polikarbonata srednje molekulske mase ukazuju da su postojale dve važne promene kvaliteta kada su u pitanju prekidno izduženje i udarna žilavost po Šarpiju (prva posle 36 meseci i druga posle 54 meseca izlaganja). Posle 96 meseci prirodnog starenja gore pomenutog materijala na obe lokacije zabeležen je značajan pad zatezne čvrstoće, umerena promena apsorpcije vode i zanemarljive promene tvrdoće po Šoru, tačke omekšavanja po Vikatu i gustine. Uticaj molekulske mase je vrlo vidljiv pošto je polikarbonat velike molekulske mase pokazao veću otpornost prema procesu degradacije u prirodnim uslovima. Dve vrlo važne karakteristike ovog polikarbonatnog materijala (zatezna čvrstoća i udarna žilavost po Šarpiju) praktično se nisu promenile tokom 96 meseci izlaganja na otvorenom, dok je značajan pad prekidnog izduženja, smanjenje apsorpcije vode je umereno, a promene tvrdoće po Šoru, tačke omekšavanja po Vikatu i gustine su vrlo male.

Ključne reči: Termoplastični polikarbonati, degradacija, prirodni uslovi, zatezna čvrstoća, prekidno izduženje, tvrdoća po Šoru, tačka omekšavanja po Vikatu, apsorpcija vode, gustina, udarna žilavost po Šarpiju, uticaj prirodnog starenja, kontinentalna lokacija, morska lokacija, uticaj molekulske mase, rezultati ispitivanja, tabele, dijagrami.