STUDIES INTO AGING OF THE ROOFING COATINGS ON THE BASIS OF CHLOROSULFOPOLYETHYLENE AND FORECASTING OF THEIR DURABILITY

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The report presents the results of an investigation undertaken to compare the aging process of chlorosulfopolyethylene roofing material under accelerated laboratory conditions vs. 17 years of field performance.

These results permit correlation of the two aging processes so as to facilitate forecasting the durability of similar polymeric roofing materials under accelerated laboratory conditions.

KEYWORDS

Aging, chlorosulfopolyethylene, durability, natural performance conditions, physico-chemical methods, roofing coatings.

INTRODUCTION

The USSR is one of the biggest manufacturers and consumers of the conventional roofing bituminous materials in the world. Annual output of these materials approaches two billion square meters.[1]

Until recently, the main raw materials for the manufacture of the soft roofing coatings were bitumens. Sheet roofing coatings made of conventional bituminous materials—roofing felts—are complicated in application. Due to the temperature sensitivity of the physical properties of bitumens, the ease of application of multilayer bitumen-saturated roofing felts is seasonally affected, particularly by cold weather. Most felts are hand-installed because they are not efficiently handled by mechanized systems.

STUDIES INTO AGING

The conventional sheet roofings application considerably complicates work on curvilinear surfaces of domes, folds, vaults and arches. These materials do not meet high performance and aesthetic requirements imposed upon the aforementioned regions.

The levels of physico-mechanical, technological and performance indices of the conventional roofing bituminous materials does not ensure reliable and durable protection of buildings which are intended for use in such specific conditions as chemical aggression and climatic features of northern and southern regions.

Repairing of the roofs covered with roofing felts operating in conditions of industrial aggression of chemical, oil-processing, metallurgical and other plants with aggressive effluents costs 1 ½ - 2 times more than the construction of the new ones.[2]

The lack of availability of chemically resistant roofing materials leads not only to the replacement of roofs but can also cause yearly losses (up to 10 percent) in the load-bearing capacity of metal reinforced units because they are subject to corrosion. This can result in a loss of its useful life and represents a waste of material resources and manpower. At a real service life for buildings and structures of 60-100 years and for soft roofing of 5 - 7 years, expenditures for repair increase quadratically.[1] Over 60 percent of the 160,000 workers in the nation's roofing industry today are involved with repair work[3] that is unprofitable and does not damage the state.

Regarding the efficieny of application in the building industry, roofing felts rank the lowest among building materials.[1] According to the data of the Scientific Institute of Research into Building Economy of GOSSTROY of the USSR, the average service life of the soft sheet bitumen roofing in the USSR is 5-7 years; up to 30 percent of such roofing begins to leak within 1-2 years, 50-70 percent needs repair within 5 years and all of them must be repaired within 7 years. Repair work constitutes more than 50 percent of the total life cost of such soft roofing.[3]

Moscow can serve as an illustrative example of the conventional bituminous roofings consumption balance. Annually, there are 300,000m^2 of new roofing installed using roofing felts and 5,000,000m^2 of existing roofing that needs repair. But, because of a shortage of repair services and supplies only 3,000,000m^2 of existing roofing are being repaired. One of the main reasons for this present situation with the conventional roofings is the use of poor (quality) and overaged manufactured materials that do not meet the present new product requirements.

Climatic service conditions is one of the principal factors defining the performance requirements for these materials, their application versatility and logistics of production.

Low temperature embrittlement of the main stock of the roofing felt and tar paper produced by the industry is not functional below – 15°C and so they do not meet the performance requirements of over three-fourths of the roof areas in the country.

Most acutely, the USSR is faced with the problem of resistance to low temperature embrittlement of these materials in the regions of the far north, Siberia and the far east. With the dislocation of "the center of gravity" of the industry toward these regions, greater amounts of insulation and other building materials will be needed to meet the more demanding installation and performance requirements.

Taking into consideration the fact that low temperature embrittlement of materials used in these regions should be at least as low as −45°C and roof construction should be carried out on a 12-month basis, the urgent nature of the problem is evident and requires an immediate solution.

Analysis of the domestic and foreign roofing experience suggests that the solution of these problems may lie in the use of polymeric membranes.[4-7]

Based on the progress achieved in chemistry, oil-processing chemistry and roofing technology, all the necessary prerequisites have been satisfied to permit the manufacturing of the materials with a predetermined set of acceptable properties. A wide range of roofing materials in various countries provides support for this view. Suffice it to say that in such small countries as Czechoslovakia and Yugoslavia, not to mention such highly developed countries as the United States, Japan and Federal Republic of Germany, the range of polymeric insulating materials (roofings and waterproofing materials) accounts for many of the names. These materials have found extensive application in building with many being based on elastomers (rubber-like polymers) and thermoplasts.[8-15]

Studies carried out in various countries, and in the USSR as well, revealed the fact that the most desirable installation and performance qualities in roof construction are being demonstrated by elastic roofing materials that resist strains caused by internal and external forces in the supporting structures. The variations between elastomeric and plasticized thermoplastic films from the technical point of view are negligible.

Analysis of physico-mechanical indices of the film roofings on the basis of the above-mentioned polymers showed that they exhibit the following range of the main properties:

- Tensile strength, MPa 0.5 - 25
- Relative elongation, % 100 - 500
- Resistance to low temperature embrittlement, °C −30 - −60
- Shrinkage, % 0.5 - 3.0
- Water absorption, % 0.1 - 5.0
- Durability, years 10 - 50

Despite the considerable variations in indices, all the materials are being recommended for roofing applications. This is brought about by the fact that at present there are no scientifically based requirements on properties and indices of the polymeric roofings. Such a situation can account for the extremely complicated solution to the problem. Accordingly, much of the information relating the polymeric roofings is of a publicity nature, that is marketing of desirable unquantified attributes. Considering the variety of polymeric roofings on the market, it is difficult to judge technical and economic efficiency of the roofings without having objective methods for measuring their durability. Durability is the main factor in estimating the efficiency of the new roofing materials and structures.

The main performance characteristics of the elastomeric roofing materials are their strength and elasticity. Their retention at a certain level within the range of operating temperatures provides the roof efficiency. The available (USSR) data do not contain information relating to qualitative criteria.

Analysis of research carried out at VNIIStroypolymer indicates that, with respect to technological and performance properties, one of the most promising film-forming polymers for roofing materials meeting contemporary architectural and building requirements is chlorosulfonated polyethylene.

Studies at the institute started in the 1970s. In the early development of the roofing compounds based on CSPE, there were no scientifically substantiated requirements which would allow predicting their serviceability as a roof coating. The available information relating CSPE roofing materials was of a promotional character. It became necessary to perform a lot of research and experimental works in the following directions:

- Carrying out the laboratory tests and natural exposures directed to the determination of the scientifically substantiated requirements to the properties and characteristics of the materials.
- Developing of the roofing compounds and manufacturing technology.
- Mastering the application technology of the polymeric roofing and waterproofing materials.
- Estimating durability in the laboratory and natural conditions.

These studies have resulted in the development of mastic roofing, waterproofing and anticorrosive compounds with a complex of properties and a wide range of indices. Technical characteristics of the film coverings made of mastic CSPE compounds are listed in Table 1.

Based on laboratory test results of field exposed samples taken from experimental roofs in various climatic zones of the USSR, the requirements have been determined to properties and indices of the film CSPE coverings ensuring performance reliability of the roofings on a concrete substrate (Table 2).

The given requirements and indices are minimum ones, i.e., those at which crack resistance of the film covering laid on a concrete base is ensured (at a size of cracks in a concrete base up to 1.5mm). Our investigation revealed that the increase of strength and flexural properties of the coverings—higher than those in Table 2—contributed to their reliability. It is supported by the 20 years of performance experience of CSPE roofings. The possibility of obtaining coverings with indices considerably higher than the minimum ones creates the necessary precondition for making coverings with a high durability. There are few published papers dedicated to the investigation of the polymeric roofing durability. Most of these works' treatment of the problems concerning aging of the materials is under natural and laboratory conditions.

In a number of countries accelerated aging tests are used to estimate durability of polymeric roofing materials in laboratory conditions; however, the data confirming correlation of the accelerated tests with the natural ones are not provided.[5-16]

At VNIIStroypolymer there have been extensive and prolonged studies carried out on the aging process of CSPE roofing materials under artificial and natural conditions.

When choosing the accelerated test conditions, the important factors effecting the material aging have been taken into consideration.

Preliminary investigations into the influence of various climatic factors on changes of the main performance characteristics (strength and relative elongation) confirmed the importance of irradiation and high temperature effect, and insignificance of freezing - thawing and spraying (rain).

In view of this, the accelerated aging tests under laboratory conditions have been carried out on free film samples with 1mm thickness in artificial weather chambers—weatherometers with xenon lamps DKST - 6000 at a "black panel" temperature 55°C-60°C. Aging tests in the natural performance conditions were performed on the roof of a chemical plant, located in a subtropical climate with the influence of the active solar radiation of the south, chemically aggressive surroundings of the plant and the salty sea climate of the Caspian Sea (Sumgait, Az.SSR).

For the the material aging study, both free film samples and the material in a real performance condition of a roof were used.

The roof covering was performed by means of the application of a one-component vulcanizing CSPE mastic compound.

One of the main purposes of the investigation was to study the roofing material behavior in a real tough performance of the roof structure with regard to all the factors of exposure, both internal and external (relaxation processes, adhesional forces, etc.).

Mastic covering was applied over a concrete base. The roof structure was thermally insulated. The work was performed in April 1970 at 23°C and 25°C.

When making the roof covering with mastic CSPE compound according to the painting technology, there have been prepared films with 1mm thickness for the simultaneous tests of accelerated aging in the artificial weather apparatus.

The second purpose of the investigation was to study the material behavior and changing of its main performance indices (strength and elasticity) under the constant influence of the significant aging factors; irradiation of a xenon irradiator (the distribution of the xenon lamps irradiation is approximately that of the sunlight at the earth's surface) and a high black panel temperature (60°C) in order to compare these data with those obtained at the real performance of the roof.

The combined program of the investigation of the materials durability based on the main performance characteristics (breaking strength and elongation at break) was carried out by three methods:

- By accelerated tests under laboratory conditions comprising all the factors simulating the atmospheric ones.
- By simultaneous laboratory tests and natural exposure with the use of the standard sample of the covering.
- By the data obtained from the natural weathering test of the material in the real performing conditions.

The purpose of the work was:

- To study the aging process of the polymeric roofing material developed at VNIIStroypolymer using USSR-produced CSPE under natural and artificial conditions.
- To establish a correlation between natural and accelerated tests to forecast the durability of the materials.

One formulation was tested. It was chosen on the basis of preliminary laboratory studies. This formulation is scientifically substantiated.

The subject of the investigation was the vulcanized film polymeric roofing material made of the mastic compound containing the USSR-made CSPE similar to Hypalon 20 (DuPont Co.) as a film-forming base (Table 3).

The earlier investigations[5,17,18] revealed that CSPE is a weather resistant polymer. Roofs made from the mastic compounds on CSPE basis applied in the severe performance conditions of the north, the south and in the environment with the presence of chemical aggression also demonstrated high performance characteristics.[5]

Aging tests of the film samples prepared from mastic were carried out in a weatherometer with the xenon irradiator DKST - 6000 for 3800 hours. The selection of samples was performed in 100, 32, 750, 1750, 3000 and 3800 hours. Reference samples were placed into ordinary laboratory conditions (temperature 20°C, humidity 65-70 percent). Natural exposure of the samples was carried out in a subtropical climate for 17 years (Sumgait). Samples were cut from a real roof covering, i.e., they were subjected to all effects of the natural roof performance (climatic, adhesional and relaxing), which is of significant importance for the reliable correlation of the aging process in natural and artificial conditions.

The aging process of a mastic material on CSPE basis studied by means of differential thermal analysis (DTA), thermogravimetric analysis (TGA), infrared (IR) spectroscopy and physico-mechanical tests. DTA and TGA studies have been carried out by means of Derivatograph OD-102 within the range of 20°C-800°C in air at a rate of a temperature rise of 9°C/min and a sample weight of 200 mg. IR spectra have been recorded by a spectrophotometer "Specord 751R" in the frequency range of 400-4000 cm^{-1}. Changes in CSPE molecular structure have been studied by the magnitude of the optical density of the analytical absorption bands (AB). Physico-mechanical characteristics of the samples (strength and elongation at break) were determined by means of the breaking machine M-250 at a rate of 500 mm/min.

IR spectra analysis of samples subjected to artificial and natural aging (Figure 1) reveal the decrease of AB 1365 cm^{-1} (valence fluctuations of SO_2 -groups in sulfochloride groups and intensity increase of the new AB 1720 cm^{-1} and 3400 cm^{-1} (valence fluctuations of $C=O$ and OH groups).[19] It is likely that these changes in IR spectra of the samples result from the photooxidation process of CSPE destruction comprising both dehydrochlorination of oxidation reactions developing by radical - chain mechanism and are characteristic to the chlorinated polymers. As is evident from Table 4, the greatest changes of the optical density magnitude (D) of the analytical absorption bands occur in IR spectra of samples during 1750 hours or irradiation (artificial aging). Further prolongation of the aging period (up to 3800 hours) causes minor changes in these magnitudes D_{1720} and D_{1365}. IR spectra of samples after the artificial aging for 3000 and 3800 hours and 9-17 years of the natural exposure are characterized by the broad scattered AB that are typical of the cross-linked polymeric structures with a very intensive absorption within the wave number interval of 1600-1700 cm^{-1} and 3200-3500 cm^{-1} with a maximum of 3400 cm^{-1} (valence fluctuations of OH groups). That is indicative of hydrogen links strengthening among oxygen-containing groups of the aged CSPE encouraging the structuralization of the material.[20] It should be noted that a rise in AB 1560 cm^{-1} intensity (valence fluctuations -C-O-Me-) in IR spectra of the aged samples that probably results from saltformation increases also enhancing the structuralization of the covering.

A similar tendency was observed during the artificial aging process of the samples on the curves of changing in strength and elongation at break (Figure 2): the greatest changes in properties appear within the range of 1750-2000 hours of exposure, further prolongation of the aging period has little or no effect on the material properties.

The differential thermal analysis (DTA) and thermogravimetric (TG) studies on the aging process of the polymeric covering revealed that the three endothermic effects have been marked on the DTG curve of the starting sample, which extreme temperatures are in conformity with a maximum rate of the process running: dehydrochlorination in C-C1 and sulfogroups - 250°C, depolymerization of polyethylene - 450°C, depolymerization of polymeric chains terminations - 530°C (Figure 3).

Three clearly defined steps of the samples' weight decrease on the TG curve fit these processes; it should be noted that the slight weight decrease within the range of 60°C-180°C is due to the splitting and decay of chlorosulfon groups with the formation of sulfur dioxide and hydrogen chloride.

The exothermic effect on the differential thermal analysis curve within the range of 240°C-340°C is the total expression of the exothermic processes of dehydrochlorination and exothermic processes of the spatial structure formation and polymer oxidation; the endothermic effect of minimum 410°C corresponds to the break of the double linkage formed as a result of hydrogen chloride splitting from chlorosulfon groups, and 460°C - polyethylene depolymerization; the exothermic effect within the range of 540°C-600°C is due to the thermooxidation destruction - breakage of depolymerized PE chains.

Derivatogram of CSPE film irradiated for 3800 hours (Figure 3) differs from that of the starting film by the temperature rise of all endo- and exothermic effects that is indicative of the polymer cross-linked structure formation contributing to the thermal stability of the material (Tables 5 and 6). Further changes in the samples destruction character at heating depending on the duration of irradiation can be traced by the weight decrease (Table 5).

The analysis of TGA results obtained for the films irradiated in xenon weatherometer for 750-3800 hours and in the natural conditions for 16 years reveals the fact that the extreme temperatures of thermal effects gradually increase with the increasing of irradiation time (Figure 3 and Table 6). Temperatures of the maximum dehydrochlorination rate increase by 10°C-28°C, depolymerization by 10°C-20°C, total destruction by 70°C compared to unirradiated films. These changes are indicative of the development of cross-linking in the polymeric covering during the aging that tends to increase with an increase of irradiation time.

The results of spectral, DTA and thermogravimetric studies on the aging process of the roofing materials on CSPE basis after 750 hours of xenon irradiation are the closest to the data obtained for the samples after two years of performance under the real roof service conditions. This enables one to equate 360-380 hours of films irradiation time of films in the xenon weatherometer at 55°C-60°C to a year of performance in the climatic conditions of the south (Sumgait), and facilitates forecasting the durability of the materials based on CSPE. By this means, the anticipated durability of the roofing material which has undergone the irradiation test for 3800 hours is over 10 years. Physico-mechanical tests of the covering after 16 years of exposure in the real performance conditions demonstrated the properties stability, and the absence of cracks and retaining of the integrity of the polymeric covering point to a high durability of the material and its reliability in performance.

In this manner during the studies on the aging process of the polymeric covering on CSPE basis, there have been stated reactions of dehydrochlorination, oxidation and structuralization of the polymer with the prevalence of the latter.

It should be noted that apart from the above-mentioned tests on the roofings based on CSPE, including the ones modified with bitumen, have undergone aging tests according to VNIIKrovlya procedure in chambers of artificial weather under the influence of the artificial climatic factors approaching the Moscow climate (II B climatic region).

The conditional yearly test cycle has four test conditions (Table 7) where the roofing samples undergo the influence of climatic factors (cold, heat, humidity and solar radiation) during a year of service.

After the termination of each conditional yearly test cycle, samples have undergone visual evaluation on the seven - mark system.

Aging resistance is determined by the changing of the samples appearance according to GOST 18956-73 "Roofing Materials, Aging Tests Under the Effects of Artificial Climatic Factors." The number of test cycles is taken as the value of the material aging resistance corresponding to stage V of destruction when 50 percent of a samples' surface has undergone destruction of some type. As a result of the tests, it was found out that the CSPE roofings developed at VNIIStroypolymer under the influence of the artificial climatic factors have withstood 50 conditional yearly aging cycles and reached stage III of destruction according to GOST 18956-73, confirming their aging resistance and reliability in performance.

SUMMARY

Based on the performed studies, it has been concluded that the projected durability of the tested CSPE roofing coverings would be more than 50 years and that the mentioned materials would prove to be the most durable.

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Properties	Indices	Test Method
1	2	
Breaking strength, MPa	1.5 - 15	GOST 270-75
Elongation at break, %	200 - 700	GOST 270-75
Residual elongation after break, %	0 - 100	GOST 270-75
Adhesion to concrete, MPa	0.5 - 2.0	VNIISTROY - Polymer Procedure
Adhesion to asbestos cement, MPa	0.4 - 1.5	VNIISTROY - Polymer Procedure
Adhesion to metal		
without priming, MPa	0.3 - 1.0	VNIISTROY - Polymer Procedure
with priming, MPa	1.0 - 3.0	VNIISTROY - Polymer Procedure
Water absorption of films, % (by weight)	0.1 - 1.0	VNIISTROY - Polymer Procedure
Water absorption of films, g/cm^2	1.0 - 10	VNIISTROY - Polymer Procedure
Watertightness at pressure, no less than, atm	8	VNIISTROY - Polymer Procedure
Cycling from low room temperature	300 - 500 cycles	VNIISTCRO - VLJA Procedure
Low temperature flexibility on a rod with dia. 10mm, °C	-40 - -65	VNIISTCRO - VLJA Procedure
Temperature stability, °C	120 - 150	VNIISTCRO - VLJA Procedure
Stability:		
- to solar radiation	stable	VNIISTCRO - VLJA Procedure
- to sea salted air together with solar radiation	stable	VNIISTCRO - VLJA Procedure
- to industrial aggression (gases, aerosols, smoke, dust)	stable	VNIISTCRO - VLJA Procedure
Resistance to chemical reagents for 24 h at 20°C		
- 20% H_2SO_4	no changes in a covering	VNIISTCRO - VLJA Procedure
- 20% HCL	no changes in a covering	VNIISTCRO - VLJA Procedure
- 20% NaOH	no changes in a covering	VNIISTCRO - VLJA Procedure
Flame resistance (covering applied on concrete, reinforced concrete, metal)	doesn't maintain combustion	
Drying time of a layer at 20°C:		
- up to air-dry state, h	0.5 - 1.0	
- before the application of the next layer, h	1.0 - 1.5	
Thoroughly drying time and stabilization of the film covering properties, 24 h cycle	7 - 10	
Roof service life in performance conditions,		
- with industrial aggression, years, more than	20	
- in ordinary conditions, years (according to laboratory estimation)	50	

Table 1 Technical characteristics of the film coverings made of mastic CSPE compounds.

Properties	Indices
Minimum film thickness, mm	0.5
Breaking strength, MPa, no less	1.5
Elongation at break, %, no less	150
Adhesion to concrete substrate, MPa, no less	0.15
Low temperature embrittlement at -50°C, no less	0.1

Table 2 Performance requirements of the roofing made of CSPE compounds.

Properties	Indices
Appearance, form of a finished product	white granules
Chlorine content, %	27
Sulfur content, %	1.8
Acidity (estimated from HCl), no more	0.02
Solubility in CCl_4, %	98

Table 3 Main characteristics of CSPE.

Aging time, hour/year	AB magnitude change, rel. unit	
	1365 cm^{-1}	1720 cm^{-1}
0 (starting)	0.073	0
100	0.061	0.070
320	0.055	0.101
750	0.046	0.124
1750	0.040	0.140
3000	0.040	0.162
3800	0.038	0.205
2 years	0.048	0.119
5.5 years	0.040	0.165
9 years	0.031	0.250
15 years	0.028	0.300
16 years	0.026	0.310
17 years	0.025	0.300

Table 4 Changes in the optical density (D) of some AB in IR spectra of polymeric covering on a CSPE basis.

Irradiation time,hour/ year	Weight loss at heating within the temperature range, %							Total weight loss at 70°C, %
	20-100°C	100-200°C	200-300°C	300-400°C	400-500°C	500-600°C	T°C of the total polymer destruction	
1	2	3	4	5	6	7	8	9
Starting	1.0	3.0	14.0	4.0	45.0	12.0	630 1^{x}	80.0
750	1.0	3.0	13.5	4.5	43.5	13.0	645 1.5	80.0
1750	1.0	3.0	12.0	6.0	43.5	11.5	675 3.0	80.0
3800	1.0	2.5	13.5	6.0	37.5	10.5	700 6.0	79.0
2 years	1.5	3.0	15.5	4.0	37.0	10.5	660 3.5	75.0
9 years	1.1	4.8	13.5	4.3	30.2	12.0	700 5.0	73.0
15 years	2.0	5.8	13.7	4.3	35.4	12.1	700 5.1	78.4
16 years	2.0	7.0	15.0	5.0	34.5	9.5	700 6.5	79.5

x - difference in the weight losses in % within the temperature range of the total polymer destruction minus the weight loss at 600°C.

Table 5 Weight losses at thermogravimetric analysis during the aging process.

Aging time, hour/year	Temperature of the maximum process rate, °C		Temperature of the total polymer destruction, °C
	Dehydrochlorination of sulfochloride groups	PE depolymerization	
Starting	250	460	630
750	260	460	640
1750	260	465	675
3800	260	480	700
2 years of the natural exposure	260	465	660
9 years of the natural exposure	265	470	700
15 years of the natural exposure	275	470	700
16 years of the natural exposure	278	470	700

Table 6 *Extreme temperatures of the chemical processes taking place in CSPE films obtained from differential thermogravimetric curves.*

N of test conditions	Test	Test duration
I	Irradiation at "the black panel" temperature, 50-55°C, h	4
	Immersion in water at 20-24°C, h	2
	Number of tests	70
	Total time of the test condition I, h	420
II	Transition through 0°C:	
	Exposure at + 10°C, h	3
	Exposure at - 10°C, h	3
	Immersion in water at 20-22°C, h	6
	Number of tests	25
	Total time of test condition II, h	300
III	Freezing at -40°C, h	75
IV	Temperature change from -25°C to -10°C:	
	Exposure at -25°C, h	3
	Exposure at -10°C, h	3
	Number of tests	10
	Total time of the test condition IV, h	60
	Total time of the conditional yearly cycle, h	855

Table 7 *Conditional yearly test cycle.*

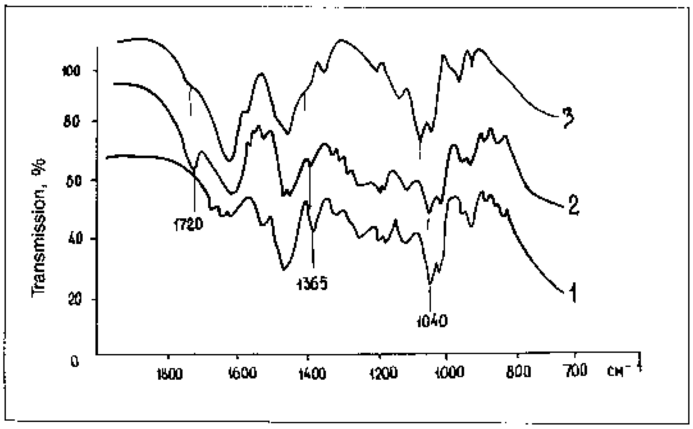

Figure 1 *IR spectra of films on CSPE basis before (1) and after the aging process during 3800 hours (2) and 16 years (3).*

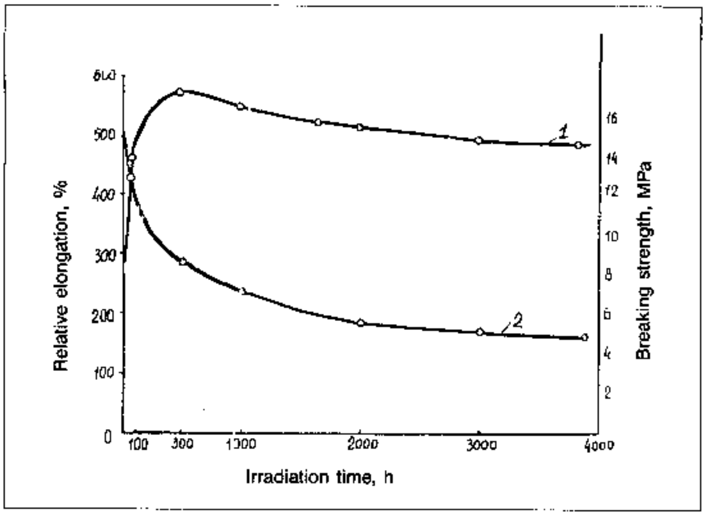

Figure 2 *The relationship between the breaking stress at tension (1), breaking elongation (2) of the films on CSPE basis and the irradiation time.*

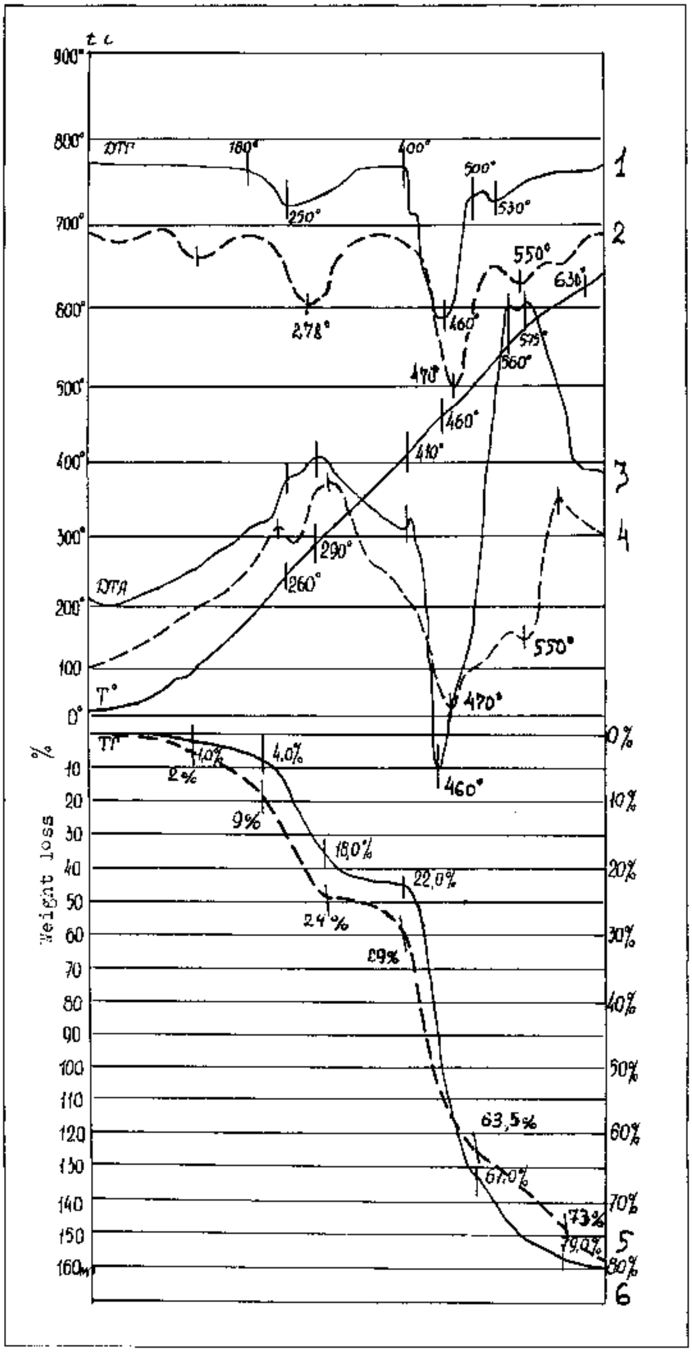

Figure 3 DGT (1,2), DTA (3,4) and TG (5,6) curves of the films on CSPE basis before (1,3,6) and after the aging process within the period of 16 years (2,4,5).