

# Oxidation of LDPE and LDPE grafted with itaconic acid, in contact with water

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## Abstract

The accumulation of carboxyl groups in low-density polyethylene (LDPE) and LDPE grafted with itaconic acid on the macromolecules kept in hot water, aqueous electrolytic solutions, ethyl and isobutyl alcohols have been investigated. Simultaneously, a photo-initiated oxidation by UV-radiation has also been studied. It was revealed that functionalization of the polyethylene accelerated its thermal and photo-oxidation processes in water. The depth which the oxidation front of macromolecules reached in aqueous media was larger than that in air. Alkalisiation of the aqueous solutions led to a faster oxidation of functionalised polyethylene, but in acidic media the oxidation behaviour did not in fact change as compared with that in an inert environment. When oxidation was photo-initiated in an aqueous medium the light caused a decisive influence on the acceleration of the process. The results obtained show that such oxidation was accelerated owing to hydroperoxide decomposition initiated by water. © 1999 Elsevier Science Ltd. All rights reserved.

*Keywords:* Polyethylene; Water; Oxidation

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## 1. Introduction

Polymeric materials are often used in aqueous media, e.g. water, steam and aqueous solutions of electrolytes. Unlike the radical reactions typical of thermal, thermooxidative or photolytic processes of polymer degradation, in aqueous media the macromolecules most often undergo hydrolytic decomposition, which process follows the ionic mechanism. To increase the service life of polymeric materials in aqueous media they must be protected against ionic-hydrolytic degradation [1–3].

Polyethylene (PE) like other polyolefins is hydrophobic, and so interacts but rather weakly with polar liquids, particularly with water. This contribution leads to its high resistance to hydrolytic decomposition. Being a linear polymer in which chain molecules interact through weak dispersive forces, PE is characterised by relatively high gas permeability [3]. Hereto the dissolution of gases, including oxygen, in PE should increase with the absorption of the liquid [3]. In studies dealing with polyolefins oxidation in aqueous medium [4–6] it was reported that dissolved oxygen would initiate thermooxidative transformations in the macromolecules followed by their increased polarity and

variations in water permeability. An aqueous medium can thus directly influence the course of the elementary stages of the chain reaction when the polyolefins undergo oxidative transformation, and can accelerate the output of oxidation products, as compared to air. It is thought [6] that oxidation of olefin macromolecules is accelerated in water owing to hydroperoxide decomposition at a stage when the chain reaction of oxidation is initiated.

The information obtained concerns only virgin polyolefins and those containing antioxidants. However, the modern chemical engineering uses often polyolefins with grafted functional (most often oxygen-containing) groups. They find application in the technology of polymer–polymer composite materials (polymer blends) and in developing of adhesion joints with different hard surfaces [7–11]. The information available shows that no detailed investigation has been made in the kinetics of oxidation, in aqueous media, of polyolefins modified as mentioned. Under real atmospheric conditions of operation, besides both water and temperature, solar radiation is a most important factor, which influences the service stability of polymeric materials. Especially harmful for polymers are UV-rays having length of  $\lambda < 400 \mu\text{m}$  ( $300\text{--}325 \mu\text{m}$  for polyethylene [12]) under the influence of which oxidative transformations take place, and degradation and crosslinking are activated. Since the mechanisms of photochemical oxidative

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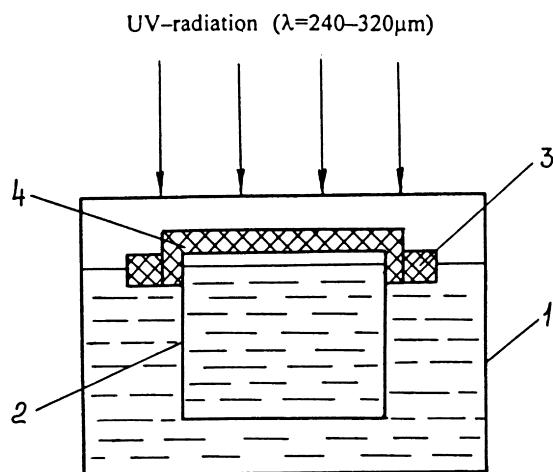


Fig. 1. Scheme of photo-oxidation for film samples.

transformations in macromolecules and similar reactions caused by thermal effect are alike [12], investigating simultaneous influences of water and UV-radiation on the oxidation kinetics of polyolefins seems useful.

The question that is important is whether the accelerating action of water shows itself during simultaneous action of UV-radiation. The present work deals with the development of ideas about the mechanisms of oxidation in aqueous media of the virgin polyethylene and that functionalised by carboxyl groups grafted onto the PE macromolecules.

## 2. Experimental

### 2.1. Materials

Antioxidant-free low-density polyethylene (LDPE), commercial grade 10803-020, USSR Standard 16337-77, density  $0.92 \text{ g/cm}^3$ , melting point  $105^\circ\text{C}$ , was used in the experiments. LDPE extracted with acetone during 72 h to remove antioxidants and low-molecular weight compounds [6] was also studied. Also, LDPE grafted with itaconic acid, LDPE-*g*-IA, was tested. IA was grafted in molten LDPE by reactive extrusion using a static mixer [13]; the shear rate was  $\gamma = 50 \text{ s}^{-1}$ . The concentration of the itaconic acid introduced into the LDPE was 1 wt.%. The grafting efficiency measured by the amount of the grafted acid was about 60 wt.%. In some experiments, ungrafted IA (about 40 wt.% from the total quantity introduced into the LDPE) was extracted in boiling ethanol for 10 h [14]. The liquid media were deionized by doubly distillation of water, and acidic and alkaline aqueous solutions made from it; also, ethyl and isobutyl alcohols were used.

The LDPE was stabilised with hydroquinone, Benzene OA (obtained from Research Institute for Chemical Additives to Polymers, Tambov, Russia), and Irganox 1010 (made by Ciba Geigy, Switzerland). The stabilised compositions were prepared on a Brabender Plasticorder equipped

with a static mixer, as described elsewhere [13]. Granulated LDPE was covered with the solution of a stabiliser and dried; the mixture was extruded, cooled and granulated again.

### 2.2. Preparation of test specimens

Test film specimens having thickness between 30 and  $400 \mu\text{m}$  were prepared from the above polymeric materials by compression moulding at  $140 \pm 5^\circ\text{C}$  and 5 MPa for 60–70 s. In a series of experiments without UV-radiation, glass balls were used to immerse the LDPE films in the open glass vessels containing liquid media and freely open to air. The liquids were heated up to certain temperatures. The specimens were kept in the liquids for a pre-set time without stirring. In some experiments the vessels throats were sealed using Teflon gaskets. The liquids in the vessels (except those in sealed vessels) were replaced once a week to ensure the required amounts of oxygen and prevent formation of products, which could etch the vessels' walls.

Experiments on the photo-initiated oxidation of macromolecules under UV-radiation were done in the manner illustrated in Fig. 1. The glass container (2) was filled with a liquid, preheated up to the temperature at which the oxidation process was carried out, i.e.  $96^\circ\text{C}$ . Then the film specimen (4) to be analysed was placed on the throat of vessel (2); the rubber seal (3) was used to fit the specimen tightly against the vessel walls. This vessel was put into the thermostatic chamber (1) in which the temperature was maintained at  $98 \pm 0.3^\circ\text{C}$ . The depth to which the vessel (2) was immersed into the working fluid, polydimethyl siloxane PMS-400 (made by Kremnepolimer Joint-Stock Company, Zaporozhe, Ukraine) in the chamber (1) was constant. The liquid level in the vessel (2) was automatically kept constant (a clearance 10–12 mm between the film specimen (4) and a fluid surface in the vessel (2) was maintained). This ensured a constant temperature in the specimen during testing, which was  $96 \pm 1^\circ\text{C}$ . Surfaces of the specimen being in a contact with air were exposed to UV-radiation,  $\lambda = 240\text{--}320 \mu\text{m}$ ; radiation power was 240 W. Water (or polyisobutyl alcohol) does enter the specimen (4) as a result of absorption from steam, created due to evaporation of these liquids.

### 2.3. Characterisation

The films having been exposed in liquids or to UV-radiation and steam were analysed by the IR-spectroscopy using a spectrophotometer (M-80 Karl Zeiss, Jena, Germany). The optical density values of the absorption bands at  $1720 \text{ cm}^{-1}$  from carbonyl groups were determined at a known of film thickness. Subsequently, they were used to find their concentrations. Here, the coefficient of extinction was taken [15] equal to  $514 \text{ cm}^3/(\text{mol cm})$ .

To examine the propagation front of oxidation through the polymer, LDPE films having thickness  $400 \mu\text{m}$  were put into hot water and kept there for pre-set periods. Then the

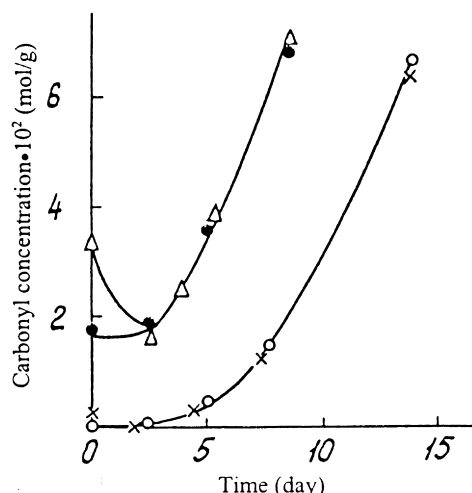


Fig. 2. Oxidation in water of LDPE (O), LDPE extracted with acetone (X), LDPE-g-IA ( $\Delta$ ), and LDPE-g-IA extracted with ethanol ( $\bullet$ ). The film thickness was 40  $\mu\text{m}$ , temperature 96°C.

films were removed and dried in a vacuum; the surface layers were removed by microtoming and used to prepare film specimens of 300  $\mu\text{m}$  thick by compression moulding to analyse them subsequently by IR-spectroscopy technique. The crystallinity of the film specimens was determined using DSC (model DSM-3A, made by Institute for Biological Instruments of Russian Academy of Sciences, and operated at the scanning rate of 16°C/min).

To learn the adhesiveness of LDPE after the films had been kept in hot water, they were used to make adhesion joints with the steel foil (50  $\mu\text{m}$  thick, carbon content 0.08 wt.%). The procedure for metal surface preparation and evaluation of adhesive strength are described elsewhere [16]. The substrate was put in a contact with the polymer coating in air at 190°C for 5 min. Under these conditions high adhesive strength values could be provided at joints of LDPE with steel [17].

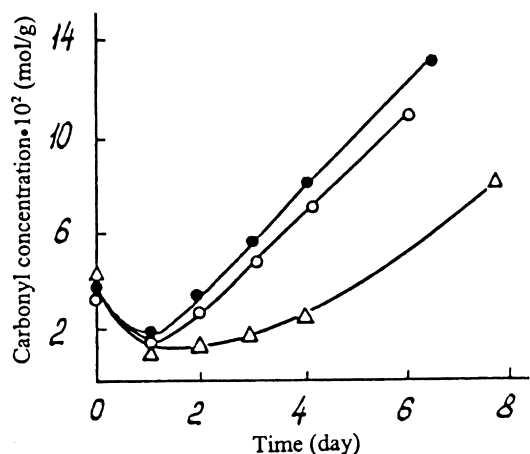


Fig. 3. Oxidation of LDPE-g-IA films in different aqueous media: ( $\Delta$ ) 0.001 N  $\text{H}_2\text{SO}_4$ ; (O) 0.0001 N KOH; ( $\bullet$ ) 0.001 N KOH. Time in days (1 day = 24 h). The film thickness and temperature as for Fig. 2.

### 3. Results

#### 3.1. Thermal oxidation in aqueous medium

The oxidation rate of the virgin LDPE is lower (Fig. 2) than that of the polyethylene modified by grafting of carboxyl groups (LDPE-g-IA). The extraction of LDPE in acetone and LDPE-g-IA with ethanol did not influence their oxidation rate. There is a minimum on the kinetics curve of the sample of LDPE-g-IA, which had not been subjected to extraction. The descending branch characterises the removal of ungrafted IA from the film specimens by a diffusion mechanism; this portion of ungrafted IA is soluble in water [13,14]. The ascending branch shows the accumulation of carbonyl groups in the polymer from oxidation in water.

The oxidation rate of polyolefins in water depends strongly on pH [6], namely, oxidation proceeds faster in an alkaline medium and is slower in an acidic medium. It was of interest, therefore, to understand the effect of the medium on LDPE-g-IA oxidation. It was established here similar behaviour of LDPE-g-IA in alkaline media. In acidic media the oxidation rate of this polymer was in fact the same as in inert media (Figs. 2 and 3).

If the temperature of water was lowered from 96°C (Fig. 2) to 50°C (Fig. 4), a longer time was required to start oxidation of LDPE-g-IA, as registered by IR-spectroscopy the induction time was from 2 days up to 15 days. Exposure to ethanol, in contrast to water, did not lead to LDPE oxidation (Fig. 4).

Introduction of antioxidant hydroquinone [12,18] also prevented LDPE from oxidation during the test period. It was observed that oxidation of LDPE decelerated substantially when the open vessel (Fig. 1) was replaced by the sealed vessel into which no oxygen could penetrate from the environment (Fig. 5).

The analyses of propagation of the oxidation front through the specimen thickness in the cases, where the specimens had been exposed under similar thermal conditions in air and water support the conclusion made in Ref. [6] is that the oxidation rate of macromolecules is higher in water than in air. Moreover, it is believed that the front of intensive oxidation in water propagates deeper into the polymer bulk, than in air (Fig. 6).

The observations that in water mostly surface layers of the film specimens undergo oxidation are illustrated by data in Table 1. It can be noticed that the average concentration of oxygen-containing groups in the specimens decreases with increasing thickness under identical oxidation conditions. This finding suggests that the oxidation process of LDPE be governed by the oxygen diffusion from the aqueous medium to the specimen.

In another study [6], PE oxidation in aqueous media was resulted in deteriorated mechanical characteristics. The results of our investigation of the oxidation in water at 96°C on the LDPE adhesiveness are shown in Fig. 7. It

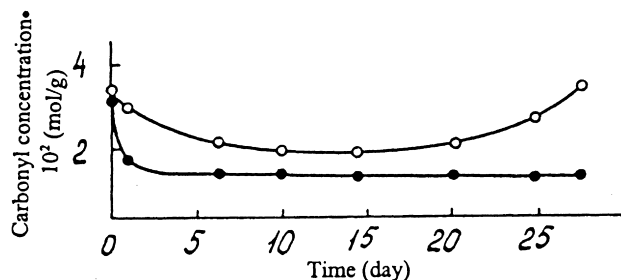


Fig. 4. Concentration of carbonyl groups in LDPE-g-IA films exposed in water (O) and ethanol (●) at 50°C. The film thickness as for Fig. 2.

can be seen that the relationship between the adhesive strength and oxidation period in water of joined systems with steel is characterised by a maximum. Similar relationships were obtained when adhesion joined systems of PE with steel were prepared in air [16].

### 3.2. Photo-oxidation in the presence of water

Maximum oxidation rates (Fig. 8(a) and (b)) characterised those specimens, which were simultaneously treated by UV-radiation and steam. So water accelerated oxidation of LDPE under UV-radiation as well. However, the kinetics curves, describing oxidation under UV-radiation in air and in steam do not differ much (Fig. 8(a)) without the light treatment [6]. This means that UV-radiation is much more efficient than water in accelerating the thermal oxidation of LDPE. Oxidation of LDPE in steam under radiation goes without an induction period. About 100 h was required for the induction period of oxidation to proceed without UV-radiation. The films oxidised without UV-radiation, but completely immersed in water, underwent more intensive oxidation than the films treated with water from steam only on one side. The latter fact is probably due to the lower concentration of water in the film's bulk for one-sided diffusion of it from steam into the polymer.

The LDPE-g-IA films were oxidised at faster rates than those of virgin LDPE were, whether or not they were subjected to UV-radiation (Fig. 8(b) and (a)). The comparison of the oxidation kinetics curves in Fig. 8(a) and (b) shows that accelerated oxidation is more pronounced in

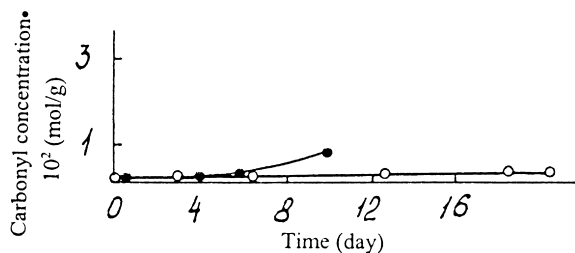


Fig. 5. Oxidation of LDPE films in an open vessel with water (●); in an open vessel with 0.3 wt.% hydroquinone aqueous solution (O). The film thickness was 30 μm; temperature 96°C.

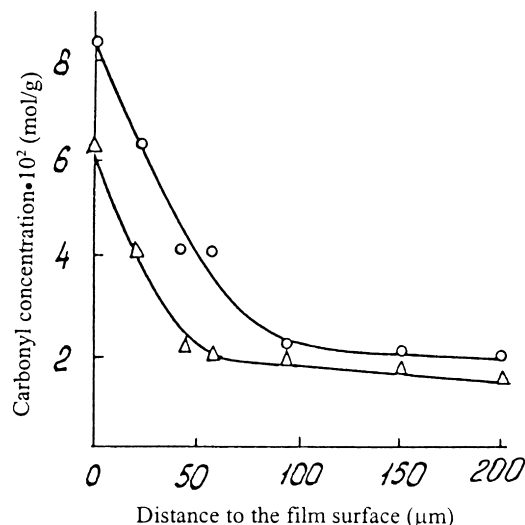


Fig. 6. Propagation of oxidation front across the LDPE film thickness aged in water (O) and air (Δ). The film thickness was 400 μm; temperature 96°C; duration of exposure 14 days.

LDPE-g-IA without radiation treatment than in the virgin LDPE.

Fig. 9 shows the role of the light stabiliser (Benzene OA) and its mixtures with the antioxidant (Irganox 1010) in the oxidation kinetics of LDPE in steam under UV-irradiation. When compared, the data in Figs. 8(a) and 9 and show the stabiliser's inefficiency here. The mixture of the light stabiliser and antioxidant could retard oxidation only a little at the initial stage (Fig. 9). Addition of Benzene OA did not retard oxidation, but it even made the process go faster for LDPE exposed in steam (Fig. 8(a)). The most probable cause of a low efficiency of Benzene OA during photo-oxidation in steam is its extraction from the film during exposure [6,12]. Washing out of Benzene OA by steam condensing on the LDPE films can be supported by data in Fig. 10. Reducing concentration of the light stabiliser is indicated by the lowered optical density of the absorption band of aryl nuclei at 1625 cm<sup>-1</sup> [19]. Extraction of the stabiliser leads to more porous film specimens; as the result, they adsorb steam more easily. Increasing the content of water in the polymer volume was believed to intensify oxidation of the stabilised specimens compared with the virgin ones (Figs. 8(a) and 9).

Table 1

Concentration of carbonyl groups 10<sup>2</sup> (mol g<sup>-1</sup>) during oxidation of samples in water at 96°C

Thickness of samples, μm	Aging time in water, days				
	1	2	4	6	8
30	1.07	1.11	4.13	11.11	18.12
90	0.32	0.40	1.22	3.09	5.40
120	0.23	0.34	0.82	1.92	3.45
245	0.19	0.23	0.72	1.98	2.65
320	0.13	0.22	0.25	0.72	0.99

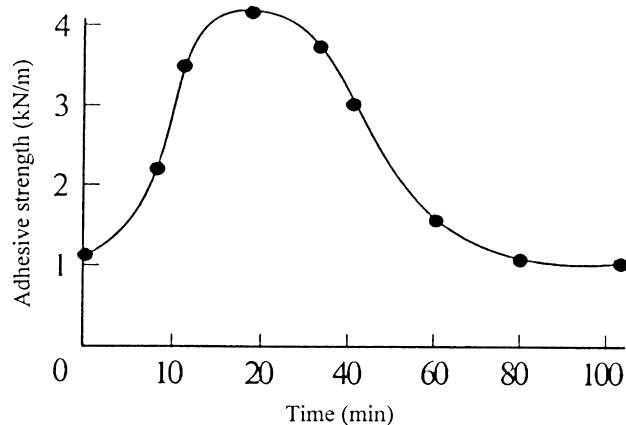


Fig. 7. Effect of oxidation period on adhesive strength of the junctions of LDPE film-steel systems aged in water. The film thickness was 300  $\mu\text{m}$ ; temperature 96°C.

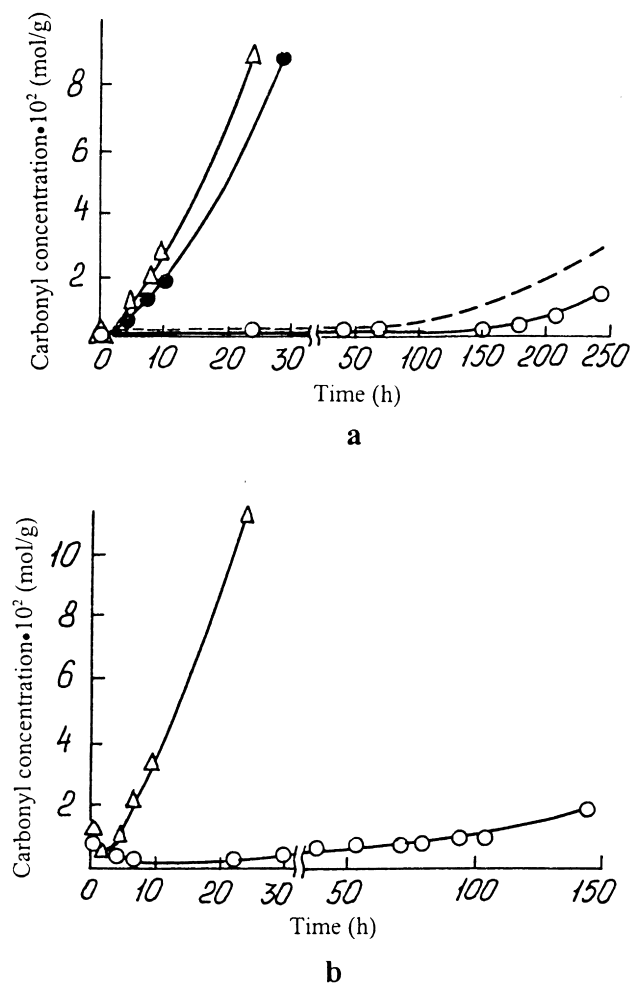


Fig. 8. Oxidation of LDPE (a) and LDPE-g-IA (b) films in steam under effect of UV-radiation ( $\Delta$ ) and without it ( $\circ$ ). Dash line stands (a) for oxidation of films in water at 96°C following conditions from Fig. 2 ( $\circ$ );  $\bullet$  (a)-oxidation in air under effect of UV-radiation.

Fig. 11 shows that photo-oxidation of films in isopropanol vapour retards compared with photo-oxidation in steam (Fig. 8(a)).

#### 4. Discussion

The LDPE can, probably, be oxidised in liquids by oxygen from the air being absorbed by the polymer or dissolved in the liquid medium and diffused from it into the polymer. Retarded oxidation of the specimens kept in the sealed vessel (Fig. 4), unlike that in open vessels (Fig. 2), points to a determinative role of oxygen entering the specimens from the aqueous media. The air dissolution in water at 20°C is 0.0187  $\text{cm}^3$  of air per  $\text{cm}^3$  of water [20] and decreases with a temperature increase. When a gas dissolves in polymers, as characterised by the solubility factor, this increases with temperature. It is due to the gas permeability for amorphous polymers or the amorphous phase of crystallizable polymers is generally determined by the mode of changing in the diffusivity with temperature [3]. About this, we believe that facilitated diffusion of water and related increased concentration of oxygen transported by water within the polymer are responsible for accelerated oxidation of LDPE at elevated temperatures of aqueous media. We do not think that water sorbed by the polymer would increase the reactivity toward oxygen. Rather the opposite may be true [21], as water adsorbed by polyolefins leads to antiplasticization, i.e. to lower segmental mobility of polymer chains macromolecules in the presence of water molecules. Due to this, one can expect that diffusion of oxygen from water will be reduced. As the result, oxygen concentration (one of the reagents) will be lowered, what results in lower rate of oxidation process.

Similar behaviour of the oxidation process for LDPE-g-IA immersed in water and solution of sulphuric acid (Figs. 2 and 3, respectively) can be explained by an extremely low permeability of this electrolyte into hydrophobic polymers. As the result, it is probably that mostly water diffuses into the polymer bulk [2].

Alkalis also hardly diffuse into the polyolefins' bulk. The principles of their transfer have been investigated inadequately [2]. In a general case, it is believed that polyolefins above the glass-transition temperature do not in fact contain pores whose radii exceed 1 nm. Therefore, the mass transfer follows the mechanism of activated diffusion (different types of defects that can be present in almost any polymer samples were not taken into consideration). During the sorption process polar water molecules form clusters in the matrix of hydrophobic polymers, as cohesive forces in liquids are higher than interaction forces between the diffusing molecules and the polymer. As water forms a continuous medium in clusters, probably the electrolyte (dissolved in it) is in a dissociated state. Therefore, in an alkaline medium LDPE may undergo accelerated oxidation caused by catalysis owing to acid–base interactions of the reagents, which

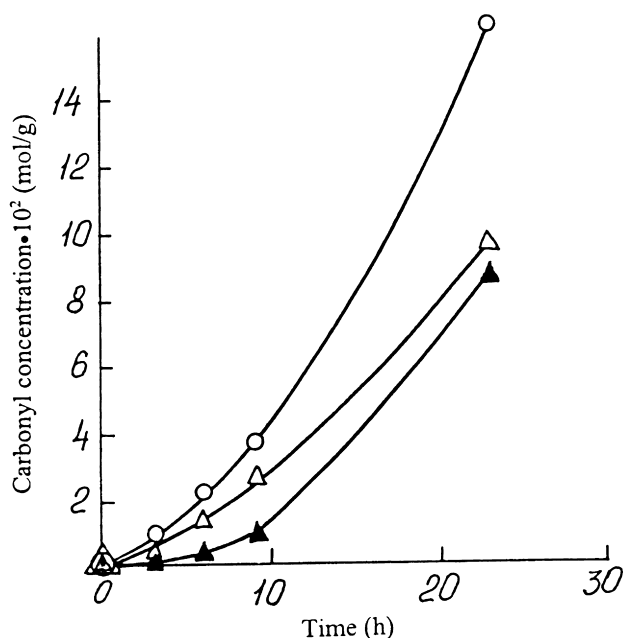


Fig. 9. Oxidation of LDPE containing Benzene OA 0.3 wt.% (○) and 0.5 wt.% (△); LDPE containing a mixture of 0.5 wt.% Benzene OA and 0.3 wt.% Irganox-1010 (▲) in steam under UV-radiation.

can activate the processes that follow the radical mechanism [22]. Along with this, a sodium carboxylate can diffuse within the film from the surface layer where they are formed by the reaction of a sodium hydroxide with carboxylic acids being the oxidation products of LDPE.

In another study [4] LDPE had been subjected to long-lasting oxidation in aqueous-alkaline solutions. Here absorption bands with a maximum at  $1580\text{ cm}^{-1}$  were detected, which are related to carboxylate ions. This supports our suggestion about penetration of a hydroxide into the polymer, which is consumed by interaction with carboxyl groups. The accelerating effect of salts of organic

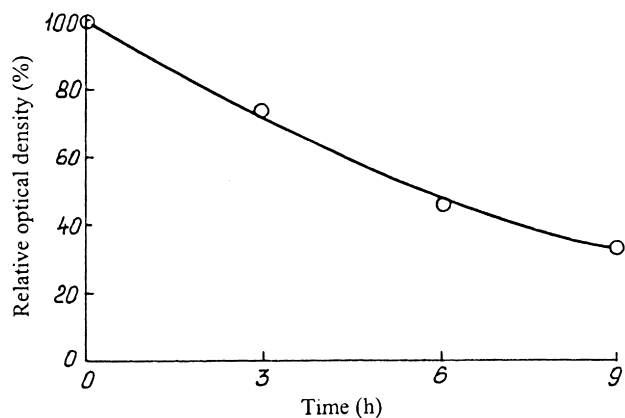


Fig. 10. Dependence of optical density of the  $1625\text{ cm}^{-1}$  absorption band on oxidation period for LDPE containing 0.5 wt.% Benzene OA, in steam under UV-radiation.

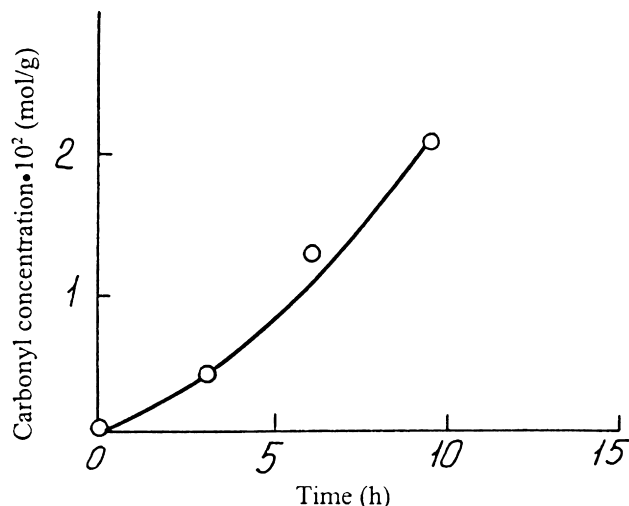


Fig. 11. Oxidation of LDPE films in *iso*-butyl alcohol vapor under UV-radiation.

acids on polyolefins oxidation had been described earlier [23].

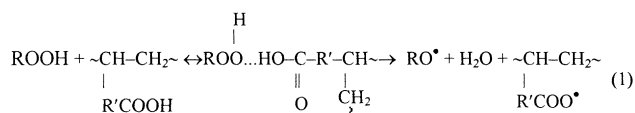
The lack of oxidation in ethanol (Fig. 4) can be explained by the following: first, the adsorbed oxygen was consumed to oxidise ethanol [24]; second, the mechanism of the gas permeability in ethanol was different (during air sorption from the alcohol at adsorption–desorption acts, the molecular contact between the sorbed oxygen and active sites of the macromolecules, required to initiate the chemical reaction, did not accrue).

The following could explain the accelerated oxidation of LDPE-*g*-IA as compared with the virgin LDPE in water (Fig. 2). First, grafted carboxyl groups on the macromolecules led to a higher polarity of the polyolefins and its higher sorption capacity toward water; so, to a higher oxygen concentration in the test specimens. Second, it is known [24] that carboxylic acids can accelerate decomposition of ROOH hydroperoxides that get accumulated in the polymer at the stage where self-oxidation starts, participating in the reaction with them.

Another phenomenon might be responsible for the accelerated oxidation of LDPE-*g*-IA, viz., decrease crystallinity of the film samples, as it is known [3] that oxygen dissolves only in the amorphous phase. However, DSC observations showed that LDPE and LDPE-*g*-IA test films had approximately similar crystallinity. The melting heat was  $27.3\text{ J/g}$  for LDPE and  $27.1\text{ J/g}$  for LDPE-*g*-IA. Therefore, changes in the physical structure caused by grafting should not influence the oxidation kinetics.

Photo-oxidation of LDPE carried out in the presence of water and *iso*-butanol (Figs. 8(a) and 11) showed that UV-radiation was mostly responsible for the oxidation kinetics.

Photo-oxidation of macromolecules taking place at relatively low temperatures was shown to have led to a high amount of hydroperoxides accumulated in the polymer [12]. These lowered the resistance of the polymer to the thermal



Scheme 1.

oxidative ageing. Activated decomposition of the hydroperoxides could accelerate the effect of water under a photo-initiated oxidation of the macromolecules. This was mentioned in Ref. [6], which describes model experiments on the thermal decomposition of *tert*-butyl hydroperoxide in water. It was believed that accelerated decomposition of the hydroperoxide in water was associated with the thermal nucleophilic attack of the peroxide anions formed through the hydroperoxide dissociation in water. Hydroperoxides are acidic, having a  $\text{pK} = 10\text{--}12$  depending on their structure [25]. Therefore, in aqueous solutions they can be as anion peroxide of polyethylene  $\text{PE}-\text{OO}^-$  being a strong nucleophilic substance. This hypothesis was supported by the results of the present work dealing with accelerated thermal and photo-oxidation of LDPE-*g*-IA as compared with LDPE in aqueous media, namely, the grafted carboxyl groups accelerated decomposition of macromolecular hydroperoxides, e.g. as in Scheme (1). Therefore, accelerated decomposition of macromolecular hydroperoxides under the influence of grafted carboxyl groups can be one cause [12]. In alcohols, the ionic dissociation of hydroperoxides is hindered which excludes nucleophilic attack by peroxyanion of the hydroperoxides accumulated during photo-oxidation, and thus does not allow the reaction of chain self-oxidation to develop.

These findings show that resistance to thermal or atmospheric ageing in the presence of water as liquid or steam can be achieved by using light-and-thermal stabilisers or aqueous media containing dissolved stabilisers. The results obtained must be also considered when chemically analysing the behaviour of polyolefins, modified systems based on them and compositions in aqueous media, e.g. when determining the grafting efficiency of oxygen-containing functional groups onto macromolecules [13,14].

## 5. Conclusions

Functionalization of LDPE by grafting of itaconic acid to the macromolecules was found to accelerate its thermal and photo-oxidation in water. The LDPE-*g*-IA underwent accelerated oxidation in alkaline media whereas no essential changes were detected during testing in acidic media. Under identical thermal conditions films of LDPE would oxidise in water to a greater depth than does in air. Aqueous media containing dissolved stabilisers appear rather efficient. The data suggest that the main cause of the accelerated oxidation of both the virgin and functionalised LDPE in water is a decomposition of macromolecular hydroperox-

ides initiated by water. These hydroperoxides are formed at the initial stage of the oxygen interaction with the macromolecules.

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