Результаты литературного обзора показывают, что микроволновое выщелачивание является перспективным направлением благодаря энергоэффективности, ускорению кинетики реакций, повышению степени перехода алюминия в раствор и снижению экологической нагрузки. Кроме того, в работе определены основные научные преимущества данной технологии и ключевые направления её дальнейшего развития, включая глубину проникновения микроволн и оптимизацию режимов мощности и температуры.

Таким образом, данная обзорная работа систематизирует научные основы применения микроволновых технологий в переработке золошлаковых отходов и способствует разработке новых экологически безопасных методов эффективного извлечения алюминия из отходов.

Ключевые слова: *золошлаковые отходы, оксид алюминия, графит, электромагнитные технологии, алюминий, микроволновая обработка, выщелачивание, химическая термодинамика.*

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RESEARCH OF THE EFFECT OF UV-IRRADIATION ON UNSATURATED POLYESTER RESINS OBTAINED IN THE PROCESS OF «COLD» CURING WITH ACRYLIC ACID

Annotation: *The investigation of the influence of UV irradiation on polymers represents an important and relevant area of science and technology, as it enables the development of new approaches for designing more durable and efficient materials. This work examines the changes occurring in polymers based on unsaturated polyesters under prolonged exposure to ultraviolet radiation. The objective of the study was to evaluate the effect of long-term UV irradiation on the thermal stability of polymeric materials based on polyethylene glycol maleate and polypropylene glycol maleate copolymerized with acrylic acid at a component*

ratio of approximately 70:30 wt.%. The samples were examined using microscopy to identify microcracks formed as a result of UV exposure. In addition, visual assessment of color variation and the appearance of turbidity in the studied materials was performed. Additionally, the carbonyl index was determined to quantitatively evaluate the degree of photooxidative degradation of the polymers. This parameter characterizes the accumulation of carbonyl groups formed as a result of polymer chain scission under UV irradiation and allows estimation of the extent of photodegradation processes in the materials. The obtained results demonstrate that the polymer synthesized on the basis of polyethylene glycol maleate exhibited a more pronounced decrease in the onset temperature of thermal deformation after UV exposure compared with the copolymer based on polypropylene glycol maleate, indicating higher resistance of the latter to UV-induced degradation. The thermal stability of the copolymers was evaluated by thermogravimetric analysis through determination of the onset temperature of thermal deformation before and after 21 days of UV irradiation.

Key words: *UV-irradiation, thermal deformation, thermal stability, copolymerization, «cold» curing, unsaturated polyesters, polyethylene glycol maleate, polypropylene glycol maleate.*

Introduction

With the advancement in technology and increasing use of polymeric materials in various industries such as automotive, construction, medical and packaging, special attention has been paid to the durability and resilience of these materials [1-3]. Polymers have carved a significant niche in the global market due to their versatility, lightness and low cost, however, their performance is largely dependent on the effects of various environmental factors [4]. One of the most damaging effects on polymers is ultraviolet (UV) radiation, which is a portion of the solar radiation spectrum. UV-rays can significantly affect the structure and properties of polymeric materials, leading to their degradation.

UV-irradiation causes photochemical reactions in the molecular structure of polymers, which can lead to the breaking of chemical bonds, formation of free radicals, and degradation of polymer chains. These processes can affect many physical characteristics of materials, including strength, elasticity, transparency and resistance to external aggressive influences such as moisture, temperature and chemicals [5, 6]. The effect of UV-radiation is particularly noticeable on polymers that are exposed to prolonged sunlight, such as in exterior coatings, product packaging, medical and construction materials. Polymers can lose their original characteristics when exposed to UV-radiation, which limits their service life and reduces the cost-effectiveness of their use [7].

However, the study of the effects of UV-radiation on polymers provides valuable information for the development of new sustainable materials as well as methods to protect polymers from UV-radiation. Current research is aimed at improving the UV-stability of polymeric materials through the use of various stabilizers, UV-absorbers, and the introduction of innovative polymer compositions with improved properties [8]. In addition, UV-radiation is used not only as a destruction factor, but also as a tool to improve the properties of polymers, for example, in the curing process of some types of coatings and resins [9].

Thus, the results of the study can be used to further optimize the composition of such polymers in order to increase their resistance to UV-exposure, which will open new opportunities for their use in conditions of prolonged solar irradiation.

This work is devoted to the investigation of prolonged exposure to UV-irradiation on the thermal stability of polymers based on unsaturated polyesters, polyethylene glycol maleate (p-EGM) and polypropylene glycol maleate (p-PGM) with acrylic acid (AA).

Materials and Methods

Reagents used in this work: ethylene glycol, propylene glycol, maleic anhydride, acrylic acid, benzoyl peroxide, dimethylaniline («Sigma-Aldrich»), aluminum chloride («Reachim»). All reagents were used without additional purification.

Initial polyesters – polyethylene glycol maleate and polypropylene glycol maleate – were obtained by polycondensation reaction of ethylene glycol and propylene glycol, respectively, with maleic anhydride according to the method [10, 11] at the temperature of 150-160°C. Aluminum chloride was used as a catalyst.

«Cold» curing of solutions of the obtained unsaturated polyesters in acrylic acid of ~70:30 wt.% composition was carried out using an optimized binary initiating system consisting of benzoyl peroxide (PB) and dimethylaniline (DMA) (ratio of components 1:0.15 wt.% of the mass of the initial solution). The curing was carried out at 20°C [12, 13].

The reaction products were identified by IR spectroscopy. Analysis of the obtained IR spectra showed the presence of characteristic bands in the range of 1570-1590 cm^{-1} (1572 cm^{-1} and 1583 cm^{-1}), indicating the presence of unsaturated double bonds of p-EGM and p-PGM, which did not

react. Both IR-spectra of the copolymers of p-EGM and p-PGM with AA exhibited absorption bands with peaks at 1718 cm^{-1} and 3432 cm^{-1}, confirming the presence of –COOH carboxyl groups. Peaks at 2846 cm^{-1} and 2918 cm^{-1} were also detected, which is characteristic of the methylene groups of p-PGM.

Next, the effect of UV-irradiation on samples of binary systems of p-EGM and p-PGM with AA in the ratio of ~70:30 wt.% was investigated. The experiment was carried out in accordance with EN 1297:2004, MOD (GOST 32317-2012) using Weiss UV3 instrument of Weiss Technik (Germany) [14]. This device is a closed chamber for observation, providing irradiation of samples with ultraviolet light with wavelengths 290 and 450 nm [15].

For the experiment, plates of different thicknesses from 1 to 5 mm were made (three samples of each thickness for parallel experiments, which allowed to ensure the reliability of the results. The irradiation intensity was kept constant and the experiment itself was continued for three weeks (21 days). The condition of the specimens after irradiation was assessed visually, focusing on color changes and the appearance of microcracks.

To quantitatively assess the degree of photooxidative degradation, the carbonyl index (CI) was calculated, defined as the ratio of the area of the carbonyl group band (1710-1720 cm^{-1}) to the area of the reference band (e.g., deformation vibrations of methylene groups in the 1450 cm^{-1} region):

$$CI = \frac{A_{1710-1720}}{A_{1450}} \quad (1)$$

where A is the area of the corresponding peak.

To evaluate the thermal stability of the samples, their thermograms were analyzed by determining the thermal deformation temperature in the range from 30 to 200°C. The process of complete decomposition of copolymers was recorded at a temperature of about 550-600°C *on a device for the synchronic thermal analysis* Labsys Evolution TG-DTA/DSC («Setaram») in dynamic regimen [16].

Results and Discussion

Nowadays, polymeric materials play an important role in almost all areas of industry. The exploitation of polymers includes their use in different environments, depending on their purpose. Thus, polymers can be used in a wide variety of applications such as construction, packaging, medicine, electronics and automotive. During their use, they are subjected to various influences that can affect their mechanical, thermal, chemical and physical properties. Polymer aging is a set of processes occurring in a material that cause deterioration of its properties over time. One of the main factors affecting polymer aging is UV-radiation and temperature. In particular, UV-exposure leads to polymer chain breakage, color change, loss of strength and elasticity (photoaging), and high temperature accelerates degradation processes, for example, thermal aging causes changes in the polymer structure, which can lead to its brittleness or softening.

Taking into account that photo-aging is accompanied by disruption of polymer structure, appearance of cracks (surface and in the thickness of the material), surface opacity, etc., reduction of mechanical characteristics of materials, the influence of UV-rays on polymeric materials should be taken into account in the calculation of building structures or other polymer products by appropriate coefficients of working conditions [17].

In double-reactive bonded polymers, aging is usually manifested by two types of processes: degradation and cross-linking of macromolecules. The degradation process caused by UV-irradiation is called «photodegradation» [15].

To determine the effect of UV-exposure on copolymers of p-EGM and p-PGM with AA of ~70:30 wt.% ratio, we performed visual evaluation of the samples to detect color changes and appearance of turbidity. Further, microscopic analysis was performed to detect microcracks on the surface of the samples due to irradiation. The results obtained by visual and microscopic analysis are presented in Table 1.

Based on the experimental data obtained, it was found that as a result of prolonged exposure of the analyzed binary system of p-PGM with AA to UV radiation, all samples, regardless of thickness, acquired a yellowish tint. However, the surfaces did not tarnish or become matte. Also, the color changes were more pronounced in plates of smaller thickness (1-2 mm) compared to thicker samples. Although polymers are often subjected to cracking or degradation under UV exposure, in this case microscopic analysis did not show the appearance of microcracks or other

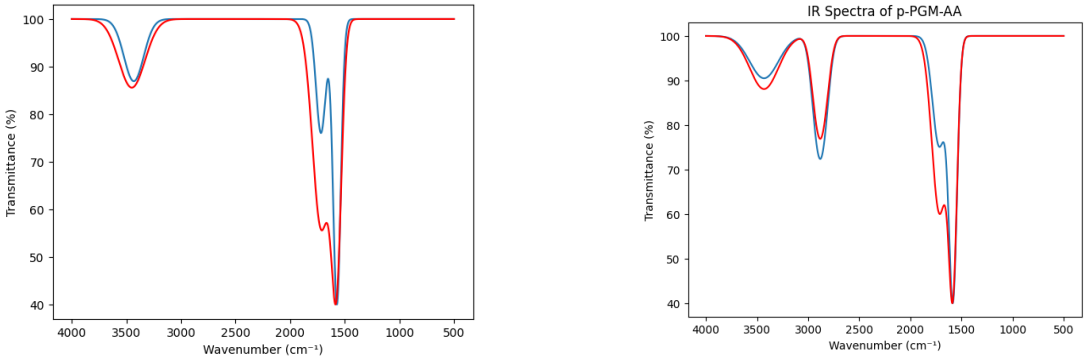
deformations on the surface of the samples. No visual changes in color, appearance of bubbles or turbidity were recorded for the p-EGM samples with AA, and microcracks were also absent on their surface.

Table 1 – Effect of UV-irradiation on copolymers of p-EGM and p-PGM with AA of ~70:30 wt.% composition (after exposure to UV-radiation for 21 days)

Unsaturated polyester	Co-curing agent	Color change, turbidity	Presence of microcracks, bubbles	Other changes
p-EGM	AA	–	–	Slight hardening, loss of flexibility
p-PGM		Slight yellowish tint	–	Slight hardening, loss of flexibility

After UV irradiation, significant changes were observed in the IR spectra of copolymers in the carbonyl group absorption region. For the p-EGM–AA copolymer, an increase in the intensity and area of the absorption band in the range 1710–1720 cm^{-1} was noted, indicating an increase in the concentration of carbonyl fragments formed as a result of photooxidative destruction of the macromolecular chain.

In the case of the p-PGM–AA copolymer, an increase in the intensity of the carbonyl band in the range 1710-1720 cm^{-1} was also observed after UV irradiation. However, the appearance of new absorption bands in the 1680-1690 cm^{-1} region was also recorded, caused by the formation of conjugated carbonyl structures (chromophores). The formation of these structures was accompanied by visual yellowing of the samples, which is characteristic of photoaging processes involving conjugated systems (Figure 1).



a b

Figure 1 – IR spectra of p-EGM and p-PGM with AA copolymers
a – p-EGM–AA 68.94:31.06 wt.%; b – p-PGM–AA 68.65:31.35 wt.%

The calculation of the carbonyl index showed that for the p-EGM–AA copolymer, this indicator increased from 0.82 to 1.46 after UV irradiation, while for the p-PGM–AA copolymer, a less pronounced increase was observed – from 0.79 to 1.18. The more significant increase in CI for p-EGM–AA indicates a more intense photo-oxidative degradation process.

The results of IR spectroscopy are consistent with the data from thermogravimetric analysis.

Thus, instrumental analysis of the thermal stability of the binary systems of p-EGM and p-PGM with AA included thermogravimetric analysis (TGA) to determine the temperature of the onset of thermal deformation before and after exposure to UV-radiation for 21 days. The thermogram results showed that the thermal stability of p-EGM–AA decreased slightly after UV-irradiation. While there was almost no change in the onset temperature of thermal deformation for the p-PGM–AA system (Figure 2).

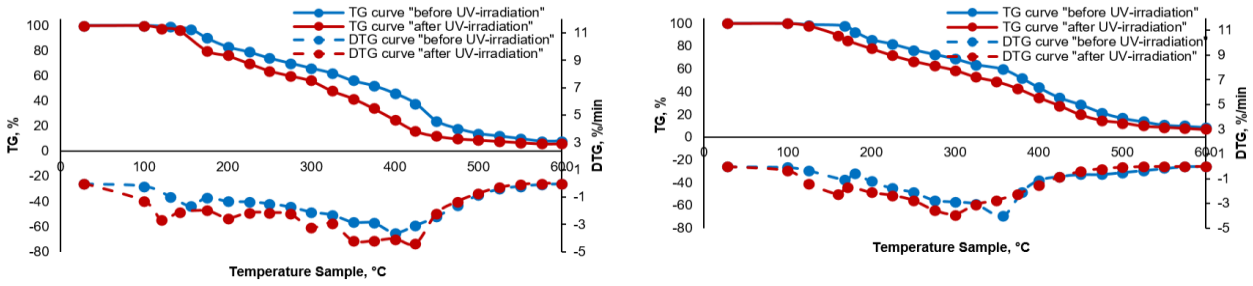


Figure 2 – TG curves of p-EGM and p-PGM with AA copolymers at heating rate of 10°C/min before and after UV-irradiation:
a – p-EGM–AA 68.94:31.06 wt.%; b – p-PGM–AA 68.65:31.35 wt.%

Thus, the onset temperature of thermal decomposition of the p-EGM–AA copolymer before irradiation was 156°C, and after UV-irradiation it slightly decreased to 141°C (Figure 2 a). For the p-PGM–AA binary system, the onset temperature of thermal decomposition before and after irradiation was 168°C and 159°C (Figure 2 b).

Thus, based on the obtained data, it should be noted that exposure to UV-rays, even for a long time, does not significantly affect the onset temperature of thermal deformation. However, with respect to the binary system p-PGM–AA, it should be noted that its application in the production of polymeric materials may be limited due to its color change as a result of exposure to UV-rays. In comparison with the above-mentioned binary system p-PGM–AA, the copolymers of p-EGM with AA showed better performance, including a fairly good thermal stability after UV-irradiation. The obtained results make it possible to speak about the preferability of using the binary system p-EGM–AA as a polymer base in the production of materials for various purposes.

Conclusions

The studies of thermal stability as a result of prolonged exposure to UV rays on binary systems of p-EGM and p-PGM with AA of ~70:30 wt.% composition have shown high thermal stability of copolymers of these unsaturated polyesters after prolonged UV-irradiation, which reveals broad prospects for their use as special-purpose materials.

The negative consequences of UV-irradiation on the copolymers of p-PGM with AA should be attributed to the appearance of yellowness, which may limit their use in the manufacture of products from the polymer, in which the importance is given to the color performance of the finished product.

Thus, as a result of the conducted experiment it is possible to draw a conclusion about the preferability of choosing the binary system of p-EGM with AA for obtaining polymeric materials for various purposes on their basis.

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ИССЛЕДОВАНИЕ ВЛИЯНИЯ УФ-ОБЛУЧЕНИЯ НА НЕНАСЫЩЕННЫЕ ПОЛИЭФИРНЫЕ СМОЛЫ, ПОЛУЧЕННЫЕ В ПРОЦЕССЕ «ХОЛОДНОГО» ОТВЕРЖДЕНИЯ АКРИЛОВОЙ КИСЛОТОЙ

Изучение влияния ультрафиолетового облучения на полимеры является важным и актуальным направлением науки и техники, поскольку позволяет разрабатывать новые подходы к созданию более долговечных и эффективных материалов. В данной работе рассматриваются изменения, происходящие в полимерах на основе ненасыщенных полиэфиров при длительном воздействии ультрафиолетового излучения. Целью исследования являлось изучение влияния продолжительного УФ-облучения на термическую стабильность полимерных материалов на основе полиэтиленгликольмалеината и полипропиленгликольмалеината, отвержденных акриловой кислотой при соотношении компонентов ~70:30 масс.%. Образцы исследовались методом микроскопии для выявления микротрещин, возникающих в результате воздействия УФ-излучения. Дополнительно проводилась визуальная оценка изменения окраски и появления мутности исследуемых материалов. Дополнительно для количественной оценки степени фотоокислительной деградации полимеров определялся карбонильный индекс. Данный показатель характеризует накопление карбонильных групп, образующихся в результате разрушения

полимерной цепи под действием ультрафиолетового излучения, и позволяет оценить глубину протекания процессов фотостарения материалов. Термическая стабильность сополимеров определялась методом термогравиметрического анализа путем установления температуры начала термической деформации до и после УФ-облучения образцов в течение 21 суток. Полученные результаты показали, что полимер, синтезированный на основе полиэтиленгликольмалеината, демонстрирует более выраженное снижение температуры начала термической деформации после УФ-облучения в сравнении с сополимером на основе полипропиленгликольмалеината, что свидетельствует о более высокой устойчивости последнего к деградации, обусловленной УФ-облучением.

Ключевые слова: *УФ-облучение, тепловая деформация, термическая стабильность, сополимеризация, «холодное» отверждение, ненасыщенные полиэфиры, полиэтиленгликольмалеинат, полипропиленгликольмалеинат.*

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АКРИЛ ҚЫШҚЫЛЫМЕН «СУЫҚ» ҚАТАЮ ПРОЦЕСІНДЕ АЛЫНҒАН ҚАНЫҚПАҒАН ПОЛИЭФИРЛІ ШАЙЫРЛАРҒА УК-СӘУЛЕЛЕНУДІҢ ӘСЕРІН ЗЕРТТЕУ

Полимерлерге ультракүлгін сәулеленудің әсерін зерттеу ғылым мен техника саласындағы маңызды әрі өзекті бағыт болып табылады, себебі ол неғұрлым берік және тиімді материалдарды жасауға жаңа тәсілдерді әзірлеуге мүмкіндік береді. Бұл жұмыста ультракүлгін сәулеленудің ұзақ уақыт әсері кезінде қанықпаған полиэфирлер негізіндегі полимерлерде жүретін өзгерістер қарастырылды. Зерттеудің мақсаты – полиэтиленгликоль малеинаты мен полипропиленгликоль малеинаты негізінде акрил қышқылымен шамамен 70:30% массалық қатынаста алынған полимерлік материалдардың ұзақ мерзімді ультракүлгін сәулеленудің әсерінен термиялық тұрақтылығын зерттеу. Үлгілер ультракүлгін сәулеленудің әсерінен пайда болатын микрожарықшаларды анықтау үшін микроскопиялық әдіспен зерттелді. Сонымен қатар зерттелген материалдардың түсінің өзгеруі мен лайлану құбылысы визуалды түрде бағаланды. Сонымен қатар полимерлердің фотооксидтік деградация дәрежесін сандық тұрғыда бағалау мақсатында карбонил индексі анықталды. Бұл көрсеткіш ультракүлгін сәулеленудің әсерінен полимер тізбегінің ыдырауы нәтижесінде түзілетін карбонил топтарының жинақталуын сипаттайды және материалдардың фототозу процесінің тереңдігін бағалауға мүмкіндік береді. Сополимерлердің термиялық тұрақтылығы термогравиметриялық талдау әдісі арқылы, яғни үлгілерді 21 тәулік бойы ультракүлгін сәулеленуге ұшыратқанға дейін және кейін термиялық деформация басталу температурасын анықтау арқылы бағаланды. Алынған нәтижелер полиэтиленгликоль малеинаты негізінде синтезделген полимерде ультракүлгін сәулеленуден кейін термиялық деформация басталу температурасының анағұрлым айқын төмендейтінін көрсетті. Бұл көрсеткіш полипропиленгликоль малеинаты негізіндегі сополимермен салыстырғанда төмен, яғни соңғысы ультракүлгін әсерге анағұрлым жоғары төзімділік көрсетеді.

Түйін сөздер: *УК-сәулелену, жылу деформациясы, термиялық тұрақтылық, сополимерлеу, «суық» қатаю, қанықпаған полиэфирлер, полиэтиленгликолмалеинат, полипропиленгликолмалеинат.*

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INVESTIGATION OF CONVERSION OF N-BUTANE TO LIGHT OLEFINS ON CATALYSTS Pd/γ-Al_2O_3 AND Pd/SiO_2

Abstract: *The results of the conversion of n-butane on supported palladium catalysts in a stationary mode in the temperature range of 400-600 °C, at atmospheric pressure and a volumetric feed rate of n-butane of 250 h^{-1} are presented. The catalysts were obtained by the method of incipient wetness impregnation using γ-Al_2O_3 and SiO_2 (silica gel) as supports, the palladium loading was 1 and 3%. It was found that, along with the dehydrogenation of n-butane, cracking processes occur in parallel, leading to the formation of both olefins (ethylene, propylene) and alkanes (methane, ethane, propane), isomerization of n-butane to isobutane, and other reactions. Hydrogen contained in contact gases is equivalent to the amount of dehydrogenation and degradation products. In trace amounts, non-condensed liquid products such as pentane and aromatics are present in the gas phase. The catalysts were studied by SEM-EDS to determine the morphology of catalysts*