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Investigation of the Influence of UV-Irradiation on Thermal Stability of Binary Systems on the Basis of Polyethylene Glycol Fumarate with Some Vinyl Monomers

This work is devoted to the investigation of continuous exposure to UV-irradiation on thermal stability of polymeric base of sealants. The copolymers of polyethylene glycol fumarate with acrylic and methacrylic acids, and acrylamide of the composition of "unsaturated polyester: vinyl monomer" ~35:65 mass.% (correspondingly) were chosen as the polymer base. The samples were analyzed on microscope to the presence of microcracks as a result of exposure to UV-irradiation on them. The studied samples were evaluated visually on color change and the appearance of turbidity. Thermal stability of the studied polymeric base was determined by establishing the temperature of the start of thermal deformation by thermogravimetry before and after the process of UV-irradiation within 21 days. It was established that for the polymeric base with acrylamide in its content the temperature of the start of thermal deformation had reduced after exposure to UV-irradiation. In contrary, for the polymeric bases with acrylic and methacrylic acids in their composition the above said thermal index almost had not changed after affecting with UV-irradiation on the samples.

Keywords: polymer base, sealants, UV-irradiation, thermal deformation, unsaturated polyesters, polyethylene glycol fumarate.

Introduction

Investigation of the processes of ageing of the materials when influencing various external factors on them is a complex research and practical task and its solution defines the safety and quality parameter of the use of a building [1–2]. When accelerating the process of operation of construction objects there are not only special requirements to the materials, but there is also the task of providing the reliance of their use within long period of time in natural conditions. In this regard it is necessary to know not only the properties of the materials, but also it is important to predict their change under the effect of negative external factors [3–7].

Sealing materials (the sealants) are used in a various area of technique and construction providing working capacity of structural elements in technique, waterproof compartments, also they define the durability of stiches of inter-wall panels, building blocks and so on [8–11]. In comparison with other classes of polymeric stuffing materials the sealants are used not only as the goods ready to use, but in the forms of fluid-flow and paste-like masses. The sealing materials are spread using rather simple technological operations in the areas of stiches, backlashes, junctures and are poured to the cracks. After technological curing time the sealants form relatively hard and elastic substance and drop into resin-like state. The polymeric film formed seal the holes and cracks hermetically and, in some cases, acts as a glue. The sealants are irreplaceable in sinking the sanitaryware products, in filling-in the stitches inside buildings with high level of humidity, where other types of constructional materials become quickly inapplicable. In housebuilding they are used for filling-in external stitches and cracks in foundations, walls, ceilings, for processing the junctures, holes and cracks inside the rooms. In the rooms with high level of humidity they are used for waterproofing the stitches and junctions. They provide the impermeability in the conditions of drops of pressure, changeable temperatures, loads and they are one of the pointers of reliability of thermo-, water- and vapor-proofing. Thus, the stability of sealants defines the consistency of work and durability of sealing sews in various constructions [10, 11].

The most promising compounds for the development of sealing materials are the solutions of unsaturated polyesters in different monomers of vinyl row [12], which preserve needed fluidity in wide range of concentrations. As a result of having reactive double bond the unsaturated polyesters are the most important rep-

resentatives of polymeric reactive oligomers which are able to react with many monomers forming the cured reaction products [13]. High indices of stability to the influence of external medium, simplicity of preparation and operation, good compatibility with other components (mechanical fillers and pigments) and availability, low primecost and ecological safety make the unsaturated polyesters more preferable co-reagent when obtaining a polymeric base of sealing materials.

Considering the characteristics of sealants are defined by the properties of fillers used as well as the initial polymeric base, the study of the behavior of latter after ageing in natural conditions is of an applied significance.

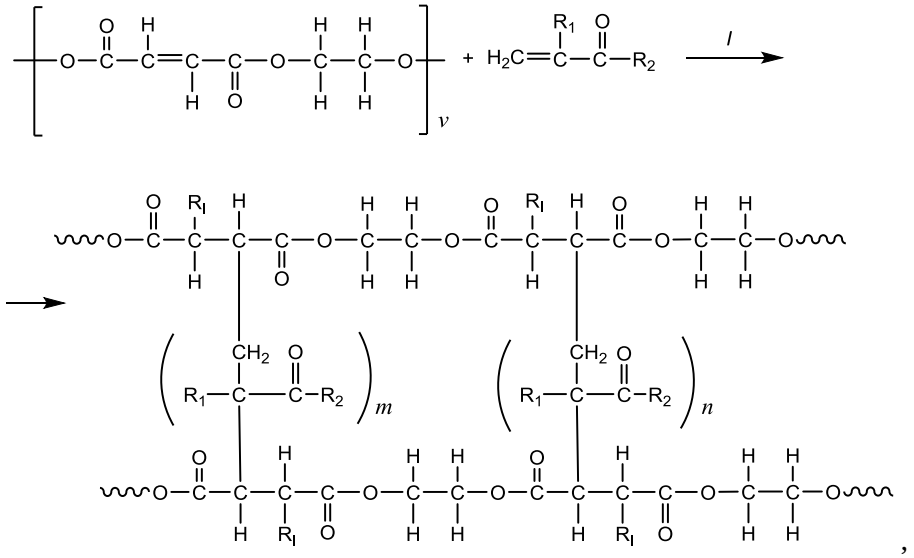
In this work a continuous effect of UV-irradiation on thermal stability of polymeric base of the sealants was studied. The copolymers of polyethylene glycol fumarate (p-EGF) with acrylic (AA) and methacrylic (MAA) acids and acrylamide (AAm) were chosen as polymeric bases.

Experimental

The reagents used in this work are ethylene glycol, acrylic and methacrylic acids, acrylamide, benzoyl peroxide, dimethylaniline ("Sigma-Aldrich"), fumaric acid ("Vekon"), aluminum chloride ("Reachem"). All of them were used without additional purification.

Initial polyester, i.e. polyethylene glycol fumarate was obtained by condensation polymerization of ethylene glycol with fumaric acid according to the procedure given in [14, 15] at a temperature of 150–160 °C in the presence of catalyst — aluminum chloride.

The objects studied — binary systems of p-EGF with AA, MAA and AAm of composition ~35:65 mass.% were obtained by radical copolymerization in the presence of optimized binary initiating system of "cold" curing (initiator (benzoyl peroxide, BP) : promoter (dimethylaniline, DMA) of the following ratio 1 %:0.15 % calculated from initial monomer mass) at a temperature of 20°C [16, 17]. Schematically the synthesis of the copolymers of p-EGF with AA, MAA and AAm is presented in Figure 1.

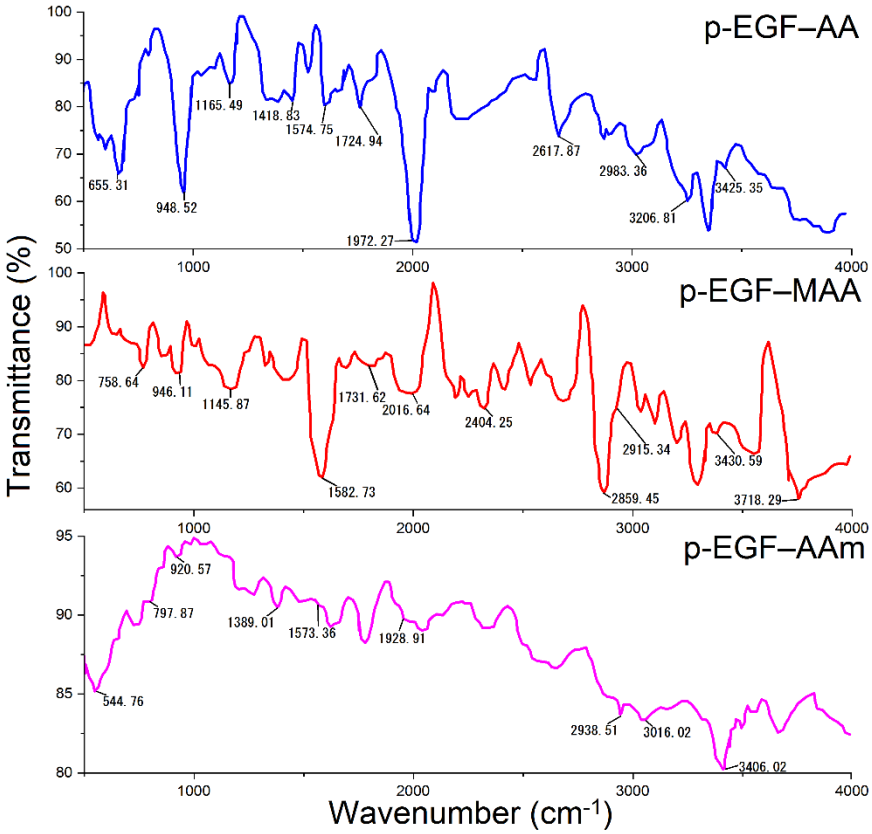


where R_1 — H, CH_3 groups; R_2 — H, NH_2 groups; R_I — the radical of initiator

Figure 1. The scheme of polymerization of p-EGF with the monomers of vinyl row

This ratio was chosen on the basis of previous studies of the rheological properties of these binary systems. Thus, the most important parameters of sealing compositions are such parameters as the density and viscosity of the initial solutions, as well as the time of their gelatinization and curing. According to [16, 17], it was found that with an increase in the content of p-EGF, those indicators grew significantly. In particular, the dynamic viscosity of p-EGF–AA solutions rise with an increase of the amount of p-EGF from 35 to 45 mass.%, respectively, from 37.6 to 182.3 mPa·s. This also applies to density, where this indicator varies from 1.1562 to 2.069 g/cm^3. The time of gelatinization and curing of these binary systems also changes. Thus, in particular, for the p-EGF–AA system with a composition of ~35:65 mass.%, these meanings are equal to 55 and 90 min., respectively, while for the p-EGF–AA ratio of ~45:55 mass.%, they are 69 and 188 min. In view of this, the ratio of comonomers ~35:65 mass.% was chosen as an optimal.

Obtained reaction products were identified using IR-spectroscopy. The analysis of IR-spectra points on the presence of characteristic bands which appear within the interval of 1570–1590 cm^{-1}, which correspond to some quantity of non-reacted unsaturated double bonds of p-EGF. The presence of absorption bands at 1724 cm^{-1} and 3425 cm^{-1} in the IR-spectra of the copolymers of p-EGF with carboxylic acids confirms the presence of –COOH groups. There are also the absorption bands at 2859 cm^{-1} and 2915 cm^{-1}, which are characteristic for the methylene groups of MAA. The presence of the peak within the range of 1910–1950 cm^{-1} is typical for amide group $–NH_2$ (Fig. 2):



a — p-EGF–AA; *b* — p-EGF–MAA; *c* — p-EGF–AAm

Figure 2. IR-spectra of unsaturated polyester–vinyl monomer

For the study of the influence of UV-irradiation on the samples of binary systems of p-EGF with AA, MAA and AAm of the composition of ~35:65 mass.% was carried out according to all-Union State Standard 32317-2012 [18] on the device Weiss UV3 of the firm Weiss Technik (Germany). It is a camera isolated from external lighting which allows studying the irradiation of plate like samples with UV-light with the wavelengths 290 and 450 nm [19].

When studying the effect of UV-irradiation on tested samples the platelets of various thicknesses ranging from 1 to 5 mm (the platelets of each thickness were prepared three times for carrying out parallel tests which provided obtaining reliable results) were prepared. During the study the irradiation intensity was constant. So, the experiment was carried out within 3 weeks (21 days) in total. Estimation of the states of the samples after irradiation was made visually i.e. the change of color and the presence of microcracks were observed. In order to establish the thermal stability of the tested samples, their thermograms were analyzed and the temperature of thermal deformation was determined [20] within the temperature interval of 30–*200°C. The end of the process takes place when the copolymer decomposes completely at a temperature of ~550–600 °C.*

Estimation of form and shape of the samples after exposure of UV-irradiation on them was done visually and then using binocular-type microscope MicroOptix MX50 (Micro Optix, Austria).

The study of thermal properties of the samples (TGA) before and after exposure to UV-irradiation was done on a device for the synchronic thermal analysis Labsys Evolution TG-DTA/DSC (“Setaram”) in dy-

namic regimen within the temperature range of 30–600 °C. Calibration of the equipment for thermogravimetry and heat current was made on $CaCO_3$ and in standards correspondingly [21]. Finely ground platelet samples after influencing with UV-irradiation and the samples of binary systems of p-EGF with AA, MAA and AAm which were not undergone to the exposure of UV-irradiation were heated in an aluminum crucible at a heating rate of 10 °C/min in nitrogen atmosphere at a flow rate of 30 ml/min. The sample's mass was ~1 g.

Results and Discussion

At present time the sealing materials on the basis of polymers play an important role almost in all fields of industry. The main volume of sealants is consumed for building and car-construction. So, the use of sealing materials allows increasing the energy-efficiency of different enterprises, including the ones working in aggressive conditions. The sealants find application in solving wide range of household problems, such as, sealing the sanitaryware. Sealing materials are of great importance in building sector as a result of improving the operational properties of construction objects, such as, heat- and vapor-isolation, waterproofing and so on.

However, during the process of operation of constructing materials they undergo to the influence of various negative factors, amongst which is UV-irradiation. While exposure to UV-irradiation within long period of time there is the change in the structure of the material and in its basic physicochemical characteristics, i.e. the photo-ageing of the product. This process is accompanied by the structural damage of polymer, appearance of the cracks (on the surface and within the thickness of the material), growing turbid of the surface and so on. Ageing causes the deterioration of the mechanical characteristics of the materials which is taken into account when calculating the building constructions by corresponding coefficients of the working conditions [22].

There are usually two types of processes take place when ageing the polymers which have reactive double bond. These are destruction and crosslinking of macromolecules. The destruction processes which take place when the product ages after affecting by UV-irradiation is called "photodestruction" [23].

With the aim of establishing the effect of UV-irradiation on binary systems of p-EGF with AA, MAA and AAm of the composition of ~35:65 mass.% the samples were analyzed visually to the change of color and appearance of turbidity. Further, a microscopic analysis on presence of microcracks on the surfaces of the samples after exposure to irradiation was done. The results of the studies obtained after visual and microscopic analyses are presented in Table.

T a b l e

The effect of UV-irradiation on the copolymers of p-EGF with AA, MAA and AAm of the composition of ~35:65 mass.%

Unsaturated polyester	Vinyl monomer	Color change, turbidity	The presence of microcracks, bubbles	Other changes
p-EGF	AA	–	–	–
	MAA	Negligible yellowish tincture	–	–
	AAm	–	–	Strong hardening, the loss of flexibility

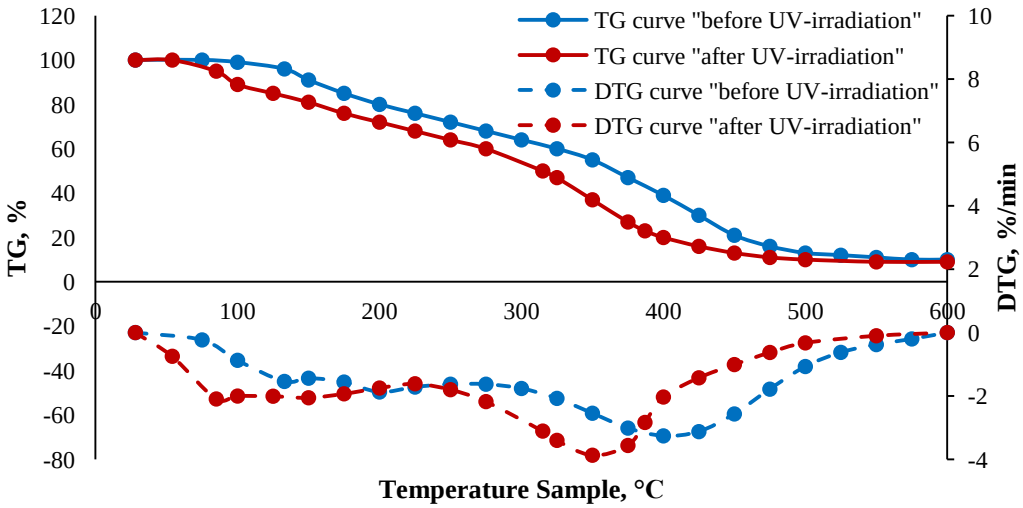
On the basis of experimental data obtained the attention should be paid to the exposure of UV-irradiation on binary system of p-EGF with AAm. In particular, the plates prepared from the copolymers of p-EGF-AAm hardened very much and had some roughness on their surfaces as a result of prolonged exposure to UV-irradiation. In this case no change in color was observed for the given samples of p-EGF-AAm of different thicknesses, however the plates lost their flexibility completely turning into rigid framework material, which can be explained by the formation of additional crosslinks within the macromolecule due to the formation of hydrogen bonds during their partial photoinitiation [24]. It is worth noting that this kind of solidifying may cause some difficulties when further operation of the sealants which are made using this polymeric base.

Analyzing the state of plates of p-EGF with MAA after prolonged exposure to UV-irradiation it was found out that all of the samples of different thickness acquired yellowish tincture. In this case the surfaces of plates did not grow dull and did not become tarnish. Also, it was expectable that the color of the plates

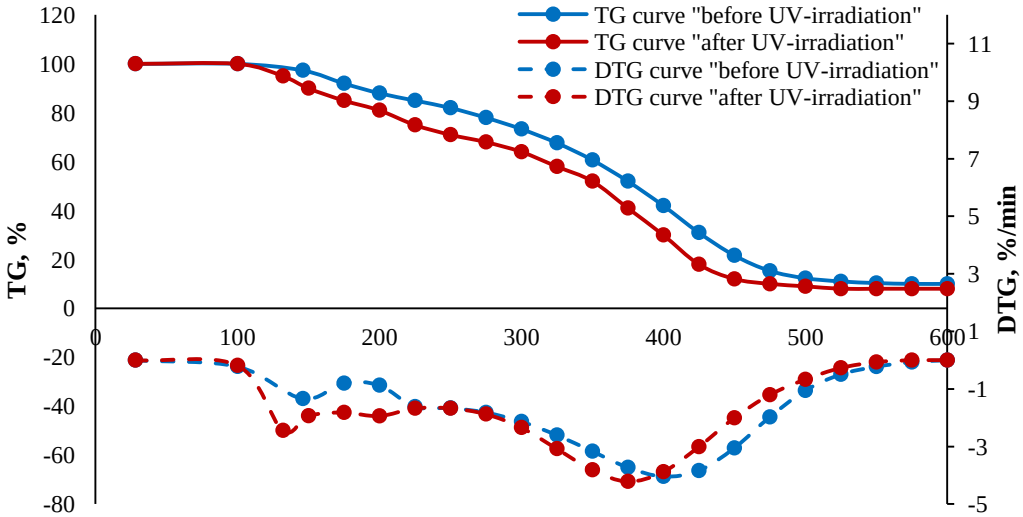
with smaller thickness (1–2 mm) changed significantly in comparison with the samples with wider thickness. Taking into account that the surfaces of the copolymers either undergo cracking or erode completely very often under the exposure to UV-irradiation, in this particular case the appearing of the microcracks and other deformations on the surface of plates were not observed by microscopic analysis.

When analyzing the samples of p-EGF with AA visual changes either in color, or appearing the bubbles or turbidity were not noticed. The presence of microcracks on their surfaces was also not observed.

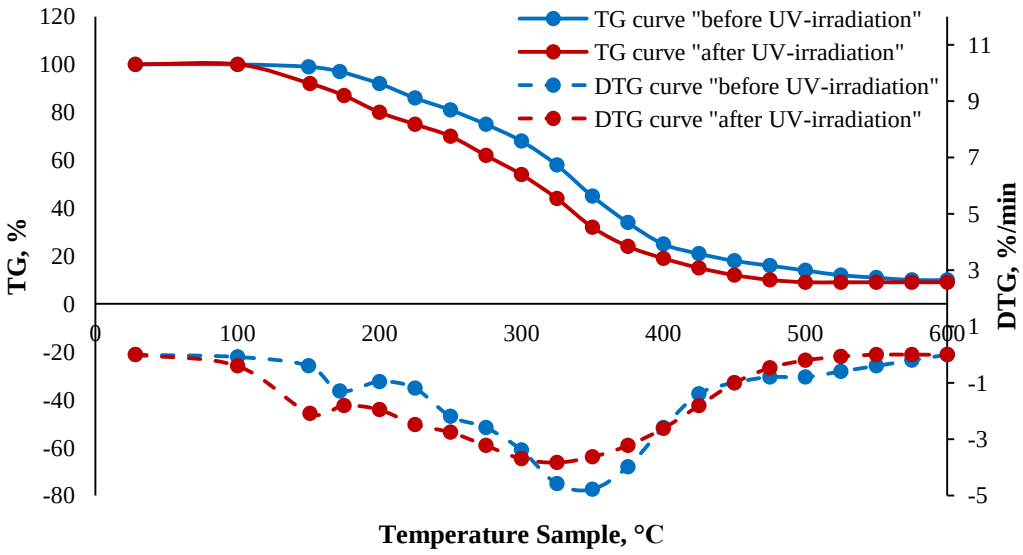
Further for instrumental analysis of thermal stability of the studied binary systems on the basis of p-EGF with AA, MAA and AAm a TGA was done to establish the temperature of start of thermal deformation before and after exposure to UV-light within 21 days. Analysis of obtained thermograms has shown a decrease of thermal stability of the sample of p-EGF-AAm after UV-irradiation. At the same time, there were no significant changes in temperature coefficient of the start of thermal deformation of the samples of p-EGF with AA and MAA. So, in particular, analyzing the graphs in Figure 3 (*a*–*c*) it is necessary to note that the temperature of the start of thermal decomposition of the copolymer of p-EGF-AAm before irradiation was 134 °C, whereas after exposure to UV-light it fell to 85 °C (Fig. 3 *a*). Similarly, the results for the other binary systems were recorded. So, the start of thermal decomposition of the copolymers of p-EGF-AA before and after exposure to UV-irradiation was at the following points: 146 °C and 132 °C (Fig. 3 *b*) accordingly, and for p-EGF-MAA these values were equal to 172 °C and 151 °C (Fig. 3 *c*):



a



b



c

a — p-EGF–AAm 32.41:67.59 mass.%; *b* — p-EGF–AA 32.54:67.46 mass.%;
c — p-EGF–MAA 32.92:67.08 mass.%

Figure 3. TG and DTG curves of the copolymers of p-EGF–vinyl monomer of various composition at a heating rate of 10 °C/min before and after UV-irradiation

Thus, in spite of the hardening the samples of p-EGF with acrylamide after influence of UV-light on them the temperature of the start of thermal deformation reduced considerably before and after irradiation. It is explained by the formation of additional crosslinks via hydrogen bonds which are formed during partial photoirradiation of macromolecules and are relatively fragile and can be easily decomposed under the influence of high temperature [23, 24].

So, on the basis of thermogravimetric analysis [25] a conclusion about decrease of thermal stability of synthesized copolymers based on p-EGF with AAm after the exposure to UV-irradiation can be made. Accordingly, the use of these copolymers as polymeric base for obtaining the sealants has strictly limited opportunities: in particular, they can be used in the places where they are not influenced by extensive UV-irradiation. Concerning the binary system of p-EGF-MAA it is necessary to note the possibility of its application for obtaining the sealants, however the use of the latter may be limited because of the change of their color as a result of the exposure to UV-irradiation on them. In comparison with the above said binary systems the copolymers of p-EGF with AA have shown the best physicochemical indices, including thermal stability after the radiation with UV-light. The results obtained allow us to say about preferable use of binary system of p-EGF-AA as a polymeric base of sealing materials.

Conclusions

The studies of thermal stability as a result of prolonged exposure to UV-irradiation on binary systems of p-EGF with AA, MAA and AAm of the composition of ~35:65 mass.% have shown high indices of thermostability for the copolymers of this unsaturated polyester with carboxylic acids after UV-irradiation within the range of 132–151 °C. In case of analyzing the temperature of the start of thermal deformation of the copolymer on the basis of p-EGF with AAm the reduce of this index before and after exposure of UV-irradiation on the samples from 134 to 85 °C was established; this limits the possibility of its application as a polymeric base of sealants.

The negative consequences of exposure to UV rays on copolymers of p-EGF with AAm should also include a change in their physicochemical parameters, expressed in the loss of flexibility and strong hardening. It should be noted that copolymers of p-EGF with carboxylic acids, AA and MAA, do not undergo such changes.

Thus, as a result of the experiments the conclusions can be made about preferable choice of binary systems of p-EGF with AA and MAA for obtaining the sealing materials on their basis.

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