

Studying the polymerization efficiency of photosensitive compositions by using the surface plasmon resonance method

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Abstract. In a real-time scale, it has been investigated the polymerization process in photosensitive compositions based on oligourethanacrylates with various concentrations of photoinitiators by using the method of surface plasmon resonance. Experimentally determined were the speed of polymerization and induction period for the studied samples of photopolymerization compositions as well as their optimum ratio of components for the chosen illumination wavelength 407 nm. It has been ascertained that with increasing the concentration of photoinitiators Irgacure 651 and Irgacure 819 from 1 up to 2% the speed of polymerization is increased by 3 and 7 times, respectively, while the induction period is decreased by almost 2 and 10 times, respectively. The presence of Irgacure 819 in these compositions provides the highest polymerization speed and the lowest induction period, which is related with the optimal choice of the illumination source wavelength inside the absorption spectrum of initiator, and the competitive absorption in other components of the photopolymer composition is minimal. These investigations are useful for developing optimal compositions of the studied materials for using them in various branches of industry.

Keywords: surface plasmon resonance, refraction index, photosensitive composition, photopolymerization, photoinitiator.

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1. Introduction

The up-to-date demand for fast prototyping goods and devices by using technology "lab-on-a-chip" (LOC) stimulates considerable interest to alternative methods of manufacturing (in particular, 3D printing), which are aimed at lowering the cost and simplicity in producing these goods. Originating from 1984, 3D printing is the process that provides creation of a three-dimensional model by building-up the material of construction. Among the ways of 3D printing, stereolithography is widespread as the process of layer-by-layer production of goods, where liquid photopolymer composition (PC) is transferred into the solid state under action of

illumination. Also, liquid PCs are used in jet-stream polymerization that can be applied with several PCs sensitive to light. This method is suitable for spraying the liquid polymers and depositing them by very thin layers, *e.g.*, 16 μm .

Photopolymers are light sensitive polymer materials capable to change their properties, for example to be polymerized under light action. They can contain monomers, oligomers, fillers and additives, such as photoinitiators, dyes *etc.* Auxiliary compounds can enhance the polymerization efficiency. The photoinitiators can decompose during the polymerization process, which results in cross-link of polymers with creation of free radicals, cations and anions [1] under

action of light irradiation. Available in the process of free-radical polymerization are light sensitive molecules of photoinitiators, *e.g.*, acrylates and methacrylates [2]. The free radicals migrate from one monomer to another in the course of chain growth. Radical polymerization stops, when two radicals, or creating polymer chains, or fragments of initiators combine [3]. PC can contain several photoinitiators, which enables to simultaneously enhance the efficiency of polymerization.

The problem to create new photopolymerization materials with a necessary complex of physico-chemical and physico-mechanical properties was always topical, and now it is solved by embedding a definite amount of structured fragments with formation of nano-dimensional systems to obtain compositional materials for various purposes. There is a necessity to study and choose an optimal composition of PC with a minimum polymerization time, which is important for fast 3D printing. The most widespread methods for studying polymerization properties and refraction index of photopolymer compositions are as follows: the refractometric one and that of infrared spectroscopy. The refractometric method is simple in usage, but it does not allow tracing the changes inside a sample in a real-time scale. Besides, it requires a considerable volume of the studied substance (minimum 300 μL). IR spectroscopy is widely used to obtain information about mechanisms and kinetics of polymerization as well as to determine relations between reaction capabilities and activation energies [4]. This method enables to permanently trace the key elements of polymerization (monomers and polymers) and provides information about the kinetics of polymerization reactions. However, this method is expensive, needs additional reagents and special equipment (optics transparent in the IR range and the system for drying air).

The method used in our work for determining the PC refraction index and studying the polymerization process in a real-time scale is the method based on the phenomenon of surface plasmon resonance (SPR). The optical measurements based on SPR are widely used in the chemical-and-biological analyses when registering molecular adsorption in various substances from gases up to liquids and solids [5]. Analytical devices based on SPR possess the high accuracy of measurements, for example $\pm 3 \cdot 10^{-8}$ RIU (refractive index unit) in the device Biacore T200, and small volume of the studied substance: from 0.1 to 10 μL . The diagnostic SPR devices have high sensitivity to low concentrations of analyzed substances, which allows using them as precise analytical devices in various investigations. Shown in the work [6] is the possibility to apply the SPR method for determining the refraction index of photopolymer adhesives. Performed there are the calculations of the speed and duration of photopolymerization process in them.

As a rule, to activate photopolymerization, ultraviolet light sources are used, in particular lasers. However, these lasers are expensive and insufficiently reliable. Due to recent commercialization of blue light laser diodes with bright emission at the wavelength

405 nm and low losses, there observed is an increased interest to developing PC for stereolithographic 3D printing, which can function within the spectral range approximately 400...420 nm [7].

The aim of this work was to create photosensitive compositions with various ratios of constituent components including oligourethanacrylates, to study photopolymerization efficiency under action of light with the maximum 407 nm in the emission spectrum by using the method of surface plasmon resonance.

2. Materials and methods

Chosen as the objects for investigation were 13 samples of PC with various component compositions (see Table 1). These PC were studied both in the non-polymerized (liquid) state and in the polymerized (solid) one. They included the following substances: photoinitiator 2,2-dimethoxy-1,2-diphenylethanone (Irgacure 651), photoinitiator phenyl-bis-(2,4,6-trimethylbenzene)-phosphine oxide (Irgacure 819), photoinitiator 2-hydroxy-2-methylpropiophenone (Darocur 1173), dimethylaminoethyl methacrylate (DMAEM), triethyleneglycol dimethacrylate (TMG-3) as well as the main component – oligourethanacrylates based on aliphatic diisocyanate (OUA-27).

As a basis for PC chosen for investigations, we used polymerization-capable urethanacrylate oligomer based on isophorone-diisocyanate, polypropylene glycol and acrylate monomer (OUA-27) with the molecular mass 1700 and functionality through double bonds – 2. As acrylate oligomer, we added triethyleneglycol dimethacrylate (TGM-3) that can perform the function of active solvent for this composition. To enhance PC reactivity and photosensitivity, its composition was supplemented with synthesized oligomer modifier – oligomethacrylate with tertiary amino group that functions as polymerization accelerator. This choice of oligomer component to OUA-27 composition system is based on the necessity to provide such properties of materials and coatings as high photosensitivity, transparency, solidity of a polymer layer, wear resistance and adhesion strength.

The studied PC were deposited onto two types of substrates in the form of glass plates coated with a top gold layer (thickness 50 ± 2 nm) and without this gold coating. These plates had the thickness 1 mm, dimensions 20×20 mm and were made of glass $\Phi 1$ (with the refraction index $n = 1.61$ relatively to vacuum). The sandwich structure “glass–gold–PC–glass” was used to determine the following characteristics of the polymerization process: values of refraction index before and after the process, speed of the process, delay of it (induction period), as well as its duration. The structure “glass–PC” was used to determine the value of refraction index after polymerization with account of the critical angle in the SPR curve.

To study the photopolymerization process in the real-time scale, we used the device “Plasmon-71” with the emission wavelength $\lambda = 850$ nm. This device was designed using the optical geometry by Kretschmann [8]

Table 1. Component composition of the studied PCs.

Sample No	OUA-27, %	Irgacure 651, %	Irgacure 819, %	Darocur 1173, %	DMAEM, %	TGM-3
1	99	–	1	–	–	–
2	98	–	1	1	–	–
3	99	1	–	–	–	–
4	98	1	–	1	–	–
5	98	–	2	–	–	–
6	97	–	2	1	–	–
7	96	–	2	2	–	–
8	98	2	–	–	–	–
9	97	2	–	1	–	–
10	94	–	2	1	3	–
11	92	–	2	1	5	–
12	87	–	2	1	10	–
13	87	–	2	1	–	10

at the V. Lashkaryov Institute of Semiconductor Physics, National Academy of Sciences of Ukraine. The devices of “Plasmon” series provide measurements of the angle of incidence from 54 up to 74 degrees and determination of the refraction index within the range 1.33...1.48 with the accuracy $\pm 25 \times 10^{-6}$ RIU. The optical scheme capable to provide the conditions for SPR is shown in Fig. 1.

Light from the emission source 1 passes through the collimation lens 2, polarizer 3 and is directed to the glass optical prism 4 (refraction index $n = 1.61$, glass of the grade $\Phi 1$), the top face of which is joined with the studied sample 7. Light reflected from the prism through the lens 5 falls onto the surface of photodetector 6. Changing the angle of incidence for the light from the source onto the sample surface was provided by turning the prism. It was measured the dependence of reflected radiation intensity on the angle of light incidence (SPR reflection characteristics with the typical resonance dip) on the sample with PC. Using numerical processing, we determined the minimum of reflection characteristic, which then was re-calculated into the value of refraction index inherent to the non-polymerized PC. Further, we performed the PC polymerization process by using a semiconductor diode emitting light with the wavelength 407 nm, which was mounted at the distance 4 cm from the sample surface.

The photopolymerization process was controlled with account of the angle shift observed for the minimum of reflection characteristic, change in the reflection intensity or change in the angular position of the critical angle typical for total internal reflection. The plates were placed using the immersion liquid onto the operation face of the device prism. Then, using a micropipette we brought $40 \pm 5 \mu\text{L}$ of PC onto the plate from the gold layer side. After that, this structure was covered with a glass plate (without any metal layer) to provide uniform spreading of PC over the gold surface. It enabled to obtain the PC layer thickness within the range $100 \pm 15 \mu\text{m}$.

Then, we performed measurements in two modes: i) making periodical angle scanning with simultaneous recording the minimum of reflection characteristic and ii) making periodical measurements of the reflected light intensity at the slope of reflection characteristic under the condition of a fixed angle. In the case of quickly polymerizing PCs, the measurements were carried out in the mode of recording the reflected light intensity with the step 0.1 s. The mode of recording the minimum of reflection characteristic was used in the case of slow polymerizing PCs, since this regime provides the period between measurements 3 s.

The induction period was determined as the time interval from the beginning of exposure up to the start of changes in the PC refraction index, which is determined as the beginning of changes in the angular position of SPR minimum or in the value of reflection intensity. Duration of the polymerization process was determined as the time interval from the beginning of exposure up to

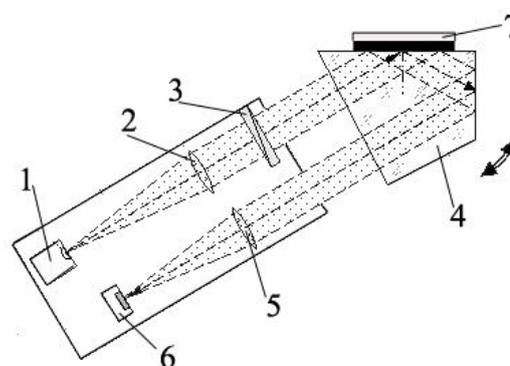


Fig. 1. Optical scheme of the device “Plasmon-71” [9]. 1 – source of light, 2 – collimation lens, 3 – polarizer, 4 – glass optical prism, 5 – lens, 6 – photodetector, 7 – studied sample.

the ending of changes in the PC refraction index, which was indicated by stabilization of the SPR minimum angular position. The error of determining the polymerization time was no higher than ± 0.1 s. The speed of polymerization was estimated as the ratio of effective penetration depth for the surface plasmon field δd to the polymerization duration. In the case of light wavelength 850 nm, the effective field penetration depth reached $0.42 \mu\text{m}$.

To determine the refraction index after completion of the polymerization process, PC of the volume $0.5 \mu\text{L}$ was deposited using a Pastier pipette onto the glass plates without any gold layer. Then, photopolymerization was performed. After completion of the process, we placed the samples through the immersion liquid onto the device prism operation face and measured the reflection characteristics, which enabled to determine the critical angle.

The value of refraction index before polymerization for the wavelength $\lambda = 589$ nm, we found using the refractometer RL-3 (providing to measure that index within the range 1.3...1.7). The PC sample of the volume 0.5 mL was placed between the prisms of the optical refractometer RL-3. After compensation of dispersion and determination of the critical angle value, we registered the value of refraction index. The error of results of measurements with the refractometer RL-3 was $\pm 2 \cdot 10^{-4}$.

3. Results and discussion

In the process of PC polymerization under action of light, with time we observed changes in the position of reflection curve minimum. The reflection curve as a whole shifted to the right, so did its minimum. It is indicative of increasing refraction index values calculated for the light wavelength 850 nm. Each PC polymerized with a different speed. The kinetic curves of polymerization inherent to the samples 1 to 4 are shown in Fig. 2.

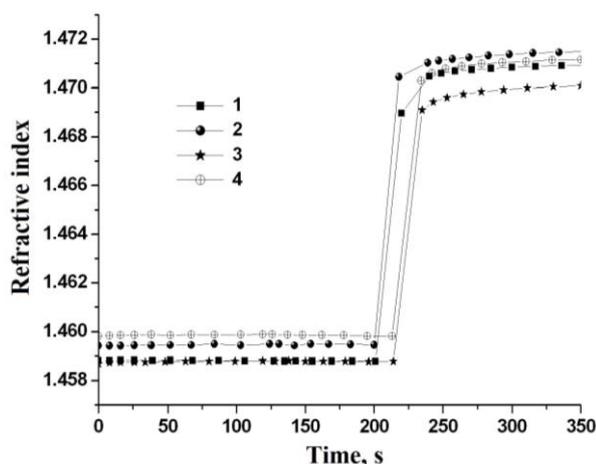


Fig. 2. Kinetic curves corresponding to polymerization of PC samples 1 to 4 measured using the SPR method.

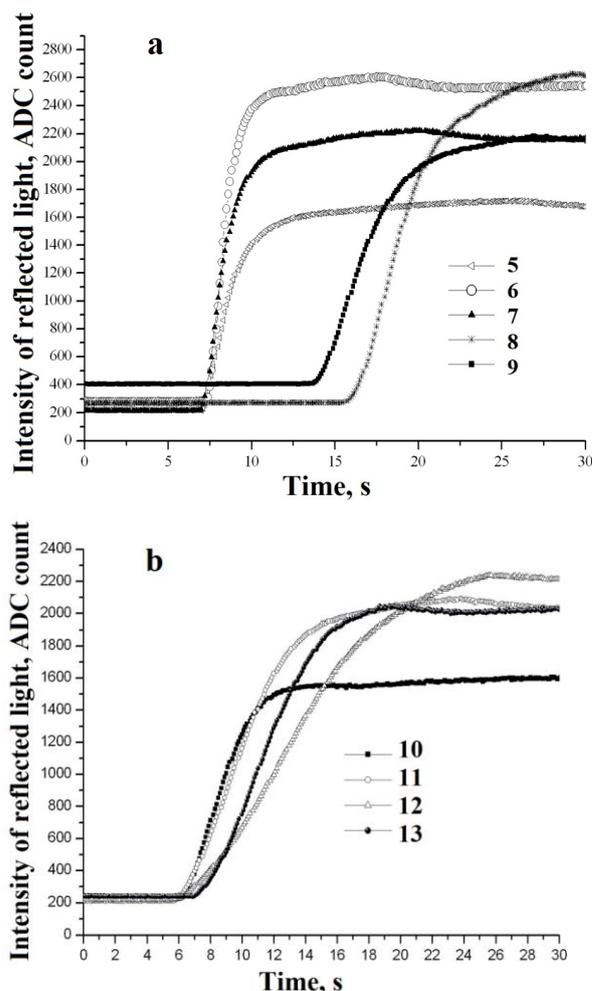


Fig. 3. Kinetic curves of PC polymerization for the samples 5 to 13 measured using the SPR method.

The PC samples 1 to 4 showed the duration of polymerization within the range 46 ± 9 s. The samples 1 and 2 have practically the same induction period. The polymerization kinetics inherent to the other PC samples are added in Fig. 3 in the form of dependences of the reflection intensity on time with the measurement step 0.1 s. Illumination started from the 5th second of measuring the kinetics.

Different slopes of the kinetic curves (Fig. 3) are indicative of various polymerization speeds inherent to the PC samples. In the case of the samples 5 to 7, this slope is steeper, and respectively, the speed is higher. It is seen from Fig. 3 that the difference between values of reflected light intensity before polymerization and after it is very close. The obtained polymerization characteristics for various samples are summarized in Table 2.

The value of relative changes in the refraction index, as a consequence of polymerization, for the studied PC samples, lies within the range $0.89 \pm 0.31\%$. Duration of polymerization is the longest in the sample 3 (55 s), and in the sample 6 it is 9-fold less (6 s). The lowest value of refraction index after polymerization is inherent to the sample 8 (1.4611 for the measuring

Table 2. Characteristics of the PC samples.

No	Refraction index before polymerization, RIU	Refraction index after polymerization, RIU	Relative change of the refraction index, %	Duration of polymerization, s
1	1.4588	1.471	0.84	48
2	1.4595	1.4716	0.83	38
3	1.4588	1.4703	0.79	55
4	1.4598	1.4713	0.79	54
5	1.4504	1.4636	0.91	7
6	1.4526	1.4635	0.75	6
7	1.4515	1.4659	0.99	7
8	1.4512	1.4611	0.68	18
9	1.448	1.4653	1.2	17
10	1.4547	1.4631	0.58	9
11	1.4512	1.4641	0.89	11.3
12	1.4506	1.465	0.99	19.4
13	1.4521	1.4683	1.18	12.9

wavelength $\lambda = 850$ nm), while the highest one is related to the sample 2 (1.4716). The shortest duration of polymerization is typical for the photoinitiator Irgacure 819, which indicates its high photosensitivity.

We measured the refraction index values for the samples before polymerization by using the optical refractometer ($\lambda = 589$ nm), too. The respective data are shown in Fig. 4.

The lowest refraction index before polymerization was inherent to the sample 8 (1.4629), while the highest one is related to the sample 7 (1.4675). The higher the refraction index value, the higher the speed of photopolymerization, and *vice versa* (Fig. 4). Shown in Fig. 5 is the induction period for the various PC samples.

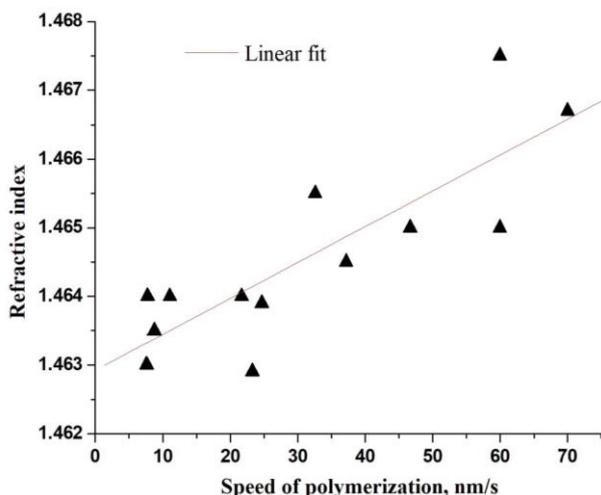


Fig. 4. Dependence of the calculated refraction index value on the speed of PC polymerization.

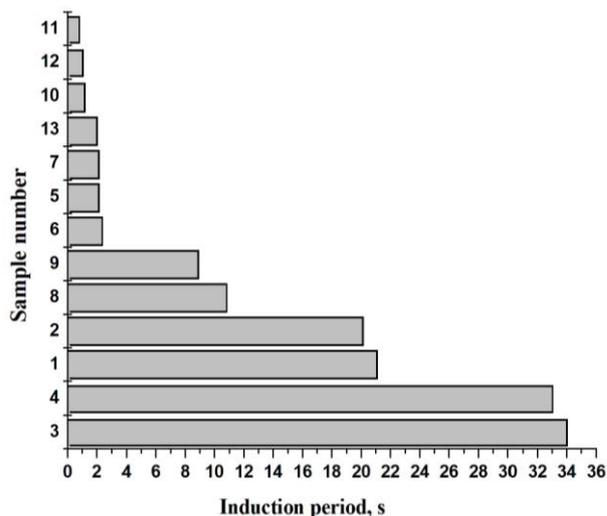


Fig. 5. Ordered induction period histogram of various PC samples.

The longest induction period was observed in the sample 3, and the shortest one – in the sample 11. It was equal 0.7 s. The sample 11 contained such photoinitiators as Irgacure 819 and Darocur 1173 with the concentrations 2 and 1%, respectively (Fig. 5). Availability of different induction periods in different PC samples is related with the concentration level of inhibitor (*e.g.*, oxygen) and initiative radicals that arise in the process of photolysis inherent to photoinitiator molecules under light action. Besides, PCs were added with various amounts of modifiers with the blocks of tertiary amines that easily interact with oxygen playing the role of inhibitor (samples 10 to 12), which had a considerable effect on lowering the induction period during polymerization.

As seen in Fig. 5, the largest influence on the polymerization process is provided by dimethyl-aminoethyl methacrylate. It can be explained by the content of tertiary amines, which reduces the induction period, as well as by presence of the acrylate oligomer component. These two substances interact with the oligourethanacrylate oligomer and create a branched polymer structure. As a consequence, this process is considerably accelerated, and the level of transformations becomes more complete. Accordingly, the initial speed of polymerization depends on the relationship between the rate of initiation reactions and the speed of polymerization chain growth. The speed of polymerization for various samples is shown in Fig. 6.

Among all these PCs, the sample 6 has the highest polymerization speed. It contains such photoinitiators as Irgacure 819 and Darocur 1173 in the concentration 2 and 1%, respectively. The lowest speed is inherent to the sample 3 (Fig. 6). Conventionally, the range of PCs with the highest polymerization speed comprises also the samples 5 to 7 (65 ± 5 nm/s), while the range with the lowest speed includes the samples 1 to 4 (9.3 ± 1.7 nm/s).

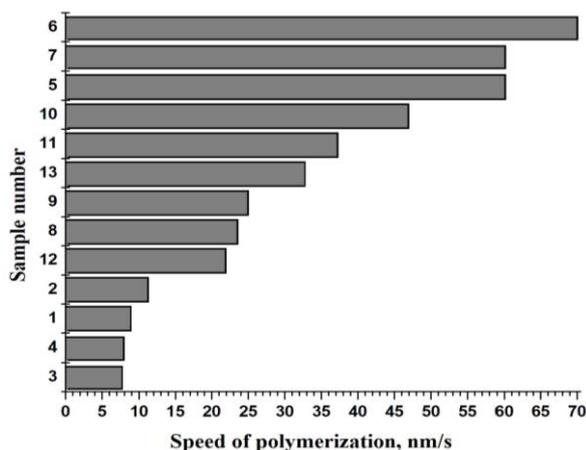


Fig. 6. Ordered polymerization velocity histogram of various PC samples.

It follows from these data that the higher concentration (2%) of the photoinitiator phenyl-bis-(2,4,6-trimethylbenzene) phosphine oxide (Irgacure 819) results in the growth of polymerization efficiency. Thus, the increase in the concentrations of photoinitiators enhances the polymerization speed of PCs, which is related with coincidence between the used wavelength of active light (407 nm) and the absorption band of the studied material. It is noteworthy that the highest polymerization speed corresponds to the samples modified with the photoinitiator Irgacure 819 (2%), the absorption band of which comprises the range of active light. At the same time, the samples with Irgacure 651 (2%), the absorption spectrum of which is shifted to the shorter wavelengths [10], have 2.6 times lower photopolymerization speed as compared with the case of Irgacure 819.

4. Conclusions

The method based on surface plasmon resonance has enabled to determine the speed of polymerization and induction period for the studied thirteen photopolymer compositions. It has been also ascertained the optimal relationship between their components, when using the wavelength of actinic light 407 nm. The used photoinitiators Irgacure 651, Irgacure 819, Darocur 1173, and the additives DMAEM and TGM-3, being in the concentrations of 1 to 5%, have a considerable influence on the efficiency of polymerization from the viewpoint of shortening the induction period (2 to 10 times) and accelerating the process (3 to 7 times). The efficiency of polymerization process strongly depends on the spectral position of active light wavelength relatively to the absorption bands of photoinitiators, and on the absence (or minimum) of competing absorption inherent to other components in the studied materials.

Thus, the SPR method is a powerful tool for studying the above processes in a real-time scale, which is very important to develop new effective photopolymer compositions for 3D printing and ascertain their physico-chemical properties as well as regularities of their modification.

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Дослідження ефективності полімеризації фоточутливих композицій за допомогою методу поверхневого плазмонного резонансу

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Анотація. У масштабах реального часу було досліджено процес полімеризації світлочутливих композицій на основі олігуретанакрілатів з різними концентраціями фотоініціаторів за допомогою методу поверхневого плазмонного резонансу. Експериментально визначено: швидкість полімеризації та індукційний період для досліджуваних зразків композицій, а також оптимальне співвідношення їх компонентів для вибраної довжини хвилі опромінення 407 нм. Було встановлено, що зі збільшенням концентрації фотоініціаторів Irgacure 651 та Irgacure 819 від 1 до 2% швидкість полімеризації збільшується відповідно в 3 та 7 разів, тоді як індукційний період зменшується майже в 2 та 10 разів відповідно. Наявність Irgacure 819 в цих композиціях забезпечує найбільшу швидкість полімеризації та найменший час індукції, що пов’язано з оптимальним вибором довжини хвилі джерела опромінення всередині спектра поглинання ініціатора, а також з мінімальним конкурентним поглинанням в інших компонентах фотополімерної композиції. Ці дослідження корисні для розробки оптимальних композицій досліджуваних матеріалів у різних галузях промисловості.

Ключові слова: поверхневий плазмонний резонанс, показник заломлення, фоточутлива композиція, фотополімеризація, фотоініціатор.