

# Influence of radical photoinitiators on features of polyacrylate syrups and self-adhesives\*)

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**Abstract:** Pressure-sensitive adhesives (PSAs) were prepared via UV-irradiation of an acrylic syrups with reactive diluent (functional diacrylate) and photoinitiator. The acrylic syrups were received via free radical bulk polymerization process (FRBP) and influence of radical photoinitiator ( $\alpha$ -hydroxyketones, acylphosphine oxides or mixtures thereof) was investigated. It was revealed that the mixture of bisacylphosphine oxide (Omnirad 819) and  $\alpha$ -hydroxyketone (Omnirad 184) was the most effective in FRBP (the highest monomer conversion), but preferable features of acrylic syrups (viscosity and molecular weight of copolymers) and self-adhesive properties of PSAs (especially cohesion) was achieved due to  $\alpha$ -hydroxyketones using.

**Keywords:** bulk photopolymerization, acrylic syrup, radical photoinitiators, pressure-sensitive adhesives.

## Wpływ fotoinicjatorów na właściwości poliakrylanowych syropów i klejów samoprzylepnych

**Streszczenie:** Z syropów akrylanowych oraz wielofunkcyjnego monomeru i fotoinicjatora rodnikowego otrzymano poliakrylanowe kleje samoprzylepne. Syropy akrylanowe uzyskano metodą fotopolimeryzacji w masie monomerów z zastosowaniem różnych fotoinicjatorów rodnikowych (z grupy  $\alpha$ -hydroksyketonów, tlenków acylofosfiny i ich mieszanin). Stwierdzono, że najbardziej efektywna w procesie fotopolimeryzacji jest mieszanina fotoinicjatorów: tlenku bisacylofosfiny (Omnirad 819) i  $\alpha$ -hydroksyketonu (Omnirad 184) (największa wartość konwersji monomerów), jednak większą lepkością oraz większymi ciężarami cząsteczkowymi cechowały się syropy akrylanowe z udziałem  $\alpha$ -hydroksyketonów (Omnirad 127 lub Omnirad 184), a uzyskane z nich kleje wykazywały lepsze cechy samoprzylepne, zwłaszcza kohezję.

**Słowa kluczowe:** fotopolimeryzacja w masie, akrylany, fotoinicjatory rodnikowe, kleje samoprzylepne.

The first article on photopolymerization initiated using the sun light appeared in 1845 [1]. However, photochemical reactions began to be used on a larger scale in the mid-twentieth century. Printing technology companies (Eastman Kodak and DuPont) significantly contributed to the development of this technology [2, 3]. In the 1990s, UV technology was expanding rapidly – in 1995 the global market of UV-cured systems has reached a billion dollars [4]. High reaction rate, lower energy costs or the possibility of solvent-free polymerization – these benefits allow considering the photopolymerization method as pro-ecological, willingly used in many industries and still intensively

developed by research centers [5, 6]. Currently, photopolymerization are encountered in various experimental conditions, for example, in film, gas phase, aerosol, multilayers, solid state, on surface, in ionic liquid or under magnetic field [7]. However, the most common photopolymerization method is film photopolymerization (radical or cationic), for example photopolymerization of acrylates, epoxides, vinyl ethers. This type of photopolymerization is mainly used for cross-linking of polymeric materials, such as: varnishes, hydrogels, dental fillings, printing inks and adhesives [8–13]. Photopolymerization in film is extremely effective, especially radical photopolymerization of multifunctional monomers and oligomers is very fast – in seconds, with the help of light, a viscous liquid turns into a solid. This process has been discussed in many publications [14–18]. On the other hand, the process of free radical bulk photopolymerization (FRBP) is relatively rarely mentioned [19–22]. FRBP process is a photopolymerization initiated by the decomposition of the photoinitiator into radicals, that occurs in a large volume of reagents with mixing. Finally prod-

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uct from FRBP is solution of polymer in unreacted monomers (called polymer syrup), less often a polymer. Received syrup, which is a solvent-free binder, only need to be mixed with additives (additional part of photoinitiator, multifunctional monomers or oligomers) to create a composition for coating materials (e.g. varnishes) or adhesives. After UV-crosslinking of such composition, finished product is achieved. Using this method, in the last few years, a group of scientists from Dankook University (Republic of Korea) obtained acrylate binders for optically clear pressure-sensitive adhesives (PSA's) [20, 23–27]. Researchers focused on obtaining optically clear adhesives using new monomers (tetrahydrofurfuryl acrylate, acryloyl morpholine, menthyl acrylate and derivatives of monosaccharides) and mainly in the presence of 1-hydroxy-cyclohexyl-phenyl-ketone as photoinitiator and 1-dodecanethiol as chain transfer agent. To the best of our knowledge, the available literature has no information on the effect of photoinitiators on the bulk photopolymerization process. However, numerous scientific studies are known about the effect of photoinitiators in film or solid state photopolymerization processes [28]. This paper presents preliminary results of our investigations on preparation of pressure-sensitive adhesives (PSAs) by means of free radical bulk photopolymerization process. The use of the FRBP process to obtain PSA tapes is particularly advantageous compared to conventional preparation methods, *i.e.* the time to obtain an adhesive binder is much shorter (only *ca.* 30 min in FRBP process and 6–8 h in free radical copolymerization of acrylate monomers in solvent) and no solvents are used, as is the case with conventional methods. The aim of this work was to determine the impact

of selected radical photoinitiators (*i.e.*  $\alpha$ -hydroxyketones, acylphosphine oxides and mixtures thereof) on FRBP process of (meth)acrylate monomers, properties of obtained polymer syrups and self-adhesive properties of final product (*i.e.* acrylic pressure-sensitive adhesives). Additionally, the photopolymerization kinetics were monitored and polymerization rate and monomer conversion were determined.

## EXPERIMENTAL PART

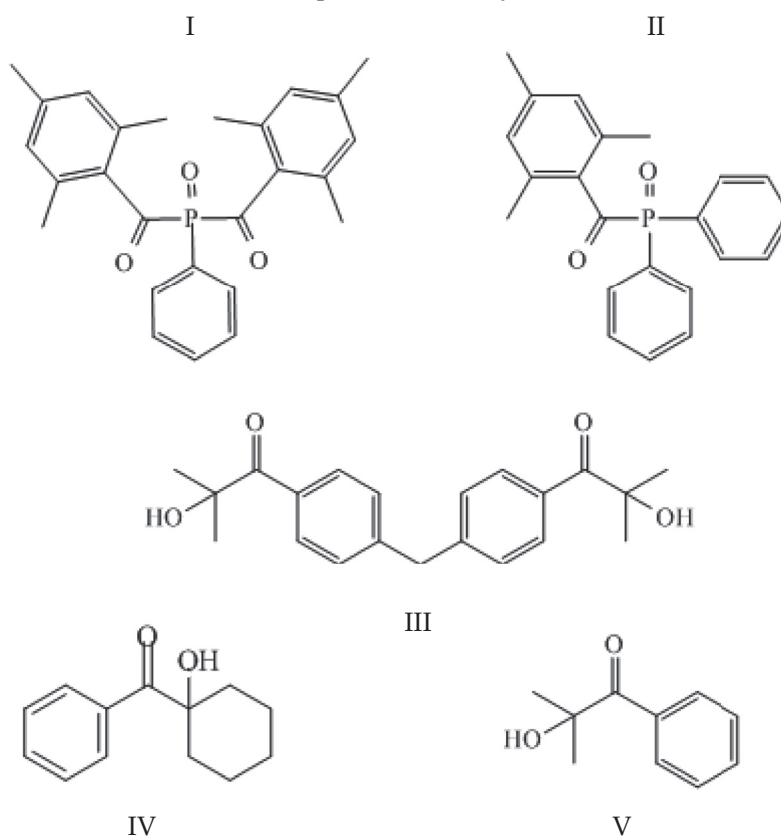
### Materials

The following monomers were used for the preparation of polymer syrups using FRBP process: butyl acrylate (BA, Sigma Aldrich, Germany), butyl methacrylate (BMA, Sigma Aldrich, Germany), 2-hydroxyethyl acrylate (HEA, Sigma Aldrich, Germany). As the radical photoinitiators (PIs, IGM Resins, USA) were tested:

– acylphosphine oxides: bis(2,4,6-trimethylbenzoyl)phenylphosphine oxide [Omnirad 819, Formula (I)]; diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide [Omnirad TPO, Formula (II)];

–  $\alpha$ -hydroxyketones: 2-hydroxy-1-(4-[4-(2-hydroxy-2-methylpropionyl)benzyl]phenyl)-2-methylpropan-1-one [Omnirad 127, Formula (III)], 1-hydroxy-cyclohexyl-phenyl-ketone [Omnirad 184, Formula (IV)].

The adhesive compositions consisted of: (i) obtained syrup; (ii) photoinitiator Irgacure 2022 – mixture of Irgacure 819 (80 wt %) and Darocure 1173 (20 wt %) (BASF, Germany) [Formula (V)]; (iii) 1,6-hexanediol diacrylate



(HDDA, BASF, Germany) as reactive diluent. The monomers and photoinitiators were applied without purification.

### Preparation of acrylic syrups

The acrylic syrups (AS) were synthesized via free radical bulk photopolymerization (FRBP) of BA (3 mol), BMA (1 mol) and HEA (1 mol) and 1 mmol of PIs. The FRBP process was carried out in a glass reactor (250 cm<sup>3</sup>) equipped with a mechanical stirrer (anchor) and thermocouple, in the presence of argon as an inert gas. A mixture of monomers (50 g) and PI were introduced into the reactor. Before starting the process, the reactor mixture was flushed with argon for 20 min. Then the photopolymerization process was started. The source of ultraviolet radiation was a medium pressure mercury lamp (UVAHAND 250 GS, by Honle, intensity 10 ± 2 mW/cm<sup>2</sup>), placed perpendicular to the side wall of the reactor. The FRBP process was realized at room temperature for 30 min.

### Preparation of PSAs

The adhesive compositions for preparation one-sided adhesive tapes (PSAs) were compounded using the acrylic syrup (93 wt %), photoinitiator Irgacure 2022 (4 wt %) and reactive diluent HDDA (3 wt %). The compositions were applied onto a polyester foil and UV-irradiated for 40 s (UV dose: 10 J/cm<sup>2</sup>) using a medium pressure mercury lamp (Aktiprint-mini 18-2, type: UN50029, Technigraf GmbH, Germany). The UV-exposition was controlled with a radiometer (Dynachem 500; Dynachem Corp., USA). Base weight and thickness of adhesive layers were 120 g/m<sup>2</sup> and 100 μm, respectively.

### Methods of testing

#### Characteristic of acrylic syrups

The viscosity of AS (at 23°C) was measured by means of DV-II Pro Extra viscometer (spindle #7, 50 rpm; Brookfield, USA).

Monomer conversion after FRBP process was determined by nuclear magnetic resonance spectroscopy NMR

(Bruker DPX Avance III HD Spectrometer; 400MHz). The average molecular weights of obtained copolymers ( $M_n$  and  $M_w$ ) and polydispersity ( $D$ ) were performed by gel chromatography using polystyrene calibration in THF at 35°C at the Centre of Polymer and Carbon Materials of the Polish Academy of Sciences in Zabrze.

### Photopolymerization kinetics

The kinetics of the photopolymerization process of monomer-PI systems (identical compositions to those involved in the FRBP) were tested using a differential scanning calorimeter with a UV attachment (DSC Q100, TA Instruments, USA; UV-light emitter Omnicure S2000; Excelitas Technologies, USA) at room temperature. Samples (5 mg) were irradiated with UV in the range 320–390 nm with an intensity of 250 mW/cm<sup>2</sup> in argon atmosphere. Polymerization rate ( $R_p$ , %/s) and conversion of double bonds ( $p$ , %) were calculated according to [29].

### PSAs properties

Self-adhesive properties of the obtained PSAs were tested according to AFERA 4001 (adhesion to steel substrate), AFERA 4015 (tack) and AFERA 4012 (cohesion). These parameters were evaluated using three samples of each PSAs using a Zwick/Roell testing machine (Z010, ZwickRoell, Germany).

## RESULTS AND DISCUSSION

The first stage of investigations was monitoring of photopolymerization process of acrylic monomers with PIs using photo-DSC method. Performing the photo-DSC tests was aimed at acquiring basic knowledge about the course of photopolymerization process of tested PIs with (met)acrylate monomers. However, it should be emphasized that the thin film photopolymerization (photo-DSC) process should not be compared with the FRBP process. Compositions of monomers and PIs were the same as for FRBP process (Table 1).

Figure 1 presents photopolymerization rate  $R_p$  as function of irradiation time in monomer mixtures. As can be

**Table 1.** Compositions used to obtain acrylic syrups

AS symbol/ PSA	Photoinitiator (1 mmol)		Monomers (molar ratio)		
	Type	Symbol	BA	HEA	BMA
819 TPO	Acylphosphine oxide	Omnirad 819	3	1	1
127 184		Omnirad 127 Omnirad 184			
819/184* TPO/184*	Acylphosphine oxide/ $\alpha$ -hydroxyketone	Omnirad 819/Omnirad 184 Omnirad TPO/Omnirad 184			

\* 0.05 mmol of acylphosphine oxide and 0.05 mmol of  $\alpha$ -hydroxyketone.

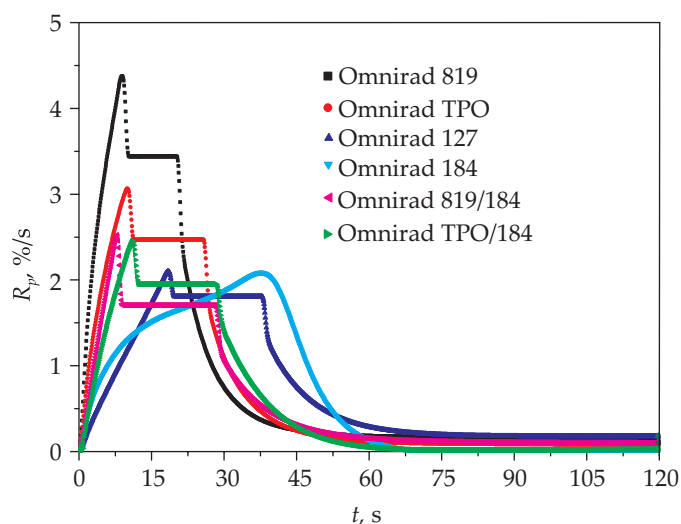


Fig. 1. Photopolymerization rate  $R_p$  as function of irradiation time in monomer mixtures (BA/BMA/HEA = 3 : 1 : 1 mol) containing 1 mmol of different PIs

observed, the highest value of  $R_p$  was noted for sample with acylphosphine oxide Omnirad 819 and the next was also acylphosphine oxide Omnirad TPO. Omnirad 819 is a bisacylphosphine oxide and generate four radicals per photoinitiator molecule, which means that one molecule of Omnirad 819 can initiate four chains (instead Omnirad TPO is monoacylphosphine oxide). For that reason, the  $R_p$  for sample 819 was the highest. Nevertheless, in case of mixture with Omnirad 819 decrease in polymerization rate was the fastest. Probably, a large number of growing chains in thin films (in case of photo-DSC measurements, photopolymerization is carried out in relatively thin film), causes the increase of the viscosity of the system and inhibits the reaction. For this reason, full conversion of acrylic monomers was achieved after a relatively long time in comparison to other samples (after 170 s, Fig. 2). Similarly was in case of sample with Omnirad TPO, the full conversion of double bond was achieved after relatively long time (50 s). However, among other initiating systems, more reactive were mixtures of acylphosphine oxide with  $\alpha$ -hydroxyketone (819/184 and TPO/184), whereas the polymerization rate was the lower for system with  $\alpha$ -hydroxyketone Omnirad 184 (than Omnirad 127, Fig. 1). In this case, after 170 s of UV-exposure, the sample did not reach full conversion of double bond (ca. 80%, Fig. 2). Among  $\alpha$ -hydroxyketones, bifunctional Omnirad 127

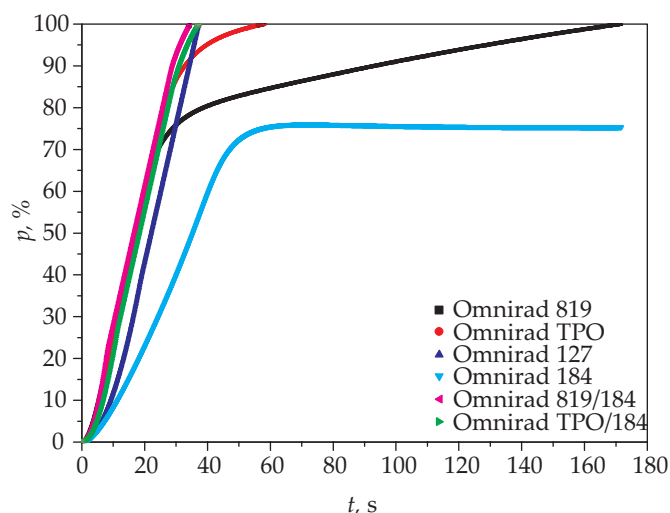


Fig. 2. Conversion of functional groups as function of irradiation time in (meth)acrylate monomer mixtures (BA/BMA/HEA = 3 : 1 : 1 mol) containing 1 mmol of different photoinitiators

showed higher reactivity. However, the fastest full monomer conversion was achieved in mixed systems (26 s for sample 819/184 and 30 s for TPO/184). Interestingly, photopolymerization process for all compositions was characterized by a short course (6–10 seconds) with a constant reaction rate. Only for sample with Omnirad 184, the kinetic curve was slightly different. Unpublished results of the authors indicate that the effect of constant reaction rate results from monomer mixture composition. In conclusion, studied using photo-DSC method have shown that the highest maximum polymerization rate characterized systems with acylphosphine oxide than  $\alpha$ -hydroxyketones and mixtures of PIs (819/184 and TPO/184) achieved an intermediate results. Nevertheless, samples with mixtures of PIs were the fastest to obtain full monomer conversion.

The main stage of work was the preparation of AS via FRBP process in glass reactor. The compositions to FRBP process, *i.e.* acrylic monomers with photoinitiator(s) were shown in Table 1.

Obtained AS were characterized according to the basic properties affecting the coating process and properties of the final product, *i.e.* monomer conversion, molecular weights and viscosity. The results were presented in Table 2.

Acrylic syrup 819/184 characterized the highest monomer conversion (65.3%), which correlates with photo-DSC

Table 2. Physicochemical properties of obtained acrylic syrups

AS symbol	Monomer conversion %	$M_n$ g/mol	$M_w$ g/mol	$D$	$\eta$ Pa · s
819	60.6	23 000	110 800	4.82	11
TPO	32.0	26 500	129 100	4.88	1
127	52.8	84 700	231 500	2.73	22
184	45.7	94 000	342 700	3.64	13.5
819/184	65.3	35 800	160 400	4.48	32
TPO/184	36.2	41 600	171 200	4.11	3



test results (sample achieved the full conversion of monomers in the shortest time). The high reactivity of mixed system 819/184 can be explained by generation of six various radicals (four from Omnirad 819 and two from Omnirad 184). It caused the relatively high photopolymerization rate. It is worth mentioning that, FRBP process is fundamentally different from film photopolymerization (like in case of photo-DSC) in that it takes place in a large volume and the sample is mechanically mixed during the process. For this reason, radicals and macro-radicals have more possibilities to react, and the effect of the increase in viscosity is no longer as strong. Sample 819/184 characterized also the highest viscosity (32 Pa·s) and one of the largest polydispersity (4.48). Probably, it is caused by effect of six different radicals in system. Generally, the viscosity of ASs increased with increase of monomer conversion. The lowest value of viscosity (1 Pa·s) was for AS with TPO and monomer conversion reached only 32%. Also sample TPO/184 exhibited low values of viscosity and monomer conversion (3 Pa·s and 36.2%, respectively). It is interestingly, that ASs obtained using acylphosphine oxide were so different. AS 819 achieved very high monomer conversion (60.6%) and relatively high viscosity (11 Pa·s), whereas AS with TPO – 32% of monomer conversion and the lowest viscosity (1 Pa·s). It is known from the literature that Omnirad 819 strongly absorbs UV radiation than Omnirad TPO and is twice as effective. NMR analysis of acrylic syrups (*i.e.* monomer conversion) showed differences between film photopolymerization (photo-DSC results) and FRBP process. In film photopolymerization, Omnirad TPO was more effective than Omnirad 819 (time to achieved the full conversion of monomers was short in comparison to sample with Omnirad 819, Fig. 2). Hence it can be concluded that until now photoinitiators dedicated to photopolymerization processes occurring in a thin film are not suitable for processes occurring in large volumes. Moreover, it was stated that monomer conversion was improved in case of systems with two kind of PIs (acylphosphine oxide and  $\alpha$ -hydroxyketone). Nevertheless, it should be noted that ASs with  $\alpha$ -hydroxyketones Omnirad 127 and Omnirad 184 characterized by relatively high monomer conversion (52.8 and 45.7%, respectively) and high viscosity (22 and 13.5 Pa·s, respectively). In the case of these photoinitiators, the principle that more functional photoinitiator is more effective (Omnirad 127 is bifunctional particle) was confirmed. Additionally, it should be emphasized that copolymers obtained using  $\alpha$ -hydroxyketones characterized by significantly higher values of  $M_n$  (84 700 and 94 000 g/mol) and  $M_w$  (231 500 and 342 700 g/mol) and lowest polydispersity (2.73 for Omnirad 127 and 3.64 for Omnirad 184) than other copolymers. Significantly low values of  $M_n$  and  $M_w$  were noticed for AS with acylphosphine oxides (819 and TPO). To sum up, the physicochemical characteristic of obtained acrylic syrups exhibited that  $\alpha$ -hydroxyketones Omnirad 127 and Omnirad 184 were more suitable for FRBP process of acrylic monomers because give AS with relatively high monomer conversion (*ca.* 50%), viscosity

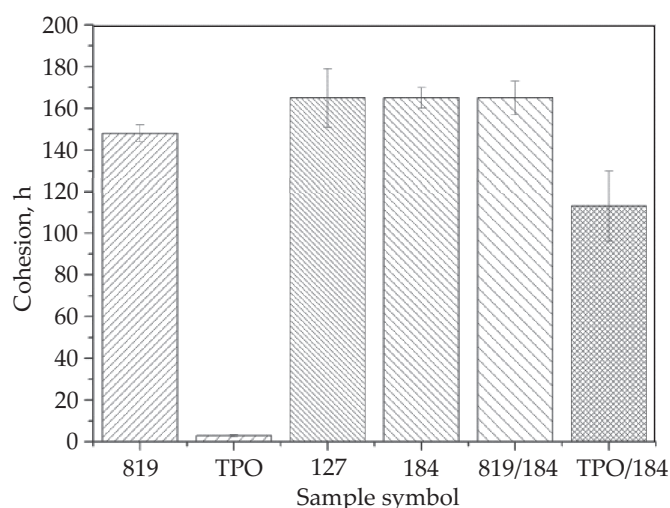


Fig. 3. Cohesion of acrylic syrups based PSAs

and molecular weight ( $M_n$  and  $M_w$ ) and comparatively low polydispersity.

Regarding the self-adhesives properties of PSAs based on AS (cohesion, adhesion to steel and tack), it should be noted that all PSAs, reached cohesion values more than desirable (*i.e.* 72 h), except PSA TPO (only 3 h), Fig. 3.

The highest value of cohesion exhibited PSAs based on ASs with acylphosphine oxides (Omnirad 127 and Omnirad 184) and one of the mixed systems (819/184), the cohesion values were more than 165 h. Additionally, very good result of cohesion was for PSA 819 (148 h). It is also worth noting that the samples obtained from syrups with higher monomer conversion and higher  $M_n$  and  $M_w$  were characterized by higher cohesion. Samples PSAs TPO and PSA TPO/184 were the weakest in this respect. It can also be caused by the fact that the amount of photoinitiator (Irgacure 2022) for UV-crosslinking of these systems was insufficient (*ca.* 60% of unreacted monomers in adhesive composition). In the case of adhesion to steel (Fig. 4) and tack (Fig. 5) of tested PSAs, the highest values of these features were noted for PSA TPO (adhesion – 12 N/25 mm and 27 N – tack). Generally, for PSA characteristic is that the

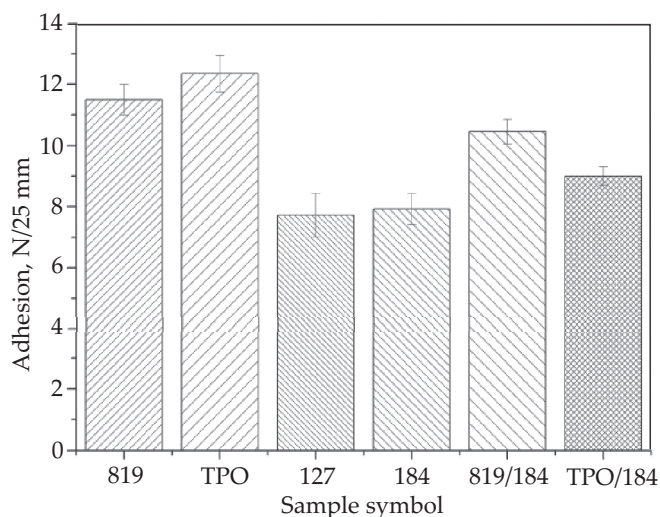


Fig. 4. Adhesion to steel of acrylic syrups based PSAs

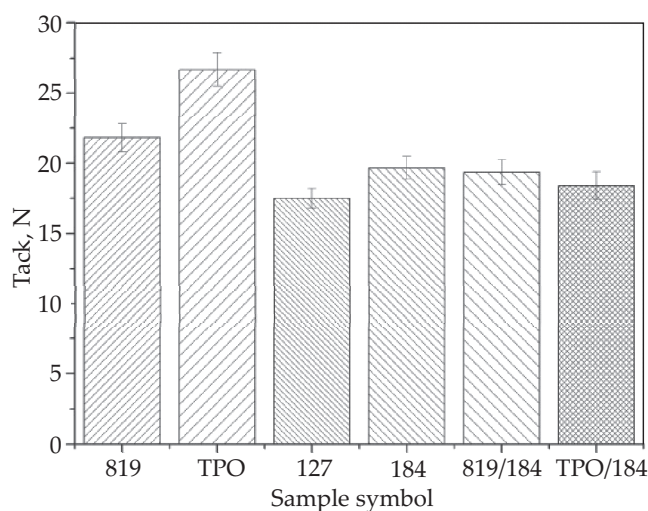


Fig. 5. Tack od acrylic syrups based PSAs

adhesive films with a low degree of crosslinking, low molecular weights of polymers from which they are formed are exhibit low cohesion and high adhesion. Despite the fact that samples PSA 127 and PSA 184 had the lowest adhesion (7.7 N/25 mm and 7.9 N/25 mm, respectively) and tack (17.5 N and 19.6 N, respectively), they exhibited very high cohesion (165 h) so the obtained result of self-adhesive features was advantageous. Nevertheless, the sample with the mixture of photoinitiators (PSA 819/184) turned out to be the best self-adhesive features (10.4 N/25 mm – adhesion, 19.3 N – tack and 165 h – cohesion).

## CONCLUSIONS

The influence of  $\alpha$ -hydroxyketones, acylphosphine oxides and mixtures thereof on photopolymerization kinetics and features of acrylic syrups and self-adhesive properties of the pressure-sensitive adhesives was investigated. Thus, the following conclusion can be drawn:

- Mixtures of acylphosphine oxides and  $\alpha$ -hydroxyketones (*i.e.* Omnirad 819/Omnirad 184 and Omnirad TPO/Omnirad 184) more effectively initiate the film photopolymerization process (photo-DSC results) of (meth)acrylate monomers, probably it is connected with absorption characteristics of the photoinitiators mixtures with monomers. Full conversion of monomers can be reached after 26 s and 30 s, respectively. Although, the highest maximum  $R_p$  exhibited acylphosphine oxides (Omnirad 819) the time to full monomer conversion was almost the longest (170 s). Generally acylphosphine oxides proved less effective in the film photopolymerization process of (meth)acrylates.

- Mixture of Omnirad 819/Omnirad 184 and Omnirad 819 were more effective in the FRBP process (monomer conversion in excess of 60%), nonetheless  $\alpha$ -hydroxyketones provided acrylic syrups with higher  $M_n$ ,  $M_w$  and viscosity and less polydispersity while maintaining the high level of monomer conversion (*ca.* 50%).

- The viscosity and monomer conversion in acrylic syrups and  $M_n$  and  $M_w$  of copolymers have a major impact on the self-adhesive properties of PSAs. Preferable

results were obtained for those with higher  $M_n$ ,  $M_w$  and high viscosity and monomer conversion.

Free radical bulk photopolymerization process is very fast and comfortable method of obtaining pressure-sensitive adhesives, however choosing a radical photoinitiator is crucial.

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