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Study of UV-initiated polymerization and UV crosslinking of acrylic monomers mixture for the production of solvent-free pressure-sensitive adhesive films

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ABSTRACT

UV-polymerizable and UV-crosslinkable pressure-sensitive adhesive acrylic compositions are widely applied for high-quality carrier-free films, one-sided or double-sided self-adhesive tapes. Comprehensive studies of the properties of novel acrylic PSAs for carrier-free films or adhesive tapes were carried out. Solvent-free acrylic pressure-sensitive adhesive layers were UV-crosslinked using various photoreactive crosslinking agents. Polymer content during UV-polymerization, prepolymers viscosity, temperature changes of prepolymer during UVinitiated polymerization process were investigated. The chemical structure of prepolymer and polymerized acrylic film were examined using FTIR spectroscopy. Prepolymer mixtures were transferred on the transparent carriers to form 1 mm thick polymer films, and then UV-crosslinked after the addition of the selected acrylic crosslinker. Final adhesive films were then tested to evaluate their functional properties as the tack, peel adhesion, and shear strength at 20 °C and 70 °C. From the results reveal that both concentrations of acrylic acid in the prepolymer, and cross-linking agent in the final polymer layer, have a significant influence on the functional properties of the produced PSA films.

1. Introduction

Over fifty years since their introduction, pressure-sensitive acrylic adhesives have been successfully applied in many fields. They are commonly used in self-adhesive tapes, labels, sign and marking films, protective films as well as in dermal dosage systems for pharmaceutical applications, and in biomedical electrodes [1–3]. Three properties that are useful in characterizing the nature of pressure-sensitive adhesives are tack, peel (adhesion), and shear (cohesion) [4]. The performance of pressure-sensitive adhesives (PSA), such as tack, peel, and shear, based on acrylics synthesized through UV-polymerization of acrylate monomers and UV-crosslinked are to a large degree determined by the kind of acrylic copolymer, and especially by the type and quantity of the crosslinking agents added to the polymerizate [5–7].

Industrial applications and UV technology of UV polymerizable and UV crosslinkable materials have grown tremendously over the past decade. An entire industry has evolved based on the existence of chemicals, which respond to UV radiation [8–10]. The balance between adhesive and cohesive strengths within the crosslinked coatings is critical for the performance of the UV-PSA [11]. UV radiation is used to induce photochemical polymerization and crosslinking of a monomer, oligomer, prepolymer, or pressure-sensitive adhesive formulation containing a certain type of unsaturation, such as an acrylic group, and an appropriate initiator [12]. The result is a combination of high cohesion and adhesion areas in the pressure-sensitive adhesive film [13]. UV technology also found application in the production of modern coatings, with excellent thermal stability and chemical resistance [14]. To get optimal pressure-sensitive performances with UV-crosslinkable

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Received 1 March 2021; Received in revised form 20 October 2021; Accepted 10 November 2021 Available online 11 November 2021 0142-9418/© 2021 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licensex/by-nc-nd/4.0/). adhesives, it is necessary to find process settings that lead to balanced values of tack, peel, and shear resistance for the aimed application [15]. The thermal resistance of PSAs has special attention for many research teams. Bai et al. presented results of the studies of thermal-resistant acrylic adhesive tapes for special applications, UV-cured/heat cured using TMPTA [16]. Scientists and technologists at a lot of famous and significant institutes and companies have had an interest in the radiation radiation crosslinking polymerization and processing of UV-crosslinkable materials, especially UV-crosslinkable pressure-sensitive adhesives based on acrylics [17]. Moreover, dismantlable adhesives and easily removable pressure-sensitive adhesives can be also manufactured using UV-irradiation during the production process [18, 191.

In summary, UV cross-linking technologies concern both the improvement of well-known existing polymer technologies and, for example, the latest hybrid nanocomposite systems [20].

The purpose of this scientific work was to conduct comprehensive studies of novel acrylic pressure-sensitive adhesives, obtained from synthesized solvent-free photoreactive prepolymers. Obtained selfadhesive films can be used for the production of high-performance mounting tapes.

2. Experimental

2.1. Raw materials

All monomers and radical photoinitiators used in the study have the purity of technical grade without additional purification. Butyl acrylate (BA) and acrylic acid (AA) were produced by BASF (Germany). Radical photoinitiators Omnirad 127 and Irgacure BAPO were supplied from iGMResin (Netherland) and BASF (Germany) respectively. Multifunctional photoreactive crosslinking agents dipropylene glycol diacrylate (DPGDA), trimethylolpropane triacrylate (TMPTA) were supplied from Arkema (France) and alkoxylated pentaerythritol tetra acrylate (Photomer 4172F from iGMResin (Netherland).

2.2. Synthesis of photoreactive prepolymers

The prepolymers (AL 1–7) were synthesized from butyl acrylate and acrylic acid by bulk radical polymerization under a nitrogen atmosphere in a 0.25-L glass reactor equipped with a thermometer, nitrogen inlet,

and mechanical stirrer (Fig. 1).

After 10 min of stirring with 200 rpm at the presence of nitrogen, the monomer mixture was exposed to UV radiation within 60 s using a UV lamp with an intensity of 1.6 mW/cm^2 . During the pre-polymerization, an increase of viscosity was observed.

2.3. Composition, viscosity, and polymer content of synthesized prepolymers

The amount of polymer content was determined by weight, after drying the samples for 45 min at 140 $^{\circ}$ C. The viscosity of the investigated solvent-free acrylics pressure-sensitive adhesives was determined with a Rheomat RM 189 from Rheometric Scientific, with spindle No 3 at 23 $^{\circ}$ C. The viscosity concerns the prepolymers containing polymer and residue unreacted monomers. The prepolymers (AL 1–7) composition, polymer content of synthesized prepolymers, and their viscosities were presented in Table 1.

The influence of acrylic acid concentration on polymer content and viscosity of synthesized solvent-free photoreactive acrylic prepolymers were shown below in Fig. 2.

2.4. Temperature during the polymerization process

The temperature changes of the reaction mixture during the UVinitiated polymerization process from start (0 s) to end of pre-

Table 1

Composition of prepolymers and, their amount of polymers content and viscosity.

Composition name	Photoinitiator Omnirad 127	Monomers		Polymer	Viscosity
		BA	AA	content	
	[wt.%]	[wt. %]	[wt. %]	[wt.%]	[Pa·s]
AL 1	0.1	99.0	1.0	6.8	2.6
AL 2	0.1	98.0	2.0	6.9	3.0
AL 3	0.1	97.0	3.0	7.3	4.0
AL 4	0.1	96.0	4.0	7.7	4.9
AL 5	0.1	95.0	5.0	8.5	5.8
AL 6	0.1	94.0	6.0	10.1	7.5
AL 7	0.1	93.0	7.0	12.3	10.1



Fig. 1. Synthesis of photoreactive acrylic prepolymers.



Fig. 2. Polymer content and viscosity of synthesized prepolymer versus acrylic acid concentration.

polymerization (60 s) for all compositions AL 1–7 were shown in Fig. 3.

2.5. Fourier transform infrared spectroscopy analysis

In order to obtain the structure of the polymer chain, the prepolymer was analyzed by Fourier transform infrared spectroscopy analysis using *FT-IR Spectrum 100* spectrophotometer from PerkinElmer.

Fig. 4 shows the infrared absorption of prepolymer AL-5 (95 wt% BA and 5 wt% AA) after 60 s conducted UV-initiated polymerization in an interesting area with peak 1408 cm⁻¹ characterized for the double bond in butyl acrylate. The peaks at 1637 and 810 cm⁻¹ also represents the absorbance of C=C double bonds of acrylic prepolymer. The strong signal at 1730 cm⁻¹ is related to ester group C = O of the acrylate.

2.6. Preparation of self-adhesive films through UV-polymerization and UV-crosslinking of photoreactive acrylic prepolymers

The solvent-free UV-crosslinkable self-adhesive layers were modified by the addition of 1.0 wt% photoinitiator Irgacure BAPO according to prepolymer content, and multifunctional acrylates used as photoreactive crosslinking agents DPGDA, TMPTA, and Photomer 4172F (Table 2) in concentration between 0.1 and 0.5 wt% according to



Fig. 3. The temperature during UV-initiated polymerization versus polymerization time and acrylic acid concentration.

prepolymer content by step of 0.1 wt%.

After modification, the prepared acrylic prepolymers were coated directly on siliconized polyester film (carrier-free samples for elongation evaluation) and on polyester film (samples in form of one-sided tapes for evaluation of tack, peel adhesion, and shear strength) with about 1 mm thick layer prepared by the use of own manufactured coating machine (Fig. 5).

The surface of the coated layer is coat at the top with siliconized polyester film to protect acrylic prepolymer from oxygen inhibition during UV polymerization. After that the sandwich polyester film-1 mm thick prepolymer-polyester film is polymerized and crosslinked using the same lamp with an intensity of 1.6 mW/cm² like by prepolymerization step. Die polymerization and crosslinking process go on 10 min on both sides (Fig. 6).

2.7. Fourier transform infrared spectroscopy analysis

In order to obtain the structure of the polymer chain after UVinitiated polymerization and UV-initiated crosslinking, the selfadhesive layer was analyzed again by Fourier transform infrared spectroscopy analysis (Fig. 7) which shows not more the peak at 1408 cm⁻¹ characterized for the double bond in butyl acrylate from sample AL-5. Other peaks attributed to double bonds also disappeared, indicating that the adhesive AL-5 was cross-linked.

2.8. Storing and properties of investigated PSA layers

The final PSA properties were determined in the form of carrier free or one-sided self-adhesive 1 mm thick adhesive layers. Before testing, the adhesive layers were stored for 7 days at room temperature and 50% relative humidity. After storing the samples were tested and the given value of tack, peel adhesion and shear strength which was the threearithmetic mean of the results obtained.

2.9. Evaluation of tack, peel adhesion and shear strength of acrylic PSA layers

Tack, peel adhesion and shear strength were determinate by standard A.F.E.R.A. (Association des Fabricants Europeens de Rubans Auto-Adhesifs) procedures. Exact details can be found in AFERA 4015 (tack), AFERA 4001 (peel adhesion) and AFERA 4012 (shear strength). Administrative address: 60, rue Auber-94408 Vitry Sur Seine Cedex (France). The tests were conducted with Zwick/Roell Z-25 testing machine.

2.10. Tack

The tack of pressure-sensitive adhesives is the adhesive property related to bond formation. Generally, the test procedure to measure tack consists of two steps: bond formation and bond separation and to be measured at low contact pressures and short contact times. The tack method measures the instantaneous adhesion of a loop of adhesivecoated material using no external pressure to secure contact. According to another definition, the quick stick tack value is the force required to separate at a specific rate a loop of material, which was brought into contact with a standard surface. The tack method according to AFERA 4015 is relatively simple and may be carried out using common tensile strength test machines. A sample of PSA-coated material 1-inch (about 2.5 cm) wide and about 7-inch (about 17.5 cm) long is bonded to a vertical of a clean steel test plate at least 10 lineal cm in firm contact. The vertical steel test plate is clamped in the jaws of a Zwick/Roell tensile testing machine. The scale reading in Newtons is recorded as the tape is peeled from the steel surface with a constant rate of 100 mm per minute. The tack values of polymerized PSA layers are presented in Figs. 8-10.



Fig. 4. Fragmentary IR spectrum for prepolymer AL-5.

Table 2

Chemical structure of investigated multifunctional acrylates.



2.11. Peel adhesion

The peel adhesion is the force required to remove a coated flexible

pressure-sensitive adhesive sheet material from a test panel measured at a specific angle and rate of removal. For 180° peel measurements (AFERA 4001) the results depend on the face stock material. A sample of



Fig. 5. Own constructed laboratory coating machine.

PSA-coated material 1-inch (about 2.5 cm) wide and about 5-in (about 12.7 cm) long is bonded to a horizontal target substrate surface of a clean steel test plate at least 12.7 cm in firm contact. A 2 kg hard rubber roller is used to apply the strip. The free end of the coated strip is doubled back nearly touching itself so the angle of removal will be 180°. The free end is attached to the adhesion tester scale. The steel test plate is clamped in the jaws of a tensile testing machine Zwick/Roell, which is capable of moving the plate away from the scale at a constant rate of 300 mm per minute. The scale reading in Newtons is recorded as the tape is peeled from the steel surface. The data is reported as the average of the range of numbers observed during the test. The given result is an arithmetic average from the available peel adhesion results. The peel adhesion values of polymerized PSA layers are illustrated in Figs. 11–13.

2.12. Shear strength

The shear strength is a measure of the cohesiveness or internal strength of an adhesive. It is based upon the value of force required to pull an adhesive strip from a standard flat surface in a direction parallel to the surface to which it has been affixed with a definite pressure, according to AFERA 4012, at 20 °C and at 70 °C. It is measured in N required to pull a standard area of adhesive-coated sheet material from a

stainless steel test panel under the stress of a load between 5 and 90 N (tested at 20 °C) and between 5 and 40 N (tested at 70 °C). Static shear strength is the adhesive's ability to maintain its position when shearing forces are applied. Each test is conducted on an adhesive-coated strip applied to a standard stainless steel panel in a manner such that a 1-inch \times 1-inch (about 2,5 cm \times 2,5 cm) portion of the strip is in fixed contact with the panel with one end of the strip being free. The steel panel with the coated strip attached is held in a rack such that the panel forms an angle of 180° with the free end tape extended; the latter then is loaded with a force of different hanging weight in g or kg applied from the free end of the test strip. The time elapsed for each coated film to separate from the test panel was 4 h (Fig. 14).

The results of shear strength measured at 20 $^\circ C$ and 70 $^\circ C$ are presented in Figs. 15–17.

3. Results and discussion

3.1. Composition, viscosity, and polymer content of synthesized prepolymers

During the preliminary tests, a significant effect of acrylic acid concentration in monomer mixture both on the increase in prepolymer viscosity (from 2.6 to 10.1 Pa s), and on the increase in polymer content (from 6.8 to 12.3 wt%) was observed (Fig. 2). This can be explained by the high reactivity of the acrylic acid monomer.

Further increasing of polymer content by bulk polymerization provides for gelation of prepolymers composition (Trommsdorff effect).

3.2. The temperature during the polymerization process

Fig. 3 is a graph illustrating the relationship between varying UVinitiated polymerization time and the temperature during the polymerization process, for the samples with different acrylic acid monomer concentration. The observed temperature increase was lower than expected. It should be noted that with increasing polymerization time the temperature of the prepolymer increase, but only to about 40 °C (for the maximum allowable AA concentration) what does not require cooling of the polymerization reactor.



Fig. 6. Both sides polymerization and crosslinking of acrylic prepolymer using UV lamps.



Fig. 7. Fragmentary IR spectrum for acrylic polymer layer AL-5.



Fig. 8. Effect of difunctional monomer DPGDA on the tack of investigated PSA layers.

3.3. Fourier transform infrared spectrum analysis of prepolymers

Fig. 4 shows the infrared absorption of prepolymer consisting of butyl acrylate and acrylic acid. As shown in the figure, the characteristic peaks of acrylic monomer were obviously ester group at 1730 cm⁻¹ and double bonds of acrylic prepolymer 1637 cm⁻¹ and 810 cm⁻¹ including butyl acrylate double bond C=C at 1408 cm⁻¹.

3.4. Fourier transform infrared spectrum analysis of polymerized PSA layers

Fig. 7 shows the infrared absorption of UV-polymerized PSA layers containing incorporated into polymer chain acrylic acid and butyl acrylate. The peak at 1730 cm^{-1} is characteristic for the ester group, the peak at 1408 cm^{-1} for the double bond in the structure of butyl acrylate



Concentration of trifunctional monomer TMPTA [wt.%]

Fig. 9. Effect of trifunctional monomer TMPTA on the tack of investigated PSA layers.

is not more present, as well as peaks at 1637 cm^{-1} and 810 cm^{-1} . It proved that all butyl acrylate from prepolymer is an integral part of polymerized PSA layers as poly (butyl acrylate).

3.5. Evaluation of tack of PSA acrylic layers

Crosslinking process by using UV radiation leads immediately to a strong decrease in tack. The application of three different photoreactive crosslinking agents (multifunctional acrylates) and the influence of their concentration on tack were shown in Figs. 8–10. With the increasing concentration of di-, tri- or tetra-functional monomers and increasing acrylic acid concentration, the tack decreases. The highest tack values are observed for 0.1 wt% tested functional monomers, especially for difunctional monomer DPGDA (relatively low crosslinking density) and for PSA layers containing 1 wt% acrylic acid. Increasing acrylic acid







Fig. 11. Peel adhesion of PSA layers versus DPGDA concentration.



Fig. 12. Peel adhesion of PSA layers versus TMPTA concentration.



Concentration of tetrafunctional monomer Photomer 4172F [wt.%]





Fig. 14. Own constructed machine for evaluation of shear strength at 20 $^\circ\text{C}$ and 70 $^\circ\text{C}.$



Fig. 15. Effect of concentration of DPGDA on shear strength at 20 $^\circ\text{C}$ and 70 $^\circ\text{C}$ of PSA layers.



Fig. 16. Effect of concentration of TMPTA on shear strength at 20 $^\circ\text{C}$ and 70 $^\circ\text{C}$ of PSA layers.



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Fig. 17. Effect of concentration of Photomer 4172F on shear strength at 20 $^\circ C$ and 70 $^\circ C$ of PSA layers.

amount in acrylic PSA compositions increases the T_g and with it, their tack decreases. The tack depends both on the interfacial interactions (intermolecular forces, electrostatic forces and hydrogen bonds), as well as on the rheological properties of the adhesive layer (viscoelasticity of distorted layer), as concluded Zhu et al. [21] in their studies of similar adhesive system. They suggest reducing the mobility of the polymer chains, which reduces the ability of PSA to form the bond to the test surface. This hypothesis seems to be correct.

3.6. Evaluation of peel adhesion of PSA acrylic layers

The significant decrease of peel adhesion with increasing concentration of crosslinking agent is shown in Figs. 11–13. The decrease of peel adhesion with increasing density of cross-linkage is a general rule in pressure-sensitive adhesives technology, but in this case, is to be noticed a rapid-break-down of this value within the crosslinking agent concentration range from 0.1 to 0.5 wt%. Changes of the crosslinking agent functionality from di-to tetra-functional and increasing concentration of acrylic acid (carboxylic groups amount) in polymerized PSA layers have also a significant influence on the peel adhesion. The use of difunctional DPGDA results in the best peel adhesion performance of investigated acrylic PSA layers (Fig. 11). Comparing these results with the studies by Ghim et al. [22]on a similar BA-AA system, but the solvent-borne, one can notice the difference in the effect of AA content on peel adhesion. They received an increase in the peel to 3% AA content followed by a gentle decrease. In our case, the peel decreases evenly with increasing AA content. These results appear to be typical of most UV cross-linked acrylic PSAs.

3.7. Evaluation of shear strength of PSA acrylic layers

Figs. 15-17 shows the effect of various amounts of used multifunctional photoreactive crosslinking agents in the shear strength of acrylic PSA layers at different concentrations of acrylic acid incorporated during the UV-initiate polymerization into the polymeric chain. As would be expected, the conditions under which peel adhesion and tack are lowest are ideal conditions to achieve strong cohesion of tested samples. This applies to the type and concentration of the crosslinking agent, as well as to the acrylic acid monomer concentration. The amount of crosslinking agent and hence the crosslinkage density always increases the internal cohesion of the adhesive layer. As can be seen in these Figures, from the shear strength results at 20 °C and 70 °C, the most efficiently crosslinked acrylic adhesive will be observed for PSA crosslinked with tetrafunctional Photomer 4172F. The internal strength of the adhesive layer is always related to the cross-link density of the polymer and in the case of the cross-linking agents used in the research, the obtained results could be expected. The photomer 4172F with the most branched structure had the best ability to form a strong adhesive layer. The measured cohesion values were very high, at 20 °C 40 N and 70 °C 120 N.

4. Summary and outlook

In summary, the results of the work concern the reaction of the prepolymer, the temperature during the synthesis of the prepolymer, the viscosity of the prepolymers, and the polymer content in the prepolymer samples due to the concentration of carboxylic groups (acrylic acid amount). The second part of the research was to check the effect of the type and concentration of the cross-linking agent from the group of photoreactive acrylates on the performance properties of the obtained self-adhesive adhesives.

From the studies reveal that AA concentration in prepolymer has a significant effect on the functional properties of the produced adhesive layers. Moreover, the same effect has been observed in relation to the type and concentration of the crosslinking agent.

It can be concluded that the properties of crosslinked PSA layers such as tack and peel adhesion are inversely proportional to the concentration of crosslinking agent after the UV-initiated polymerization process, while shear strength is directly proportional to it.

It should be noticed that the temperature into prepolymers was unexpected relatively low from room temperature to about 40 $^{\circ}$ C what allows for the opinion that it will be possible to conduct the process in an industrial scale. The next steps planned in our studies concern evaluation of the influence of UV-radiation during the polymerization process on acrylic PSA layers performance, thus it will be presented in the next publication.

Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

CRediT authorship contribution statement

Hyun-Joong Kim: Conceptualization, Methodology, Formal analysis. **Zbigniew Czech:** Conceptualization, Methodology, Writing – original draft. **Marcin Bartkowiak:** Investigation, Writing – review & editing. **Gyu-Seong Shim:** Investigation, Visualization. **Janina Kabatc:** Formal analysis. **Adam Licbarski:** Investigation, Visualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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