PROGRESS IN COLOR, COLORANTS AND COATINGS

available online @ www.pccc.icrc.ac.ir

Ministry of Science, Research and Technology
Institute for Color Science and Technology

Progress in Color Colorants Coating 15 (2022), 295-303

Effect of Different Tackifiers on Emulsion-Based Pressure-Sensitive Adhesive (PSA)

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ARTICLE INFO

Article history:
Received: 02 September 2021
Final Revised: 18 December 2021
Accepted: 29 December 2021
Available online: 20 Apr 2022

Keywords:
Pressure-sensitive adhesive
Acrylate
Tackifier
Emulsion
Terpene phenolic resin rosin ester
Pentaerythritol rosin ester.

ABSTRACT

This work was intended to investigate the effect of various tackifiers (terpene phenolic resin (TPR), rosin ester (RS), and pentaerythritol rosin ester (PERS)) on the main properties of a PSA such as tack, peel, and shear strength. In this experiment, the PSA based on butyl acrylate-vinyl acetate was synthesized. Various characters such as solid content, conversion percentage, viscosity, and particle size were evaluated. Different concentrations of tackifiers were added to the formulation. The results confirm that the RS may be most compatible with the synthesized PSA. In addition, using 7.5 wt % of RS shows a significant change in the tack of the PSA; however, RS causes a sharply declining peel strength from 0.42 to 0.1 N/mm, while by adding TPR (5 wt%) besides PERE (2.5 wt %), the peel strength reaches its maximum level of 1.3 and 1 N/mm, respectively. Surprisingly, increasing the number of tackifiers causes diminished shear strength. Prog. Color Colorants Coat. 15 (2022), 295-303© Institute for Color Science and Technology.

1. Introduction

Pressure-sensitive adhesive is generally known as a non-reactive adhesive that is adhered firmly to a solid surface when low pressure is applied for a short time. Despite some other kinds of adhesives such as epoxy and solvent-based, PSAs do not need solvent (organic), heat, or water to activate. Some of the advantages of this adhesive include instant bonding, bond to dissimilar materials, high adhesion, environmental durability, adhesion at low pressure, good cohesion, good resistance against solar light and humidity. The pressure-sensitive adhesives are classified into five categories: natural rubber, thermoplastic elastomer, silicon-based, polyurethane, and PSA based on acrylate polymers. The acrylate polymers, in particular, emulsion-based, are one of the most widely used polymer classes to produce the adhesive [1-4]. The PSAs have various applications, such as label protective films, tapes, and decorative foils [5].

Today, various methods are utilized to prepare PSAs on an industrial scale, and among all of the emulsion polymerization techniques, due to the environmentally friendly has received more attention [1, 6-8]. Emulsion system composes monomer(s), emulsifier(s), initiator(s) etc. In addition to the mentioned components, some other additives are generally used to improve the properties of PSAs, such as colloidal stabilizers, tackifiers, etc. [9, 10].

The PSAs have some unique properties, while this kind of adhesives may have severe problems, such as

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adhesive failure and cohesive failure. Adhesive failure is related to the interaction between adhesive and substrate. However, the problem in the center of adhesive is called cohesive failure. It is confirmed that both of these problems may be down to the viscoelastic properties. In other words, the viscoelastic properties of polymer-based directly influence tack, peel, and shear strength of adhesive. Tackifiers are the most common additive that can improve viscoelastic properties. In addition, the kind and compatibility of tackifiers are of utmost importance. Incidentally, the more suitable and more compatible tackifiers for various polymers, the higher the viscoelastic properties of the final adhesive [11-14].

Tackifiers are low molecular weight compound (300-2000 g/mol) [15]. They typically have a specific softening point (100 ºC) besides a glass transition temperature (T_g) of 40-50 ºC [16, 17]. The performance of the tackifiers may be affected by compatibility with other ingredients, the concentration of tackifiers, rate of separation, etc. [18, 19]. Tackifiers that are used are typically classified into two categories: natural and synthetic. Rosins, Terpene, and their derivatives have belonged to natural tackifiers [19].

The most critical parameters that should be considered to choose the best tackifier could be molecular weight plus polarity. The compatibility of tackifiers with polymer-based system might significantly influence tack and adhesion. For instance, tackifiers with high aromaticity in their chemical structure are generally utilized for polar polymers, while the suitable choice for nonpolar polymers could be aliphatic resins. In other words, the higher the compatibility of tackifiers with polymer structures, the higher the tack besides adhesion of the adhesives [20].

Terpene phenolic resin (Figure 1) is generally synthesized from the copolymerization of terpene hydrocarbons and phenols. In other words, it is considered a nonpolar compound, and it could be soluble in petroleum solvents, aromatic solvents, minerals, and vegetable oils. However, Terpene phenolic resins seem not to be soluble in polar solvents such as water plus ethanol. As a result, it is crystal clear that it could not disperse very well in water-borne materials. Moreover, this kind of tackifier is highly recommended for chloroprene besides hot melt adhesives [21].

Rosin esters (Figure 2) are known as derivatives of an especial component of Abietic acid (Figure 3). Esterification of abietic acid is carried out to produce the Rosin esters. In addition, the main properties of the rosin esters are firmly down to the kind of alcohol that reacts with abietic acid. Based on the functional groups of these compounds in their chemical structures, they could be compatible with a broad range of polymers like vinyl acetate, acrylics, polyurethane (PUR), styrene-butadiene rubber (SBR) [21].

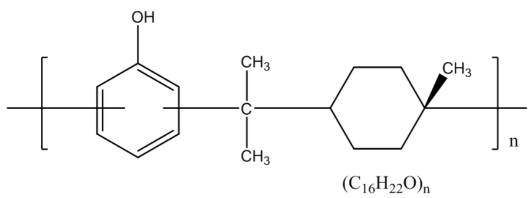


Figure 1: Chemical structure of Terpene phenolic resin.

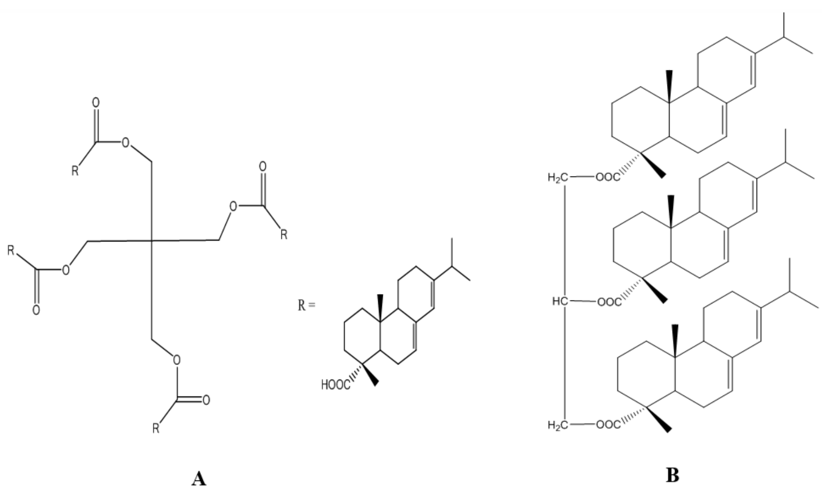


Figure 2: A) pentaerythritol rosin ester and B) Rosin ester.

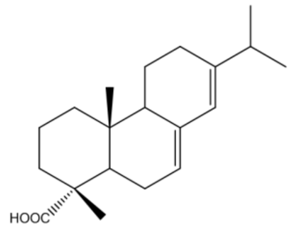


Figure 3: Chemical structure of Abietic acid.

There are two techniques to add tackifiers into the PSA's formulation "Internal" and "External." Adding tackifiers before or during the polymerization reaction is the internal method, and in the external form, the tackifiers are blended [22]. Using the external method may result in phase separation and poor miscibility. Nevertheless, an internal tackifier's adhesives produce acceptable miscibility and immediate adhesion [23].

Many articles focus on the influence of tackifiers on PSAs systems [24-36]. P. Chen and his research group utilized polymerized rosin based on abietic acid that is synthesized in the emulsion polyacrylate adhesive. They studied the effect of tackifier content on the main properties of the adhesive. The results revealed that conversion percentage, average molecular weight, cross-linking density, and gel fraction declined remarkably with the addition of tackifier. This behavior may result from the reaction of abietic acid as a chain transfer agent. However, the polymerized tackifier positively influenced the synthesized adhesive's peel and shear strength [37].

H. Li and his coworkers (2015) prepared a polyacrylate PSA. They used the hydrogenated rosin (HR) as a tackifier. The adhesive was synthesized with a mini-emulsion method. The results showed that with increasing HR, the monomer conversion, Zeta potential, and molecular weight were decreased. Nevertheless, particle size distribution and its polydispersity index were almost constant. Moreover, the HR negatively influenced the centrifugal and storage stability of the mini-emulsion composite [38].

In another experiment, the effect of tackifier on solvent-based polychloroprene contact adhesives was evaluated. Shybi and his coworkers used various amounts of four tackifiers include wood rosin (WD), coumarone-indene resin (CI), terpene-phenolic resin (TP), and para-tert-butyl phenol-formaldehyde (TBPF) resin. This study confirmed that almost all tackifiers might have good compatibility with the adhesive. In addition, besides the peel strength of its adhesive, thermal aging resistance, which includes TBPF were higher than others [39].

The primary purpose of this experiment is to look for a suitable tackifier for a synthesized PSA. The tackifiers composed of TPR, RE as well as PERE. The internal technique utilized the different amounts of tackifiers (0, 2.5, 5, and 7.5 wt %). The effect of the tackifiers on some other parameters such as compatibility, tack, peel, and shear strength is investigated.

2. Experimental

2.1. Materials

A pressure-sensitive adhesive was synthesized based on a copolymer including butyl acrylate-vinyl acetate (Tables 1 and 2). The synthesizing process was carried out in the R&D laboratory of Samed Industry & Manufacturing Co. Tackifiers that used in industrial-grade include Terpene phenolic resin (code: TAMANOL 803L), rosin ester, Pensel GA90, purchased from Arakawa Chemical Industrial LTD, Japan, and Pentaerythritol rosin ester, P100 from Summit Co. China (Table 3).

Table 1: Some main properties of the synthesized latex.

Characters	Value
Conversion percentage	98.30
Solid content (%)	50
Particle size(nm) - pdI	185- 0.376
Viscosity (cP)	6450

Table 2: Characters of the synthesized copolymer (extracted from the latex).

Characters	Value
Copolymer base	Butyl acrylate (BA)-vinyl acetate (VAc)
Copolymer ratio (BA: VAc w/w)	80:20
Tg (ºC)	-28.24
Viscosity average molecular weight ($\overline{M_v}$)	4.36×10^5

Table 3: Properties of the three tackifiers using in the formulation.

Characters	Tackifiers		
	Phenolic terpene	Rosin ester	Penta erythritol rosin ester
color (Gardner)	7 max.	5 max.	2-4
appearance	Pale brown lump	Pale yellowish flake	Pale yellowish transparent granular
acid value(ml/KOH)	45-60	7 max.	20
softening point (℃)	145-160	87-93	97-103

2.2. Equipment

The solid content and conversion percentage were calculated according to the literature [40] by using Oven (D-63450), Heraeus, USA. The polymer film was made via applicator (thickness=200 microns) on PVC substrate to determine the synthesized latex's tack, peel, and shear strength. The film was left for about 24 h to dry completely. The PSA tack was measured using a rolling ball based on ASTM, D–3121 [41]. Also, tensile apparatus ZWICK, Z-020(Ulm, Germany) was used to estimate shear and peel strength according to ASTM 3654 and ASTM 3330, respectively [42, 43]

The viscosity of the latex was measured via Brookfield rotational viscometer (RV DV–II Brookfield), USA, based on ISO 1652 standard [44]. The glass transition temperature (T_g) was evaluated using the DSC technique. Particle size, particle size distribution, and polydispersity index (pdI) of the latexes estimated using the zeta-sizer instrument, Malvern nano-zs, UK. Olympus microscope, model: BX51, Japan. An Ubbelohde viscometer was utilized to calculate $\overline{M}_v$ of the copolymer [45].

3. Result and Discussion

Table 1 demonstrates the main characters of the prepared latex. Moreover, some properties of the synthesized copolymer, such as Tg, besides the viscosity average molecular weight are determined. An anti-solvent method is used to extract the synthesized copolymer solvent. In this method, a particular amount of latex is solved in acetone (or MEK). The solid part is separated from the mixture immediately after adding less water as an anti-solvent. The process is repeated three times to ensure the extracted polymer is pure. Eventually, the polymer is stored at room temperature to dry completely.

3.1. Tackifiers

One of the main parts of this experiment is evaluating the effect of various tackifiers on vital properties of the synthesized PSA include Tack, Peel, and Shear strength. The essential properties and the physical appearance of used tackifiers are shown in Table 3 and Figure 4 (a typical camera takes these photos).

First of all, the compatibility of these tackifiers was examined via a microscope with ×100 magnification. This microscope scans the surface of the latex in which five w% of each tackifier dispersed. Pictures (Figure 5) vividly confirm that the rosin ester-based tackifier (a) might be most compatible with the synthesized latex among these tackifiers. In contrast, terpene phenolic almost did not disperse in the latex (c), which might be primarily due to the chemical structure of each tackifier. The initial characters of the prepared PSA without any additives are shown in Table 4.

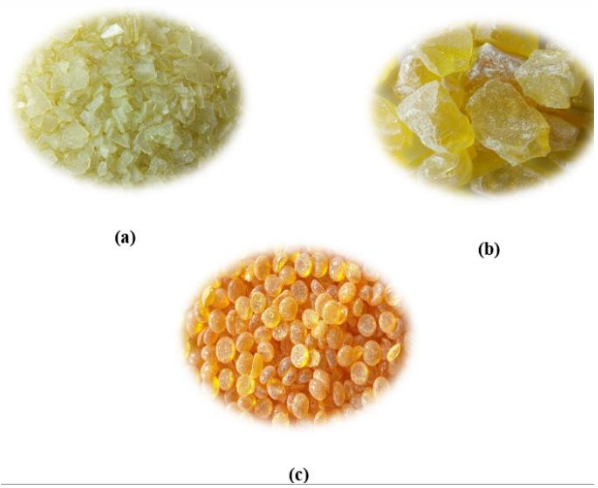
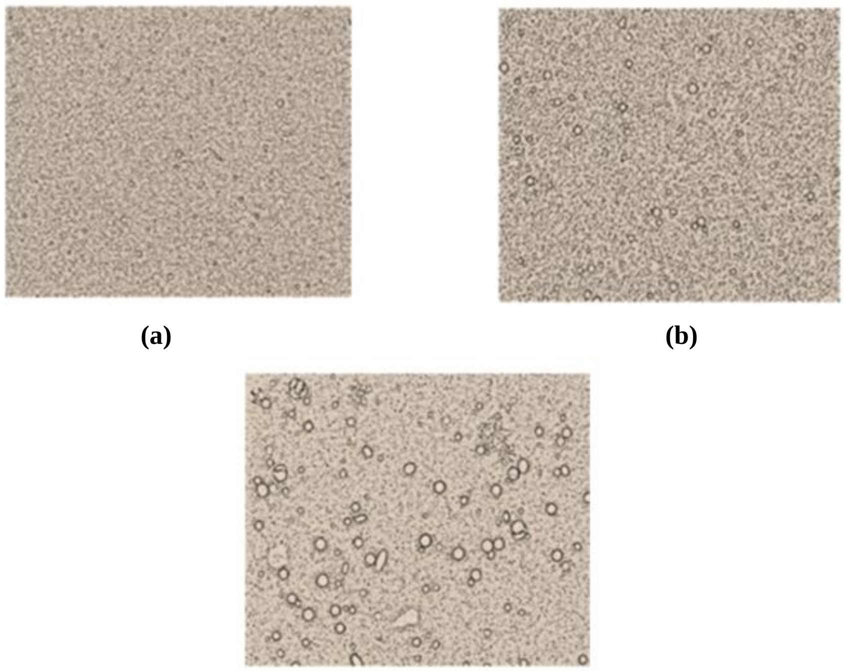


Figure 4: Illustrates physical appearance of the utilized tackifiers a) RS, b) TPR and c) PERE.

Table 4: The prime mechanical properties of the synthesized PSA without any tackifiers.

Characters	Unite	Value
Tack	cm^{-1}	0.13
Peel Strength	N/mm	0.45
Shear Strength	kPa	200

As expected, the tackifier results in improving the tack of the adhesives. This result seems true for only RE (Figure 6). The tackifier concentration increased to 7.5 wt %, caused a continually plunged of the tack, and reached a climax (0.55 cm^{-1}). The tackifier concentration increased to 7.5 wt%, caused a continually plunged of the tack, and got a climax (0.55 cm^{-1}).



(a) **(b)** **(c)**

Figure 5: Pictures from the surface of the synthesized PSA, which including 5 wt % of tackifiers a) RS, b) PERE and c) TPR.

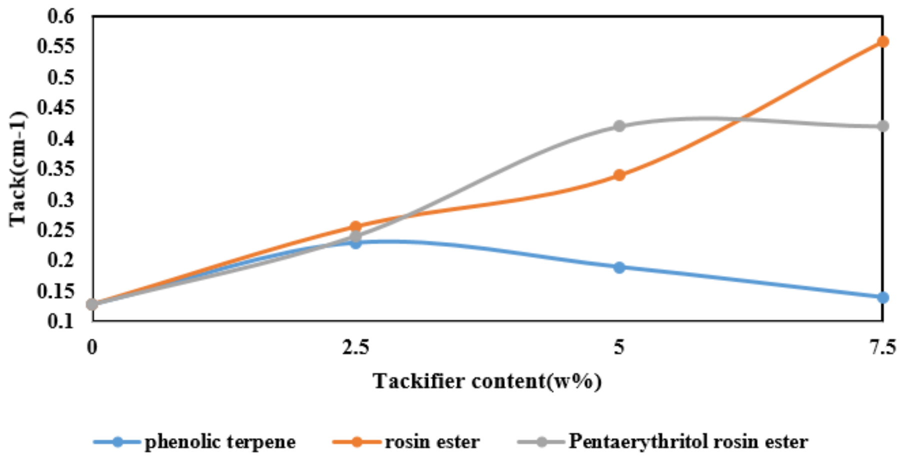


Figure 6: The tack of prepared PSAs versus tackifier content.

According to the figure, the formulation with the highest tack level, which includes TPR, was obtained (just below 0.25 cm^{-1}), while the tackifier concentration was about 2.5 wt %. In addition, the tack of the synthesized PSA is significantly increased (approximately 400 %) with raising the PERE concentration to 5 w %. In follow, tack level out. This analysis confirms that the most critical parameters that affect the function of tackifiers in PSAs might be compatibility with adhesives.

These tree tackifiers may have different influences on the peel strength of the synthesized PSAs (Figure 7). Based on the experimental results, the average peel strength of the adhesive continues the plunging trend by utilizing RE. The peel strength finally reaches the minimum point of well-below 0.2 N/mm. Interestingly, adding 5 wt % of TPR causes peeling strength to get the turning point of about 1.3 N/mm. Also, peeling strength witnessed a dramatic ascend with the addition of 2.5 wt % of PERE (well above 1 N/mm) into the formulation. In follow, peeling strength has declining trend (about 40 %). Beyond any shadow of a doubt, tackifiers as low molecular weight molecules may play a role as a plasticizer that indeed has a devastating influence on the peel strength of the adhesives. According to the results (Figure 4), the RE as a tackifier that has more compatibility (and more interaction) with the PSA could affect the mechanical properties of the PSA remarkably. On the other hand, tackifiers that have less interaction might play a role as a wetting agent that improves the wettability of the PSA in specific concentrations (in 2.5 and 5 wt % for PERE and TPR, respectively). Further increasing of the tackifiers concentration probably results in plasticizers' role in overcoming the wetting agent [46].

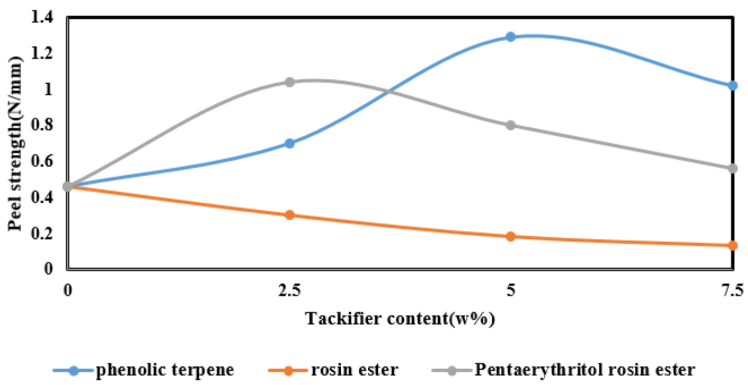


Figure 7: the peel strength of prepared PSAs versus tackifier content**.**

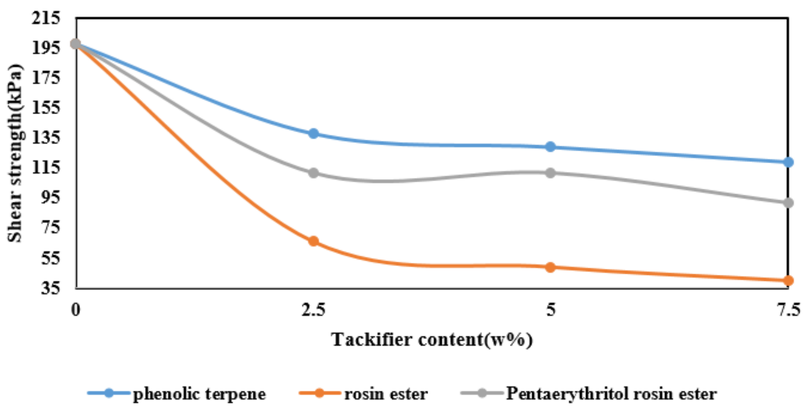


Figure 8: The shear strength of prepared PSAs versus tackifier content.

Figure 8 illustrates the influence of different tackifier content on the shear strength of the synthesized PSA. Based on this figure, it is evident that tackifiers generally harm the shear strength. In fact, with increasing RE content up to 7.5 wt %, the shear strength sharply declined and plunged to the depths of just over 35 kPa. In addition, the shear strength of PSAs, which have 2.5 wt % of TPR plus PERE, significantly diminished and reached 135 and 115 kPa, respectively. Moreover, the falling trend is continued and reached its minimum level while the tackifier content was 7.5 wt %. We can vividly realize that the tackifiers negatively influence the shear strength of the PSAs. As a result, adding these kinds of materials into formulation may cause improving mobility and a decline in the shear strength. In addition, based on Figure 8, the higher the compatibility of the tackifiers with PSA, the lower the shear strength of the polymer. In fact, in this formulation, the order of tackifier's compatibility is as follows (Figure 5): RS>PERE>TPR, so the negative effect of RE is more than the two other tackifiers on the synthesized PSA [47].

4. Conclusion

The PSA based on a copolymer of BA: VAC was synthesized using a semi-batch emulsion polymerization technique. This experiment's primary purpose was to evaluate the effect of various tackifiers, including TPR, RE, besides PERE, on different characters of the synthesized PSA. In this project, various tackifiers concentrations (0, 2.5, 5, and 7.5 wt %) were added to the internal method. The results clearly show that RE has higher compatibility with the PSA among these three tackifiers. Despite the two others, the increasing amount of RE (up to 7.5 wt %) led to continuous improvement of adhesive tack (from around 0.12 to 0.55 cm^{-1}). Moreover, it is generally confirmed that using low molecular weight tackifiers may have a devastating influence on not only the peel strength of the adhesives but also on its shear strength. Therefore, besides the concentration of tackifiers, their compatibility plays a significant role. In other words, the RE, which seems to be the most compatible with the PSA, may result in a sharp decrease in both the peel and shear strength of the prepared adhesive. However, in the formulation including TPR as a tackifier that shows the lowest compatibility level, the peel strength reaches a climax (1.3 N/mm), and shear strength declined slightly compared to the two other used tackifiers.

Acknowledgment

This work was supported by the Department of Chemistry, Faculty of Science, Ferdowsi University of Mashhad (project code: 3/39753), Mashhad, Iran, and Samed industry and manufacturing Co. (Mashad Adhesive), Mashhad, Iran, which are appreciated.

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How to cite this article:
M. Baraghoosh, G. H. Zohuri, M. Behzadpour, M. Gholami, P. Hosseinpour, S. M. Arabi, Effect of Different Tackifiers on Emulsion-Based Pressure-Sensitive Adhesive (PSA). Prog. Color Colorants Coat., 15 (2022), 295-303.