



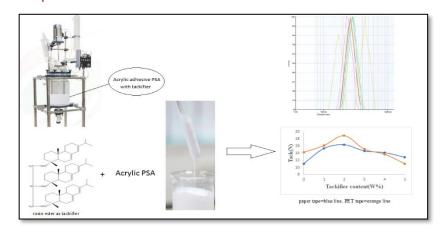
Full Paper | http://dx.doi.org/10.17807/orbital.v17i1.21667

The Influence of Tackifier Concentration on Acrylic Pressure Sensitive Adhesives

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Different combinations of tackifiers were used to create a series of pressure-sensitive adhesives (PSAs) in order to find the optimal balance between peel resistance, tack, and shear resistance. The goal of the study was to analyze how varying levels of tackifier content, specifically rosin ester (RS), impact the key characteristics of an acrylic PSA, such as tack, peel, and shear strength. The addition of rosin ester tackifiers to acrylic PSAs was found to increase the viscosity of the adhesive due to the surface-active nature of the tackifiers. As the concentration of tackifier increased, the number of micelle particles in the solution also increased, leading to higher viscosity and particle size. Increasing the tackifier concentration saturated the active space in the adhesive structure, resulting in the formation of smaller and weaker polymers. This change in the polymerization process led to a decrease in Tack and Peel strength. The study indicated that RS showed the highest compatibility with the synthesized PSA. Interestingly, the inclusion of 2 wt % of RS caused significant changes in the tack and peel properties of the PSA, but it also led to a notable decrease in shear strength.

Graphical abstract



Keywords

Tackifiers
Emulsion polymerization
Rosin ester
Acrylic pressure-sensitive
Adhesives

Article history

Received 07 Aug 2024 Accepted 06 Jan 2025 Available online 01 May 2025

Handling Editor: Sergio C. Araújo

1. Introduction

Pressure-sensitive adhesive, also known as non-reactive adhesive, has the special ability to stick firmly to a solid surface when low pressure is applied for a short period of time. Unlike epoxy and solvent-based adhesives, PSAs do not need solvents (organic), heat, or water to be activated [1-5]. This adhesive provides numerous benefits such as immediate bonding, the capability to bond different materials, strong

adhesion, durability in various environments, bonding under low pressure, good cohesion, and resistance to sunlight and moisture. It is widely used in surface protection films, pressure-sensitive tapes, labels, notepads, medical plasters for skin contact, and other applications [6-9]. Commercial PSAs are made from a variety of polymers, with most falling into six main categories: rubbers (both synthetic and natural),

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polyesters, polyethers, silicones, polyurethanes, and acrylics. The rest of the PSAs are made from esters derived from acrylic and substituted acrylic acids, providing a great combination of performance and affordability [10-12]. The PSAs have distinct features including high transparency, colorless, low toxicity, exceptional resistance to oxidation, UV radiation, organic solvents, temperature changes, and plasticizers. They also show outstanding peel adhesion on polar surfaces [13-16]. Emulsion polymerization has several advantages compared to bulk and solvent polymerization methods for producing this type of PSA. Using water instead of organic solvents not only ensures environmental safety but also takes place within the polymer particles. This allows for the production of polymers with high solid content, faster polymerization rates, and lower viscosity [17-20]. The Pressure Sensitive Adhesive (PSA) needs to have tack, which allows it to form bonds with a surface through brief contact and slight pressure, as well as specific peel and shear resistance characteristics. Peel resistance refers to the force required to remove a tape from a test panel, while shear resistance indicates the ability of the PSA tape to maintain adhesion under a constant load. Tack and peel resistance are related to adhesion forces, while shear resistance is related to cohesion forces. Adhesion and cohesion forces are important in the bonding process, with adhesion being the attraction between two different substances due to intermolecular forces, and cohesion involving the intermolecular attractive forces within the adhesive itself [21-25]. However, the main problem with adhesive performance is known as cohesive failure, which is caused by the viscoelastic characteristics of the materials. In simple terms, the viscoelastic properties of polymer-based materials directly affect the adhesive's tack, peel, and shear strength. Tackifiers are often added to improve these properties, and the type and compatibility of tackifiers are important factors [26-28]. Tackifiers are chemical compounds that give stickiness to rubbery polymers by providing adhesion. While acrylic PSAs already have the right viscoelasticity to be sticky, adding a tackifier can greatly improve their adhesion performance. Rosin esters, as shown in Figure 1, are known as compounds that come from a particular component of Abietic acid and are used as tackifiers in acrylic adhesive. The exact reason for this improvement is not fully understood, as the development of PSAs with tackifiers is usually based on empirical evidence [29-31]. The main goal of this project is to create PSAs for glass bottle labels that meet current market demands, which is a complex task. This study focuses on the impact of rosin ester Tackifier concentration on the functional properties of Irandar Company's acrylic PSA and the impact of tackifier on various parameters including compatibility, tackiness, peel strength, and shear strength and size is being studied.

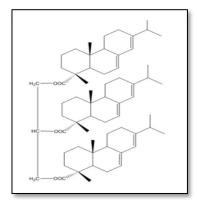


Fig. 1. Chemical structure of rosin ester.

2. Material and Methods

2.1. Materials

Acrylic acid (AA) and n-butyl acrylate (n-BA) provided by BASF (Ludwigshafen, Germany), as well as octadecyl acrylate (ODA) provided by IMCD Benelux B.V. (Amsterdam, The Netherlands), were used as comonomers in the polymerization. Tert-dodecyl-mercaptan (TDM) provided by Chevron Phillips Company LP (Tessenderlo, Belgium) was used as a chain transfer agent. The anionic polymerizable surfactant (S), MaxemulTM 6112, based in a modified alcohol ether phosphate, provided by Croda (Mill Hall, PA USA), was also used in the polymerization. Ammonium carbonate ((NH₄)₂CO₃) provided by BASF (Ludwigshafen, Germany) was used as a buffer and potassium peroxide sulphate (I) supplied by United Initiators (Pullach, Germany) was used as a thermal initiator. A combination of tert-butyl hydroperoxide (TBHP), provided by Pergan (Bocholt, Germany), and sodium formaldehyde sulfoxylate (Bruggolite®), from Brüggemann KG (Heilbronn, Germany), were used as a redox system to reduce free monomer at the end of the polymerization. A 25% ammonia solution, provided by Barcelonesa drugs and chemicals (Cornellà del Llobregat, Spain), was used to neutralize the adhesives. Tackifier that used in industrialgrade include rosin ester (code: AT 880 LG) purchased from Summit Co. China (Table1).

Table 1. Typical properties of AT 880 LG tackifier.

Particle size (nm)	≥008
Coarse particles ppm max	100
Hercules Drop softening point °C	85-95
Total solids content%	54-56%
pH	7-9
Brookfield viscosity mPaS	300≤

2.2. Equipment

Solids Content About 3 g PSA adhesive was heated at 105 °C until constant weight in a DBS 60-3 thermobalance (Kern & Sohn GmbH, Balingen, Germany). Three replicates were carried out and averaged. The average particle size was measured by dynamic light scattering (DLS) with a Zetasizer Nano Series instrument. Samples were prepared by diluting the polymer in deionized water and analyzed at 25°C, using a detector with a 90° angle. The viscosity of the latex was measured via Brookfield rotational viscometer (RV DV-II Brookfield), USA, based on ISO 1652 standard [32]. The adhesive properties were assessed using peel, tack, and shear resistance tests. A motorized laboratory coater, the RK K Control Coater from Lumaguin S.A. in Spain, was used to apply 50 g/m2 of polymer onto tapes with a 50 µm bar. After drying in an oven for 1 minute at 100 °C, a layer of approximately 25 g/m2 of polymer remained on the tapes. Standard-sized tapes were prepared for each test type. Peel resistance, which measures the force needed to remove a tape from a test panel, was evaluated using the 180° peel test after 24 hours. Paper and PET tapes of 275× 25 mm² were applied to glass panels and tested using a Zwick/Roell Z 2.5 tensioner at a speed of 300 mm/min. The average force required to remove the tape and the failure mode were recorded. Tack, the adhesive's ability to bond with a substrate under slight pressure, was determined using the loop tack test with an AT1000 tensile tester. A loop was created with a paper/PET tape of 175× 25 mm² and attached to glass panels. The maximum force needed to peel the tape off the panel and the

failure mode were recorded. Dynamic shear tests were conducted at 5 mm/min using a Zwick/Roell Z 2.5 machine on paper/PET tapes adhered to untreated steel panels at 25 $^{\circ}\text{C}$ with a contact area of 2.5× 2.5 mm². Prior to the test, the sample was pressed four times with a 2 kg rubber roller.

2.3. Sample Preparation

We used emulsion polymerization to prepare samples. The polymerizations were done in a 2.5 L glass reactor at 82 °C with mechanical stirring at 100 rpm using a semi-batch emulsion process. The initial charge in the reactor included 0.3 parts of (NH₄)₂CO₃ per 100 parts by weight of monomer (0.3 phm), 0.1 phm of emulsifier, and half of the total water. After heating and purging the reactor with N2, we added the thermal initiator, followed by the pre-emulsions made up of the emulsifier, monomeric system (Table 2), and the remaining water. We used different quantities of tackifier (0, 1, 2, 3, 4, and 5 wt %) to see how they affected parameters like tack, peel, and shear strength. In the one-step polymerization process, the thermal initiator was added followed by the preemulsion at a steady rate for 3 hours. After the pre-emulsion feed was complete, two shots of 1.0 phm of thermal initiator were added to consume any remaining monomer, with each shot allowed to react for 1 hour. The reactor was then cooled to 57 °C and a redox couple of TBHP/Bruggolite® (0.2 phm/0.3 phm) was added to further consume any residual monomer. Post-polymerization occurred over 4 hours, with gas chromatography analysis showing that the free monomer concentration was below 750 ppm.

Table 2. Initiator (I), surfactant (S), chain transfer agent (TDM), and monomer composition (phm): acrylic acid (AA), n-butyl acrylate (n-BA), octadecyl acrylate (ODA) and Tackifier (T).

Sample	AA	n- BA	ODA	ı	S	TDM	Т
Α	1.75	92.3	3.0	0.5	0.6	0.1	0
В	1.75	92.3	3.0	0.5	0.6	0.1	1%
С	1.75	92.3	3.0	0.5	0.6	0.1	2%
D	1.75	92.3	3.0	0.5	0.6	0.1	3%
Ε	1.75	92.3	3.0	0.5	0.6	0.1	4%
F	1.75	92.3	3.0	0.5	0.6	0.1	5%

3. Results and Discussion

In the dynamic field of polymer science, the development and enhancement of material properties are paramount. This comprehensive study investigates the effects of rosin ester tackifier concentrations on the essential characteristics of acrylic pressure-sensitive adhesives (PSAs), which are synthesized polymers capable of adhering to surfaces under minimal pressure, negating the need for activation by water, solvent, or heat. Acrylic PSAs, produced through the polymerization of acrylic monomers, are distinguished by their transparency, resistance to ultraviolet light, and enduring quality. The experiment meticulously examines three critical properties of these adhesives: Tack, which is the initial stickiness or quick adhesion upon contact; Peel strength, the force needed to remove the adhesive from a surface; and Shear strength, the adhesive's resistance to sliding forces. The methodological framework of the study is detailed in Table 3, summarizing the average particle size and viscosity of the aqueous polymer solution with 50% solids at various tackifier concentrations. These parameters serve as indicators of the polymer's molecular weight and degree of polymerization, which directly affect adhesive performance. A key finding is the direct relationship between increased tackifier amounts and a corresponding rise in particle size (Fig 2), viscosity, and solid content within the acrylic PSAs. This is due to the tackifiers' propensity to induce polymer chain aggregation, resulting in larger particle formations.

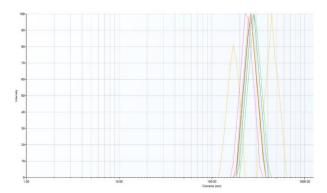


Fig. 2. Particle-size distributions of acrylic PSAs with different tackifier content. (a=yellow, b= Purple, c=red, d=blue, e=green and f=orange lines respectively).

Table 3. Physical characteristics of acrylic pressure-sensitive adhesives with varying levels of tackifier.

Sample	Particle Size (nm)	Viscosity (cp)	Solids Content (%wt)
a=Acrylic PSAs (AC)	158.12	430	55
b=AC+ 1% tackifier	223.14	438	55.2
c=AC+ 2% tackifier	292.45	449	55.5
d=AC+ 3% tackifier	314.25	572	56
e=AC+ 4% tackifier	318.12	768	58
f=AC+ 5% tackifier	456.78	1014	61

Visual data from Figures 3 and 4 illustrate how different tackifier concentrations influence the Tack and Peel strength of the acrylic PSAs when applied to substrates such as PET (polyethylene terephthalate) and paper tapes (Table 4). The data reveals a reduction in both Tack and Peel strength upon increasing the tackifier concentration to 2wt%, indicating an optimal tackifier threshold for maintaining adhesive effectiveness. Furthermore, Figure 5 highlights the negative impact of tackifiers on Shear strength, particularly when the RS content reaches 2 wt%, emphasizing the delicate equilibrium between tackifier content and cohesive strength. The study also explores the variable adhesive performance on different substrates. The porous nature of paper allows for more profound adhesive penetration, enhancing adhesion and resulting in heightened Peel resistance and Tack. In contrast, the less porous PET substrates demonstrate more substantial changes in Shear resistance. This differential behavior underscores the significance of substrate porosity in adhesive performance. This in-depth analysis reveals the complex relationship between rosin ester tackifier concentration and the fundamental properties of acrylic PSAs. It highlights the critical need for precise tackifier content to fine-tune adhesive attributes and the influence of substrate characteristics on performance. These findings are instrumental for the progression of polymer science and the creation of advanced adhesive formulations, providing industry professionals with valuable insights for optimizing product development. This study not only contributes to the existing body of knowledge but also paves the way for future research and innovation in the field of adhesive technology.

Table 4. Adhesive properties determined on both paper and PET tapes. The failure mode is indicated in parenthesis.

		Paper Tape			PET Tape	
Adhesive properties	Peel Resistance (N/25 mm)	Tack (N)	Dynamic shear (N)	Peel Resistance (N/25 mm)	Tack (N)	Dynamic shear (N)
а	0.403	14.23	25.78	0.396	11.05	28.793
b	0.468	16.12	22.14	0.418	15.32	26.992
С	0.558	18.89	20.13	0.494	16.37	26.51
d	0.423	15.12	20.89	0.402	14.63	26.98
е	0.398	13.68	23.87	0.387	14.15	27.15
f	0.345	11.04	23.98	0.335	12.84	27.354

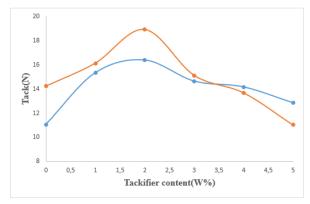


Fig. 3. The tack of prepared PSA versus tackifier content (paper tape=blue line, PET tape=orange line).

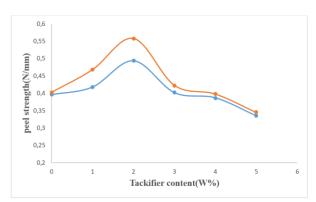


Fig. 4. The peel strength of prepared PSA versus tackifier content (paper tape=blue line, PET tape=orange line).

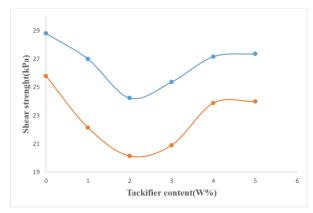


Fig. 5. The shear strength of prepared PSA versus tackifier content (paper tape=blue line, PET tape=orange line).

4. Conclusions

In the intricate domain of polymer science, the optimization of adhesive properties through the precise

manipulation of constituent concentrations is a subject of significant research. This project delves into the realm of acrylic pressure-sensitive adhesives (PSAs), focusing on the systematic incorporation of varying concentrations of rosin ester tackifiers (0, 1, 2, 3, 4, and 5 wt %) to discern their impact on the adhesive's performance. The study meticulously documents the influence of 880 LG tackifier, a specific rosin ester variant, on the adhesive characteristics, revealing a notable trend: as the tackifier concentration escalates up to 2 wt %, there is a discernible decrement in the adhesive's tack and peel strengths. Rosin ester tackifiers are renowned for their role as a fortifying agent within the acrylic adhesive matrix, fostering the formation of polymer links between adhesive particles. These links are pivotal in the adhesive's structural integrity and functional efficacy. However, the research indicates that an augmentation in tackifier concentration leads to a saturation of the active sites within the adhesive structure. This saturation culminates in the generation of smaller and inherently weaker polymers, thereby altering the polymerization process and diminishing the Tack and Peel strength-key parameters in the adhesive's ability to bond and maintain cohesion with substrates. Furthermore, the addition of rosin ester tackifiers is observed to elevate the viscosity of the acrylic adhesive. This viscosity enhancement is attributed to the tackifiers' role as a surface-active agent. With increasing tackifier concentration, there is a proliferation in the number of micelle particles within the solution. These micelles engage in interparticle interactions, which in turn amplify the solution's viscosity and particle size. Such changes in the microstructure of the adhesive are critical as they directly influence the macroscopic properties, including the adhesive's spreadability, stability, and bonding potential. The project's findings underscore the delicate balance required in the formulation of acrylic PSAs. It posits that an optimal concentration of tackifier, identified at 2 wt %, is imperative to harness the best functional properties of the adhesive. This concentration threshold ensures a harmonious balance between the adhesive's tack and peel strengths while maintaining an ideal viscosity that facilitates robust adhesion without compromising the ease of application or the integrity of the adhesive bond.

The study presents a nuanced understanding of the relationship between tackifier concentration and the resultant polymer properties within acrylic adhesives. It highlights the importance of precision in the formulation process, where each component must be carefully calibrated to achieve the desired adhesive performance. These insights not only contribute to the advancement of adhesive technology but also serve as a guideline for future innovations in the development of high-performance, pressure-sensitive adhesive products. The research thus stands as a testament to the critical role of tackifiers in the engineering of adhesives and the broader implications for material science and industrial applications.

Acknowledgments

This work was supported by Irandar industry and manufacturing Co(Irandar Adhesive), Tehran, Iran.

Author Contributions

M Shalbafan: Conceptualization, data curation, formal analysis, methodology, investigation, software, validation, formal analysis, investigation, visualization, resources, writing—original draft, and writing—review and editing. H Bayati: validation, investigation, supervision, and funding acquisition. E Hashemi: validation, investigation.

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How to cite this article

Shalbafan, M.; Bayati, H.; Hashemi, E. *Orbital: Electronic J. Chem.* **2025**, *17*, 80. DOI: http://dx.doi.org/10.17807/orbital.v17i1.21667