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DEVELOPMENT OF BIO-BASED PRESSURE-SENSITIVE ADHESIVE FORMULATIONS DERIVED FROM ROSIN ESTERS USING MIXTURE DESIGN METHODOLOGY

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ABSTRACT

Pressure-sensitive adhesives (PSAs) are commonly used in medical, packaging and industrial applications. Nevertheless, conventional petroleum-based adhesives raise concerns about sustainability, environmental impact and dependence on non-renewable resources. The objective of this study was to synthesize an eco-friendly bio-based pressure-sensitive adhesive from rosin ester by using simplex lattice design (SLD) methodology. The adhesive formulation was comprised of glycerol rosin ester, polyethylene glycol (PEG) rosin ester, lecithin, polyvinyl alcohol (PVA) and borax as cross linking agent. Adhesive performance was evaluated by peel strength, holding power and cohesion analysis. The result indicated that formulations with greater concentrations of glycerol rosin ester showed better adhesive properties with peel strength 0.8 N/cm and holding power 800 s. The optimum formulation by SLD optimization was 9 parts glycerol rosin ester, 7 parts PEG rosin ester and 4 parts lecithin. The proposed bio-based adhesive has not yet exceeded the performance of acrylic-based pressure-sensitive adhesive. However, it demonstrated adequate performance for lightweight labeling. In addition, renewable rosin-based raw materials provide a sustainable option to petroleum-derived adhesives, reducing dependence on fossil resources and driving the growth of green adhesive materials.

Keywords: Bio-based adhesive, Pressure-sensitive adhesive, Rosin ester, SLD

1. Introduction

Pressure sensitive adhesives (PSAs) are an important class of adhesives that have attracted much attention due to their simplicity of application, versatility and ability to enable rapid bonding under light pressure. The presence of these properties has led to their extensive application in various industries, especially in medical devices, wound dressings, transdermal patches, packaging systems, labels and industrial tapes. The increasing demand for high performance adhesive materials has supported continued research to improve the adhesion qualities, cohesion strength, durability and user comfort without sacrifice of process efficiency and cost effectiveness. Most commercial PSAs rely on petroleum-derived polymers and tackifiers, including acrylics, synthetic rubbers, and vinyl-based materials. Although these adhesives exhibit excellent performance, concerns have been raised regarding their dependence on non-renewable resources and potential health and environmental impacts in specific applications.

Acrylic-based adhesives, including methacrylates and epoxy diacrylates, are commonly employed in medical tapes and wound dressings. However, several studies have reported that certain synthetic adhesive components may induce skin irritation, chronic inflammation, and delayed wound healing. Adverse effects of acrylic-based adhesive materials on tissue recovery during wound treatment have been reported in the literature [1], [2], [3]. Similarly, cytotoxic effects have also been observed in other classes of synthetic adhesive. Kaplan and Baysal demonstrated the cytotoxicity of ethyl 2-cyanoacrylate using an elution test system, showing a reduction in cell viability with increasing extract concentrations [4]. Collectively, these findings have

encouraged the development of adhesive systems that utilize renewable and environmentally preferable materials.

One promising approach is the partial substitution of petroleum-derived adhesive components with renewable bio-based materials. Among various renewable resources, rosin has attracted considerable attention as a potential raw material for adhesive applications. Rosin is a natural resin obtained from pine trees and consists primarily of resin acids that can be chemically modified to improve adhesive performance [5]. Rosin-derived esters have been extensively used as tackifiers because of their excellent compatibility with polymer matrices, high tack properties, and renewable origin [5], [6], [7]. Rosin esters are derived from renewable forest resources and can be produced without damaging trees, making them an attractive and more sustainable alternative to petroleum-based tackifiers in adhesive formulations.

The development of bio-based adhesives is a promising route to reduce dependence on petroleum-based products and promote sustainability. These adhesives are derived from renewable biomass resources which have lower environmental impacts in general. They support the principles of environmental engineering, circular bioeconomy and sustainable manufacturing by reducing resource depletion and minimizing life-cycle environmental burdens.

Polyvinyl alcohol (PVA) is another important material commonly employed in environmentally friendly adhesive systems because of its film-forming ability, water solubility, and good mechanical properties [8], [9], [10]. In addition, crosslinking PVA with borax can generate a three-dimensional polymer network that improves structural stability and adhesive performance [11], [12], [13]. Lecithin may also be incorporated as a natural emulsifier to enhance compatibility between hydrophobic rosin esters and aqueous PVA solutions [14], [15]. The combination of rosin esters, PVA, lecithin, and borax offers a promising route for developing sustainable pressure-sensitive adhesive systems.

Rosin-based adhesives have been widely studied. However, the optimization of pressure-sensitive adhesive formulations comprising glycerol rosin ester, polyethylene glycol rosin ester, and lecithin using mixture design methodologies has received limited attention. Therefore, formulation optimization is required to identify the composition that provides the best balance between peel strength and holding power while maintaining a high content of renewable materials.

From the point of view of environmental engineering, the use of compounds produced from rosin in adhesives reduces the dependence on tackifiers derived from fossil fuels and favors more sustainable industrial products. The film forming polymer of the present formulation is still polyvinyl alcohol (PVA), but the partial replacement of petroleum-based components with renewable rosin esters and lecithin is a significant step towards more environmentally friendly adhesive solutions.

Therefore, this study aimed to develop and optimize a partially bio-based and sustainable pressure-sensitive adhesive (PSA) based on rosin esters using a mixture design approach. The adhesive formulation comprised glycerol rosin ester, polyethylene glycol rosin ester, lecithin, polyvinyl alcohol (PVA), and borax as a crosslinking agent. The adhesive performance was evaluated in terms of peel strength, holding power, and cohesion.

2. Materials and Methods

Preparation of Pressure-Sensitive Adhesive (PSA)

The materials used in this study include glycerol (99.8%) was purchased from PT. Wilmar Nabati Indonesia (Indonesia). Polyvinyl alcohol (PVA, BP24, degree of hydrolysis 87.93%) was supplied by Chang Chun Petrochemical Co., Ltd. (Taiwan). Soy lecithin was obtained from Shandong Lanhe Bio-Tech Co., Ltd. (China). Polyethylene glycol 200 (PEG 200) was purchased from BASF (Germany). Disodium tetraborate decahydrate (borax, analytical grade) was obtained from Merck (Germany). Glycerol rosin ester (GRE) and PEG rosin ester (PGRE) were prepared by esterification of rosin with glycerol and polyethylene glycol (PEG), respectively, using methods developed in our laboratory.

Pressure-sensitive adhesive (PSA) was prepared by mixing glycerol rosin ester (GRE), polyethylene glycol rosin ester (PGRE), and lecithin at 90 °C under constant stirring until a homogeneous mixture was obtained. Subsequently, polyvinyl alcohol (PVA) was added to the mixture and stirred continuously for 45 min. After

homogenization, borax was introduced as a crosslinking agent and allowed to react for 15 min. The resulting adhesive solution was cast onto a silicone mold (50 × 150 × 2 mm) lined with release paper and dried to form PSA films. Film thickness was measured using a digital micrometer. The prepared films were subsequently subjected to peel strength, holding power, and cohesion analyses following the procedure reported by Baraghoosh et al. [16]. The total tackifier content, including lecithin as an emulsifier, was maintained at 20 wt.%. The concentrations of PVA (3%) and borax (4%) were fixed at 75 wt.% and 5 wt.%, respectively. Demineralized water was used as the solvent for preparing the PVA and borax solutions.

Experimental Design

The formulation optimization was conducted using a Simplex Lattice Design (SLD) with three mixture components: GRE (X_1), PGRE (X_2), and lecithin (X_3). The investigated composition ranges are presented in Table 1 and 2. The mixture design and statistical analyses were performed using Minitab 22.

Table 1. Independent variables and experimental ranges

Variable	Component (%)	Level	
		Lower (%)	Upper (%)
X_1	Glycerol Rosin Ester (GRE)	7	9
X_2	PEG Rosin Ester (PGRE)	7	9
X_3	lecithin	4	6

Table 2. Experimental formulations for PSA preparation

Run	GRE (%)	PGRE (%)	Lecithin (%)	Borax (%)	PVA (%)	Total (%)
1	7.67	7.67	4.67	5	75	100
2	7.33	8.33	4.33	5	75	100
3	8.33	7.33	4.33	5	75	100
4	7.33	7.33	5.33	5	75	100
5	9	7	4	5	75	100
6	7	7	6	5	75	100
7	7	9	4	5	75	100

The responses evaluated in this study were peel strength and holding power. Analysis of variance (ANOVA) was performed to evaluate the significance of the model and formulation variables.

Peel Strength Test and Holding Power Test

The peel strength test was performed to evaluate the adhesive performance of the PSA films. Test specimens with dimensions of 50 mm × 150 mm were attached to a stainless-steel substrate and peeled at an angle of 180° using a dynamometer. The peel strength was calculated from the average peeling force divided by the specimen width and reported in N/cm. Holding power was determined using the same size of PSA specimens. The PSA is attached at 25 mm from the base of the stainless-steel metal plate with consistent pressure. A 100-g load was then suspended from the free end of the specimen, producing a vertical 180° tensile load. The elapsed time until complete adhesive failure occurred was recorded as the holding power and expressed in seconds.

Cohesion Analysis

Cohesion behavior was evaluated after the peel strength test through microscopic observation of the stainless-steel substrate surface. Surface morphology was examined using a digital microscope. Cohesive failure was identified when adhesive residues remained on the substrate after peeling, indicating that failure occurred within the adhesive layer rather than at the adhesive–substrate interface.

3. Results and Discussion

Characteristics of the Prepared PSA Films

The prepared pressure-sensitive adhesive (PSA) films (Fig.1) exhibited a gel-like appearance due to the crosslinking reaction between polyvinyl alcohol (PVA) and borax. The resulting films were flexible and foldable, indicating the formation of a three-dimensional polymer network capable of maintaining structural integrity while retaining sufficient flexibility for adhesive applications. The incorporation of glycerol rosin ester (GRE), polyethylene glycol rosin ester (PGRE), and lecithin influenced the visual appearance and surface characteristics of the PSA films.

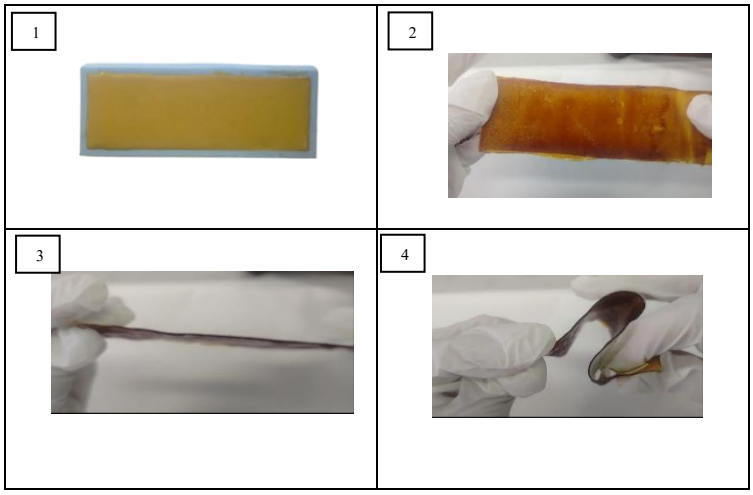

Figure 1. PSA Products

The formation of the gel structure can be attributed to the interaction between borate ions and the hydroxyl groups of PVA chains, producing reversible crosslinks within the polymer matrix [11]. This structure is advantageous for pressure-sensitive adhesives because it provides a balance between cohesion and adhesion. Adequate cohesion prevents excessive flow of the adhesive layer, while sufficient adhesion enables intimate contact with the substrate surface.

Surface Morphology Analysis

The results of surface morphology observations for each experiment (1–7) are shown in Figure 2. There are differences in texture and surface homogeneity of the resulting PSA films. These variations in surface characteristics indicate that the formulation composition influences the formation and distribution of the PSA matrix components.

Figure 2.1 exhibits a relatively smooth gel-like surface with a light-yellow color. The morphology suggests that the adhesive matrix is dominated by PVA, resulting in a less heterogeneous structure. Figure 2.2 shows a rougher surface with numerous agglomerated regions. The presence of these aggregates suggests incomplete dispersion of the components, possibly due to strong interactions between borax and lecithin, leading to localized clustering within the matrix. Figure 2.3 presents a morphology similar to Figure 2.1, although the surface appears slightly darker and rougher. This change may be associated with the incorporation of rosin ester into the formulation. Figure 2.4 displays a more homogeneous gel-like structure with increased color intensity. The improved uniformity suggests better distribution of the formulation components within the adhesive matrix. Figures 2. 5-7 exhibit relatively homogeneous gel-like surfaces with darker brown coloration. The reduction in large agglomerates and the more continuous surface texture indicate enhanced compatibility among the components. The darker appearance is likely related to the increasing contribution of rosin ester in the formulation.

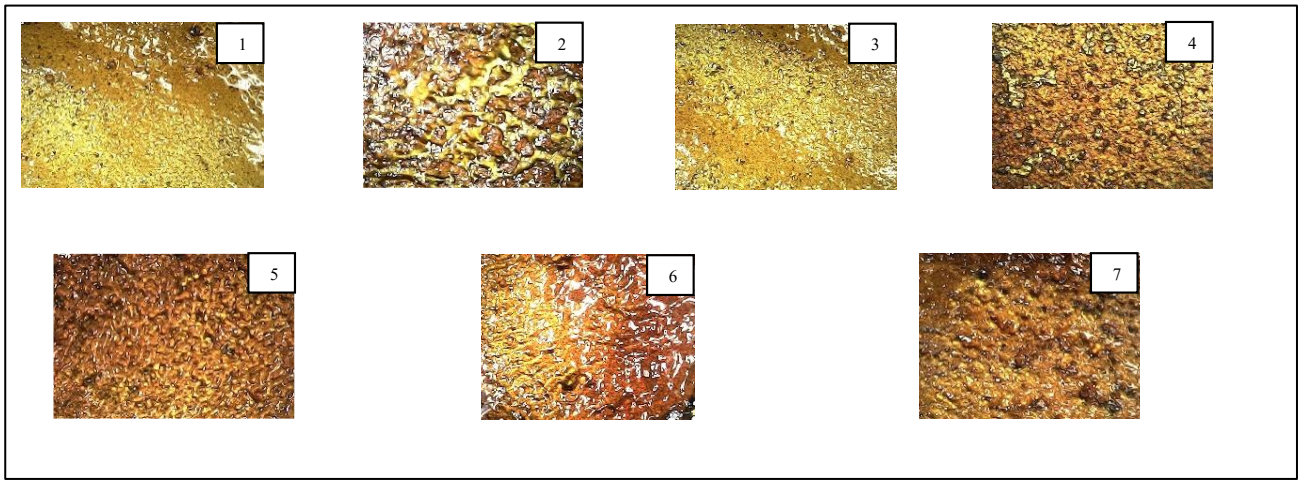

Figure 2. PSA surface Sequence 1-7 represents the mixing conditions as shown in Table 2

Overall, the optical micrographs reveal that variations in formulation composition significantly affect the surface morphology of the PSA. Lower rosin ester formulations tend to exhibit lighter and smoother surfaces, whereas formulations containing higher amounts of rosin ester show darker, denser, and more homogeneous structures. These morphological changes suggest modifications in the internal organization of the adhesive matrix that may influence its adhesive performance.

Peel Strength Analysis

The peel strength obtained from the seven formulations ranged from 0.02 to 0.80 N/cm. The highest peel strength was achieved by formulation 5, containing 9% glycerol rosin ester, 7% PEG rosin ester, and 4% lecithin, which produced a peel strength of 0.80 N/cm. In contrast, formulations with lower glycerol rosin ester contents exhibited substantially lower peel strength values.

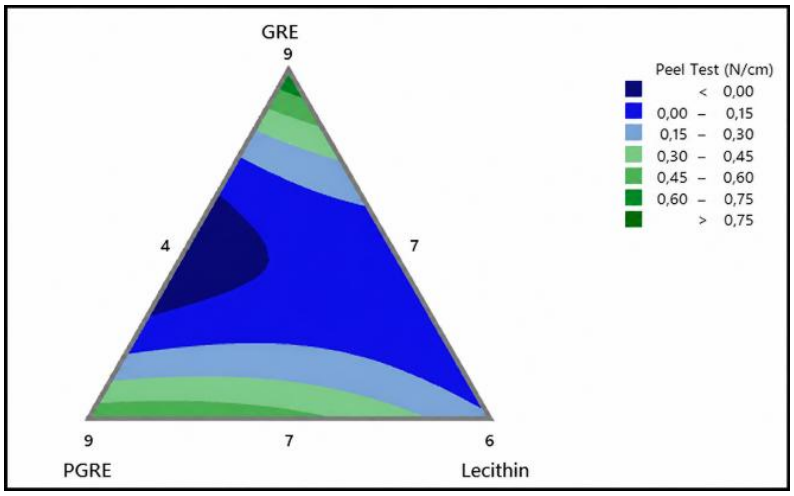

Figure 3. Surface plot of peel strength

The contour plot of peel strength (Figure 3) demonstrates that increasing the proportion of glycerol rosin ester positively influences adhesive performance. Glycerol rosin ester acts as a tackifier by helping the adhesive spread more evenly on the substrate surface, thereby increasing the contact area and improving adhesive strength. Formulations containing relatively high lecithin concentrations showed lower peel strength

values. Although lecithin acts as an emulsifier and improves component compatibility, excessive lecithin may weaken the adhesive matrix, leading to lower peel strength.

The lower peel strength observed in the present study may be attributed to differences in polymer matrix composition. Unlike the acrylic-based PSA system developed by Baraghoosh et al. [16] which is inherently designed to provide high adhesion performance. Nevertheless, the use of renewable rosin-derived materials represents an important advantage from a sustainability perspective and demonstrates the potential of bio-based PSA formulations for future development.

Table 3. ANOVA *Peel test*

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value
Regression	5	0,440	0,440	0,088	0,830	0,677
Linear	2	0,154	0,030	0,015	0,140	0,882
Quadratic	3	0,286	0,286	0,095	0,900	0,631
$X_1 * X_2$	1	0,265	0,073	0,073	0,690	0,558
$X_1 * X_3$	1	0,018	0,021	0,021	0,190	0,736
$X_2 * X_3$	1	0,002	0,002	0,002	0,020	0,907
Residual Error	1	0,106	0,106	0,106		
Total	6	0,546				

$$Y_1 = 4.86\,X_1 + 2.6\,X_2 - 0.53\,X_3 - 0.77\,X_1X_2 - 0.41\,X_1X_3 + 0.14\,X_2X_3 \quad (1)$$

where: Y_1 is the peel strength response, X_1 is GRE, X_2 is PGRE, and X_3 is lecithin

As shown in Table 3, the quadratic mixture model (Eq.1) for peel strength was not statistically significant at the 95% confidence level (P = 0.677). Similarly, none of the linear and interaction terms revealed significant impacts (P > 0.05). This study shows that the modifications of the proportions of the PGRE, GRE and lecithin in the composition range investigated did not generate statistically significant variations on peel strength. However, the model achieved an R^2 value of 80.6%, indicating that the model explained 80.6% of the variability in the experimental data. Therefore, although the model cannot be used to prove statistically significant factor impacts, it is still useful for identifying formulation trends and guiding optimization.

Holding Power Analysis

Holding power values ranged from 16 to 800 s (Figure 4) depending on formulation composition. The highest holding power was again observed for formulation 5, indicating that this composition provided the most balanced adhesive structure. The contour plot revealed that increasing glycerol rosin ester concentration enhanced holding power. This behavior suggests that glycerol rosin ester not only improves adhesion but also contributes to internal cohesion within the adhesive matrix. Stronger intermolecular interactions among adhesive components increase resistance to creep deformation under constant loading conditions.

In contrast, the higher lecithin formulations demonstrated reduced holding power. Large levels of lecithin may interfere with the molecular connections in the adhesive matrix, reducing the internal strength of the adhesive and leading to premature failure under continual strain. Similarly, PEG rosin ester is a good tackifier that improves surface wetting and initial adhesion. However, at high concentrations, the flexible PEG chains increase the mobility of the polymer chains, resulting in a weaker and less cohesive adhesive network. Consequently, the creep deformation of the adhesive is increased, and the holding power is reduced.

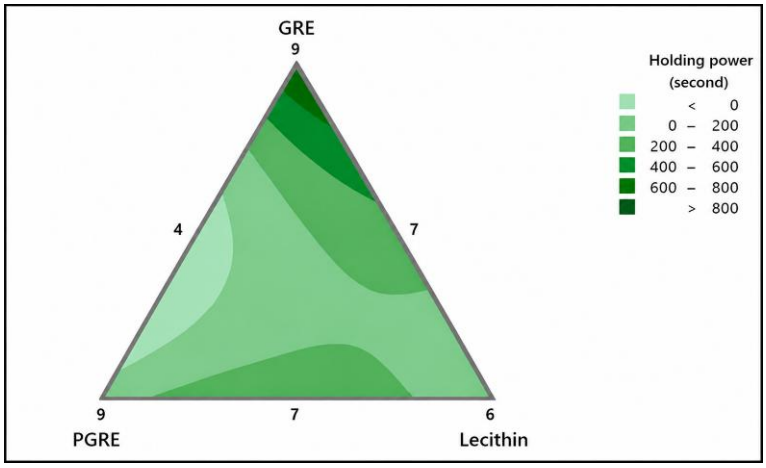

Figure 4. Surface plot of holding power

Table 4. ANOVA *Holding power*

Source	*DF*	*Seq SS*	*Adj SS*	*Adj MS*	*F-Value*	*P-Value*
Regression	5	430795	430795	86159	42,39	0,116
Linear	2	364399	30402	15201,2	7,48	0,25
Quadratic	3	66396	66396	22132	10,89	0,218
$X_1 * X_2$	1	58402	39940	39939,6	19,65	0,141
$X_1 * X_3$	1	62	863	862,6	0,42	0,632
$X_2 * X_3$	1	7932	7932	7931,9	3,9	0,298
Residual Error	1	2033	2033	2032,8		
Total	6	432828				

$$Y_2 = 3357.63\,X_1 + 1692.13\,X_2 - 2479.5\,X_3 - 564.45\,X_1X_2 - 82.95\,X_1X_3 + 251.55\,X_2X_3 \qquad (2)$$

Where: Y_2 is the holding power response, X_1 is GRE, X_2 is PGRE and X_3 is lecithin

Table 4 shows that the quadratic mixture model (Eq.2) for holding power was not statistically significant at the 95% confidence level (P = 0.116), and none of the linear or interaction terms were significant (P > 0.05). Despite the lack of statistical significance, the model exhibited an excellent coefficient of determination (R^2 = 99.53%). The high coefficient of determination suggests that the model adequately described the experimental data and can be used to identify formulation trends within the studied composition range.

Cohesion Analysis

Theoretically, cohesion refers to the strength of interaction between polymer chains or between molecules in an adhesive material. If the cohesion is high enough, then when tensile force is applied, the adhesive will tend to detach itself cleanly from the surface without leaving any residue. Conversely, low cohesion indicates that the internal structure of the adhesive is unable to withstand deformation, especially under conditions such as when the peeling test was performed.

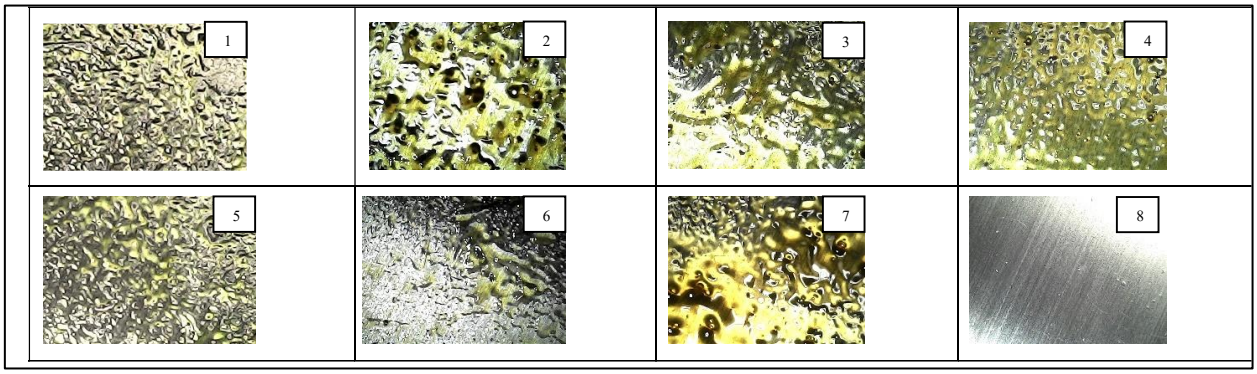

Figure 5. Cohesion analysis based on the surface morphology of PSA samples prepared under mixing conditions 1–7, as described in Table 2. Image 8 corresponds to the stainless-steel substrate without PSA and serves as the reference surface.

Cohesion analysis was conducted through microscopic examination of the stainless-steel substrate after the peel test. The observations revealed that adhesive residues remained on the substrate surface following peeling, indicating cohesive failure within the adhesive layer (Figure 5).

Cohesive failure occurs (5.1-7) when the internal strength of the adhesive is lower than the adhesive-substrate interaction strength. In such cases, failure takes place inside the adhesive material rather than at the interface. The observed residue suggests that both the adhesive and cohesive properties of the developed PSA remain inadequate and require further improvement. Several factors may contribute to the relatively low cohesion, including insufficient crosslink density and suboptimal interactions among the adhesive components [17], [18]. Increasing crosslink density or optimizing the ratio between PVA and rosin ester components may improve cohesive strength in future studies.

4. Conclusion

A mixture design technique was used to generate a sustainable pressure-sensitive adhesive (PSA) employing glycerol rosin ester (GRE), polyethylene glycol rosin ester (PGRE), lecithin, polyvinyl alcohol (PVA) and borax. The obtained PSA films showed a flexible gel-like structure owing to the crosslinking interaction between PVA and borax.

The formulation composition exhibited a considerable influence on the adhesive performance. The peel strength and the holding power increase with the increase in glycerol rosin ester content, suggesting that glycerol rosin ester acts as an important tackifier in improving the adhesive performance. The suggested best formulation from the mixture design was 9% glycerol rosin ester, 7% PEG rosin ester and 4% lecithin with peel strength of 0.80 N/cm and holding power of 800 s.

Observations of the surface morphology indicated that formulations with higher rosin ester concentrations often had more homogenous and sticky surfaces. From the investigation of the cohesion, it was found that the produced PSA still showed cohesive failure as adhesive residues remained on the substrate after peeling. This result shows that the future development must enhance more in internal cohesive strength.

The developed PSA shows the potential of renewable rosin-based materials as partial replacements for petroleum-based tackifiers. Glycerol rosin ester and PEG rosin ester are based on rosin, a renewable resource from forest products, and lecithin comes from biomass sources. The use of these renewable ingredients can lead to a reduction of the dependence on fossil resources and to lower the environmental impacts of petroleum derived materials. Therefore, the prepared PSA is a promising route for developing more sustainable adhesive materials, while maintaining acceptable adhesive performance.

Acknowledgments

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