ISSN: 1410-8917
Jurnal Kimia Sains & Aplikasi
e-ISSN: 2597-9914

Jurnal Kimia Sains dan Aplikasi 26 (7) (2023): 261-267
Jurnal Kimia Sains dan Aplikasi
Journal of Scientific and Applied Chemistry
Journal homepage: http://ejournal.undip.ac.id/index.php/ksa

The Effect of Adding Pine Tar Oil and Petrosin as Tackifier Ingredients on The Quality of Liquid Rubber Compound-based Adhesives

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https://doi.org/10.14710/jksa.26.7.261-267

Article Info

Article history:

Received: 15th June 2023
Revised: 29th September 2023
Accepted: 11th October 2023
Online: 05th November 2023

Keywords:
Tackifier; Liquid Rubber Compound; Adhesive

Abstract

This research is focused on the development of adhesives through the utilization of different types of tackifiers. The primary raw materials employed comprise 60% concentrated latex, which is subsequently blended with additives that have been dispersed, including KOH as a stabilizing agent, stearic acid and ZnO as activators, BHT as an antioxidant, $CaCO_3$ as a filler, MBTS (Mercaptobenzothiazole disulfide) and TMTD (Tetramethyl Thiuram Disulphide) as accelerators, and sulfur as a vulcanizing agent. This research explored two specific variations of tackifiers: pine tar oil and petrosin. The characterization of the adhesives encompasses an analysis of acidity (pH), viscosity, adhesive strength, functional group analysis using FTIR, thermal properties, and XRD. The research findings showed that the optimal conditions were achieved in the third variation of the adhesive, with the same ratio of pine tar oil tackifier and petrosin of 4 grams each. In this particular variation, adhesives conforming to the SNI 12- 7195-2006 standards were produced, displaying an adhesion strength of 1.506 N/mm, a viscosity of 1075 cP, and a pH level of 4.6. Furthermore, thermal testing results reveal that the adhesive created with tackifier (V3) exhibits thermal resistance up to 350°C, a glass transition temperature of 354°C, and a melting point of 378°C.

1. Introduction

Indonesia is the second largest producer of natural rubber globally, following Thailand. In 2021, the total expanse of rubber plantations reached 3.76 million hectares, and the estimated production stood at 3.12 million tons [1]. Rubber can be categorized into two main types: synthetic and natural. The application of natural rubber is somewhat restricted due to its heightened susceptibility to oxidation and its limited ability to withstand low temperatures [2]. Because natural rubber lacks vital properties like tensile strength and elongation at break and is prone to deterioration when exposed to heat and air, it is commonly blended with chemical additives. These additives enhance the final rubber product's mechanical and chemical characteristics [3]. Notably, natural rubber possesses excellent adhesive properties, making it suitable for soft rubber applications. These properties and forms of natural rubber lay the foundation for its use in the latex adhesive industry [4].

When transforming natural rubber latex into adhesive, it is processed into a liquid rubber compound. This liquid rubber compound is a blend of concentrated latex as the primary raw material and specific chemical additives. These chemical additives typically include antioxidants, accelerators, stabilizers, filler activators, and vulcanizers. Incorporating these chemical additives during the compound-making process can influence the stability of the concentrated latex. To ensure that the latex remains in a colloidal state during the compound production process, it becomes imperative to introduce a stabilizer that preserves the alkaline condition of the liquid rubber compound [5].

The adhesive is a substance designed to bond two surfaces of objects, providing sufficient strength to withstand external loads. Natural rubber can produce adhesives for shoe soles when combined with a *cucuron* resin tackifier [6]. The purpose of a tackifier is to enhance the adhesive properties of the adhesive. The performance of the tackifier itself involves augmenting the stickiness or adhesion capacity of rubber, which is influenced by factors such as chain elongation and the degree of polarity between molecules [7]. Tackifiers commonly used adhesive manufacturing include *gondorukem* (resina colophonium), *cucuron*, and phenolic resin. Pine tar oil can serve as a tackifier by influencing the tensile strength of adhesion [8].

Pine tar oil comprises turpentine, resin, guaiacol, creosol, methyl creosol, phenol, fluorol, toluene, xylene, and various other hydrocarbons. The presence of resin in pine tar oil contributes to the favorable adhesive properties of the rubber compound [9]. Additionally, petroleum or petrosin exhibits substantial adhesion, particularly after *gondorukem*. Therefore, petroleum or petrosin can be utilized as a tackifier in forthcoming research endeavors [10]. Leveraging the advantages of pine tar oil and petrosin can result in adhesives with superior tackifiers and improved adhesion compared to prior research efforts. Pine tar oil and petrosin demonstrate superior adhesion in adhesive tests compared to other tackifiers [8].

2. Experimental

2.1. Materials

The materials employed in this study encompass the following components: stearic acid (Rubber activator, Rofa, Bandung), ZnO (Rubber activator, Rofa, Bandung), sulfur (Rubber vulcanisator, Rofa, Bandung), TMTD (Rubber accelerator, Kemai, China), MBTS (Rubber accelerator, Kemai, China), $CaCO_3$ (Rubber filler, Rofa, Bandung), BHT (Rubber antioxidant, Lanxess, Jerman), KOH (Rubber stabilizer, Rofa, Bandung), sodium naphthalenesulfonate (Darvan, Jakarta), pine tar oil (tackifier, Pine tack, Bogor), petrosin (petroleum C9, Tangerang), and 60% concentrated latex.

2.2. Preparation of Liquid Rubber Compounds

Before preparing the liquid rubber compound, the additives were initially dispersed. Each solid additive, such as TMTD, ZnO, MBTS, BHT, $CaCO_3$, and sulfur, was combined with darvan and thoroughly mixed using a stirring rod until homogeneity. Once the additives had been properly dispersed, they were introduced into 166.7 grams of 60% concentrated latex. The sequence for adding the dispersion additives commenced with KOH, followed by stearic acid, ZnO, BHT, $CaCO_3$, MBTS, TMTD, and sulfur. Following this addition, the liquid rubber compound was allowed to mature for 72 hours, facilitating the vulcanization process. The formulation employed in creating the liquid rubber compound can be found in Table 1.

2.3. Fabrication of Adhesives

The adhesive production process involved blending 203 grams of a liquid rubber compound treated with pine tar oil and petrosin, with specific composition variations as outlined in Table 2. This amalgamation of the compound and tackifier was stirred until homogeneity was achieved, resulting in a denser mixture. Subsequently, the adhesive was applied to the cut. The test piece used for the shoe sole measured 80 mm in length and 20 mm in width, as depicted in Figure 1.

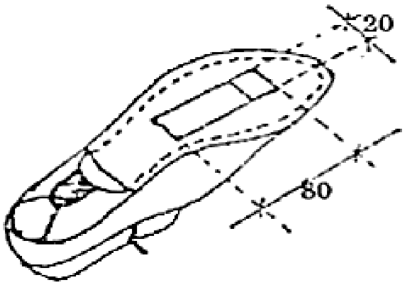

Figure 1. The size of the test species used on the shoe sole

Table 1. Liquid rubber compound formula [11]

No.	Material	Total (%)	Total (phr)	Total (g)
1	Concentrated Latex	60	100	166.7
2	KOH	10	4.1	7
3	Stearic Acid	50	2.3	4
4	ZnO	30	5.3	9
5	BHT	50	1.19	2
6	$CaCO_3$	60	4.9	8.3
7	MBTS	30	1.4	2.5
8	TMTD	30	0.35	0.6
9	Sulfur	50	1.79	3

The characterization of the adhesive encompassed several tests. These tests included measuring the acidity level using a Hanna pH meter, determining viscosity with a Brookfield Viscometer equipped with spindle 5 operating at approximately 20 rpm, and assessing adhesive strength digitally by applying the adhesive to a test sample in the shape of an 80 x 20 mm shoe sole. Functional group analysis was conducted with a Bruker FTIR instrument, crystallinity assessment with the XRD (X'Pert PRO PANalytical) instrument, and thermal analysis with the Hitachi STA2000V thermal analysis instrument, with temperatures reaching up to 900°C.

3. Results and Discussion

3.1. Acidity Assessment (pH)

The adhesive produced in this study exhibited a pH range of 4.5 to 6.5. According to SNI 06-6049-1999, natural rubber adhesives are considered suitable within a pH range of 3 to 8. A lower pH value can prolong the adhesive's shelf life because acidic environments inhibit bacterial growth [12]. Nevertheless, the lower pH value in this study caused the adhesive to become more viscous and led to a shorter storage lifespan.

In adhesive production, alterations in pH can influence the adhesive's storage duration. A higher pH value tends to slow down the adhesive curing process. It is important to mention that adding tackifiers, specifically pine tar oil and petrosin, could potentially lead to decreased pH. A graphical representation illustrating the pH variation in the adhesive produced is presented in Figure 2.

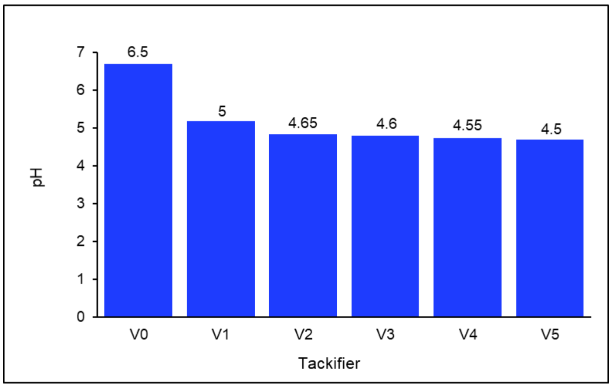

Figure 2. Relationship between adding various tackifiers and the degree of acidity (pH)

In the case of V1 adhesive, the pH level is 5. This pH value can be attributed to the exclusive use of petrosin as the tackifier. In contrast, for V2 adhesive, the pH at 4.65 is notably lower. The reduction in pH is directly linked to the incorporation of pine tar oil as an additional tackifier in this formulation. The compounds present in pine tar oil exert an influence on the adhesive's acidity level. Notably, the quantity of pine tar oil added to V2 is less than that of petrosin.

3.2. Viscosity Test

The viscosity of the adhesive plays a crucial role in determining its tensile strength. A higher viscosity value for the adhesive implies that it may encounter difficulty wetting the surface of the adhered test specimen, which, in turn, results in a lower tensile strength value during testing. Conversely, when the adhesive possesses a lower viscosity, it may lead to reduced adhesion when subjected to tensile strength testing [13]. An ideal adhesive strikes a balance in terms of viscosity, avoiding extremes of thickness or thinness. An adhesive with a suitable viscosity facilitates effective penetration into pores, forming an optimal bond and achieving strong adhesion [14]. The optimal adhesive viscosity avoids being excessively thin, as overly low viscosity (dilution) can diminish adhesive strength [15].

In the viscosity testing, a Brookfield Viscometer equipped with spindle 5 and operating at approximately 20 revolutions per minute (rpm) was employed. Figure 3 illustrates the viscosity measurements, where V3 adhesive displayed a viscosity value of 1075 cP, while V5 exhibited a higher viscosity value of 1200 cP.

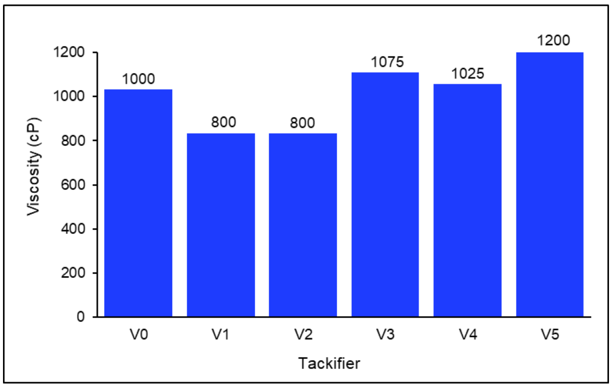

Figure 3. Relationship between adding various tackifiers and adhesive viscosity

Table 2. Variations of pine tar oil and petrosin in the manufacture of adhesives

Adhesive	Tackifier material	
	Pine tar oil	Petrosin
V0	0	0
V1	0	4
V2	1	3
V3	2	2
V4	3	1
V5	4	0

V5, having only pine tar oil as a tackifier, recorded the highest viscosity among all variations. The precise quantities of pine tar oil added are documented in Table 3. This higher viscosity in V5 is attributed to the inherently high viscosity of pine tar oil, as visually evident in Figure 3. During the measurement at 20 rpm, V5 displayed a notable increase in viscosity, which consequently impacted the recorded viscosity value. It is worth noting that adhesive viscosity significantly affects how quickly the adhesive spreads over the glued surface.

Viscosity is often used to gauge the adhesive's ability to penetrate and bond with the adhered material. A lower viscosity implies that the adhesive can permeate the material more rapidly, forming a strong bond between the adhesive and the substrate. Nevertheless, in terms of adhesive strength, V1 and V2 adhesives displayed lower adhesive strengths than V3 adhesives. This suggests that the viscosity value in V3 adhesive strikes an optimal balance for the adhesive formulation, as highlighted in prior work by Saleh *et al.* [15].

3.3. Adhesion Test

The adhesive's bonding strength can be significantly affected by introducing a tackifier. During the bonding process, the adhesive penetrates the material's pores and solidifies, creating a mechanical bond through the cohesive forces between the adhesive molecules and the material's molecules. Adhesion is a vital parameter for evaluating adhesive quality, directly influencing its strength properties.

The adhesion test involved the application of adhesive to a test sample in the shape of an 80 x 20 mm shoe sole. The test was conducted using a specialized adhesion testing instrument with a constant testing speed. Before the addition of the tackifier, the rubber compound exhibited weak adhesion. Consequently, the tackifier was incorporated to align the adhesive's adhesion with the requirements specified in SNI 12-7195-2006. The results of the adhesion test are depicted in the graph presented in Figure 4, showcasing the relationship between various tackifier variations and the adhesion strength.

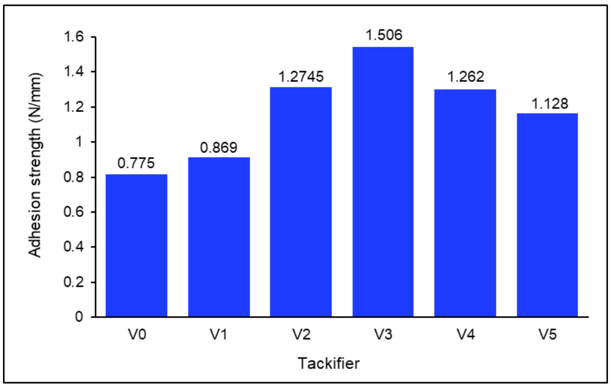

Figure 4. Relationship between adding various adhesive tackifiers and adhesion strength

Figure 4 illustrates the substantial impact of tackifier addition on adhesion strength. The adhesive strength is exceedingly low in adhesives devoid of a tackifier (V0). The introduction of tackifiers commences with V1, incorporating only the petrosin tackifier. Subsequently, V2 adhesive includes petrosin and pine tar oil, with a higher proportion of petrosin than pine tar oil. The addition of tackifiers consistently enhances adhesive strength. V3 adhesive represents the pinnacle, attaining a value of 1.506 N/mm. V3 adhesive maintains an equal combination of pine tar oil and petrosin at a 1:1 ratio.

However, when the proportion of tar oil surpasses that of petrosin, the adhesive strength begins to decline. The augmentation in adhesion strength is readily apparent. In the case of V0 adhesive, adhesion strength is minimal, but it increases significantly upon introducing a tackifier. Both pine tar oil and petrosin demonstrate commendable tackifying properties. Yet, adding a combination of pine tar oil and petrosin in a 2:1 ratio leads to a reduction in adhesive strength, albeit not to a considerable extent. The adhesion value remains relatively strong when compared to a combination of petrosin and pine tar oil at a ratio of 2:1.

The interaction between these two tackifiers and the liquid rubber compound significantly enhances adhesion compared to other formulations. This interaction primarily involves the augmentation of adhesive capability when a tackifier is introduced into the liquid rubber compound. This is attributed to the fact that both tackifiers possess favorable adhesive properties. The effectiveness of a tackifier in enhancing adhesive strength is influenced by the degree of polarity between the rubber molecules and the molecules of the tackifier [7]. Drawing from the study conducted by Puspitasari *et al.* [8], the incorporation of pine tar oil and petrosin in adhesive production results in a noticeable increase in adhesive strength. Moreover, when comparing the combination of tar oil with other resins, the adhesion value is notably higher than when pairing it with paraffin oil or petrosin.

3.4. Functional Group Analysis using FTIR

Pine tar oil and petrosin can effectively be tackifiers in adhesive formulations. Pine tar oil is characterized by its adhesive properties and comprises turpentine, resin, guaiacol, creosol, methyl creosol, phenol, fluorol, toluene, xylene, and various other hydrocarbons. The molecular structures of compounds found in pine tar oil and petrosin are depicted in Figure 5.

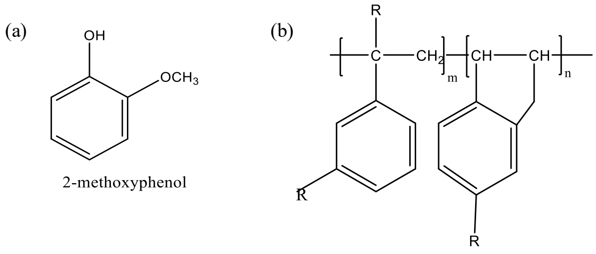

Figure 5. Structures of (a) guaiacol (2-methoxyphenol) from pine tar oil, and (b) petroleum resin

Functional group analysis of the adhesive was conducted using FTIR, and the results are presented in Figure 6. The FTIR analysis revealed a broad absorption band in the range of 3380-3410 cm^{-1}, indicative of the presence of the O-H functional group. Furthermore, the peak observed at 2918 cm^{-1} signifies the presence of alkane functional groups. Additionally, bands within the ranges of 1575-1657 cm^{-1} and 90 cm^{-1} suggest the existence of aromatic C=C functional groups originating from polyisoprene and other functional groups introduced by incorporating various tackifier formulations.

As illustrated in Figure 6, several notable differences are observed regarding absorption intensity and the emergence of new peaks. The functional group represented by the absorption peak around 1441-1457 cm^{-1} corresponds to the C-H bending vibration of CH_3 within the alkane functional group, while the bending vibration within the range of 2912-2918 cm^{-1} indicates the presence of the C-H stretching vibration of CH_2 within the alkane functional group.

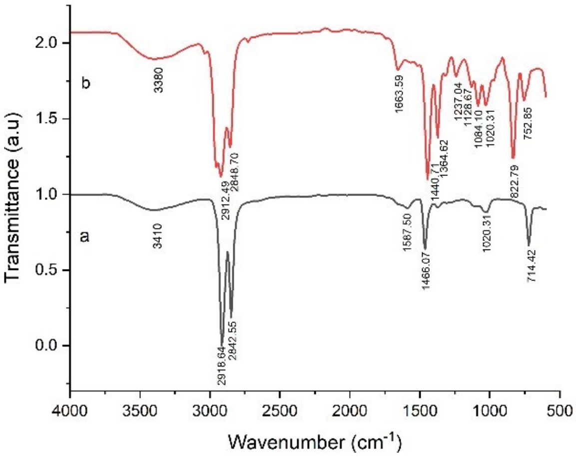

Figure 6. FTIR spectrum of (a) adhesive without tackifier (V0) and (b) adhesive with tackifier (V3)

Table 3. Absorption areas of pine tar oil and petrosin

Tackifier	Absorption area (cm^{-1})	Functional group
Pine tar oil [16]	3063, 1602, 1498, 884, 820	C-H, C-C, and C-H aromatic
	2954, 2924, 2866, 1445, 1378, 1362	C-H aliphatic and C-H (alkyl ester and acid groups)
	1695	C= O carbonyl
	1268, 1038	C-O and C-O-C
Petrosin	2929	Stretching CH_3, CH_2, C-H, dan C-H aldehyde
	1713, 1442, 1353	Stretching C=O
	1211.81	C-O
	980, 858, 743, 702	C=C-H

In the adhesives containing tackifiers (V3), there is an observable peak in the absorption range of 822.79 cm^{-1}, indicating the presence of the alkene C=C bending functional group. This particular peak arises due to incorporating petrosin and pine tar oil as components. However, this peak is notably absent in the case of V0 adhesive. Instead, only the following absorption bands are discernible, each signifying different functional groups: the C=C bending alkene functional group around 715 cm^{-1}, the presence of C-H and C=C functional groups within the range of 1457.58–1560 cm^{-1}, and the C-H stretching vibration of CH_2 alkanes between 2850 and 2912.42 cm^{-1}.

Furthermore, the aromatic C=C functional group originating from polyisoprene is consistently present in the absorption range of 1575-1657.90 cm^{-1} across all variations of adhesive. Details on the absorption bands for pine tar oil and petrosin oil are provided in Table 3. Based on Table 3, functional groups appear in the spectrum of adhesive with tackifier (V3) added with tackifier, which indicates the presence of groups from each tackifier. This is found in the 822.79 cm^{-1} area found in V3 adhesive with the addition of pine tar oil and petrosin tackifier. Some absorption areas on the adhesive and tackifier experienced slight shifts.

3.5. XRD Analysis

The XRD test results reveal a notable alteration in the level of crystallinity between adhesive formulations with and without a tackifier, as depicted in Figure 6. In the absence of a tackifier, there are no discernible peaks indicative of crystallinity. Conversely, when a tackifier is introduced, the XRD analysis exhibits multiple sharp peaks, signifying the presence of crystalline properties. These sharp peaks emerge at 2θ angles of 18.63°, 31.78°, 34.58°, 36.18°, 47.58°, 56.59°, and 62.80°. The diffractogram of V3 adhesive presents evident disparities in comparison to V0 adhesive. This disparity is attributed to the inclusion of tackifiers, specifically pine tar oil, and petrosin, leading to the visualization of a greater number of peaks than in V0. These peaks indicate a more well-defined crystal lattice structure, highlighting that adhesive V3 exhibits a heightened degree of crystallinity compared to V0.

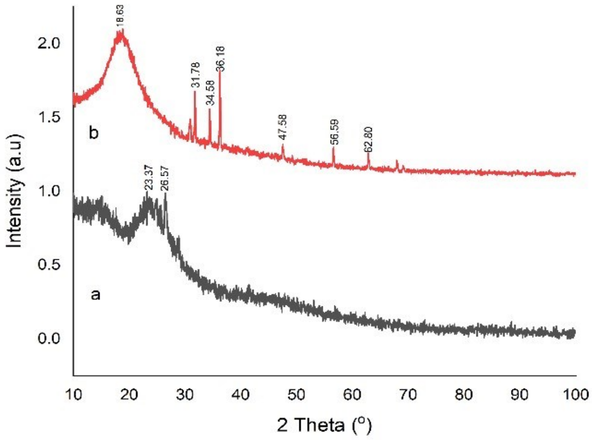

Figure 6. XRD curves of (a) adhesive without tackifier (V0), and (b) adhesive with tackifier (V3)

3.6. Thermal Analysis using TGA-DTA-DSC

In the thermal analysis conducted using TGA (Thermogravimetric Analysis), the adhesive exhibits a characteristic degradation process, manifesting as changes in mass within a specific temperature range, as visually represented in Figure 7. The TGA curve in Figure 7 illustrates that incorporating a tackifier has no discernible impact on the heat resistance of the adhesive in question. This is evident through the consistent mass changes, signifying the degradation process in tackifier-enhanced and non-enhanced adhesive formulations, transpiring within nearly the same temperature range, specifically between 350°C and 470°C. The adhesive experiences a decomposition process at approximately 470°C, accompanied by a decrease in mass between 400°C and 500°C.

Variations in mass alteration are observed among different samples within each formulation, primarily between 100°C and 300°C. These fluctuations are attributable to the evaporation of solvents and additives employed in the adhesive. The thermal degradation of polyisoprene commences at a temperature of 400°C, involving the breakage of cross-links and molecular chains, and becomes particularly pronounced at 470°C [16]. Beyond 470°C, no further degradation is observed, as temperatures exceeding this threshold lead to the formation of residual ash, gaseous byproducts, or inorganic substances arising from impurities [17].

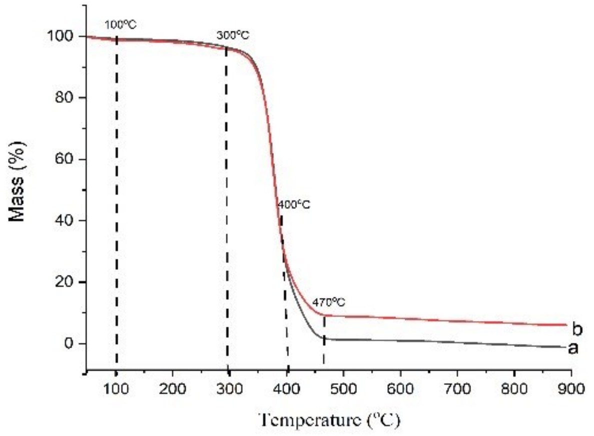

Figure 7. TGA curve of (a) adhesive without tackifier (V0) and (b) adhesive with tackifier (V3)

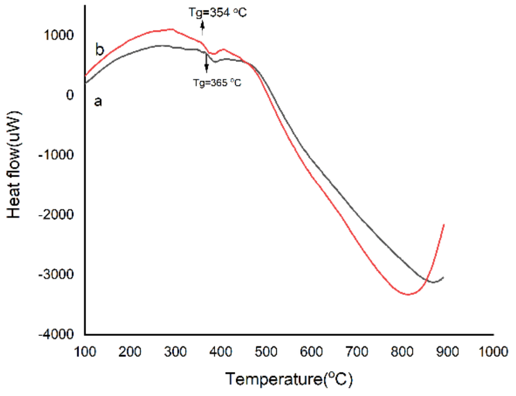

Figure 8. DSC curve (a) adhesive without tackifier (V0) and (b) adhesive with tackifier (V3)

In addition to the thermal analysis conducted with TGA, Differential Scanning Calorimetry (DSC) was employed to investigate the glass transition (Tg) of the adhesive. The DSC thermogram provides insights into various transitions during heating, including endothermic reactions observed within the temperature range of 300-400°C, which can be attributed to the evaporation of solvents and additives [18]. As Kind and Hull [19] suggests, the heating of polyisoprene polymer leads to the disappearance of mass and exothermic reactions, primarily due to the decomposition of polyisoprene into fragments due to evaporation.

The DSC curve presented in Figure 8 reveals that the incorporation of a tackifier results in a reduction of the glass transition temperature. In the case of the best-condition adhesive, variation 3, the glass transition temperature decreases from 365°C in the adhesive lacking a tackifier to 354°C in the adhesive containing a tackifier. This decline indicates that the interaction between polyisoprene and the tackifier compound leads to a reduction in cross-linking. Figure 8 illustrates that various adhesives exhibit glass transitions within the temperature range of 290-400°C. While there is a subtle variation in the temperature range for the glass transition in each adhesive formulation, adhesives without a tackifier (V0) exhibit a glass transition (Tg) at approximately 365°C, while adhesives with a tackifier (V3) display a glass transition at 354°C.

Notably, Figure 8 highlights that the glass transition in V3 adhesive has a longer duration than other adhesive formulations. This distinction can be attributed to the quantity of tackifiers added during adhesive production. In the case of V3 adhesive, an equal amount of tackifier is introduced for pine tar oil and petrosin. Consequently, V3 adhesive records a lower Tg temperature than V0 adhesive, contributing to enhanced polymer flexibility. More flexible polymer chains have the capacity to move more freely within the porous structure of a medium. Additionally, this characteristic could be attributed to the presence of pine tar oil in the V3 adhesive, which contributes to reducing the glass transition temperature [20]. Pine tar oil also functions as a plasticizer or softening agent, contributing to this effect [8].

4. Conclusion

This study establishes that pine tar oil and petrosin are effective tackifiers in adhesive production. The optimal condition, denoted as V3 adhesive, demonstrates adhesion values that conform to the standards outlined in SNI 12-7195-2006. Specifically, it exhibits an adhesion strength of 1.506 N/mm, a viscosity of 1075 cP, and a pH level of 4.6. Furthermore, the thermal testing results indicate that V3 adhesive, which incorporates tackifiers, displays remarkable thermal resistance, withstanding temperatures of up to 350°C. Additionally, it exhibits a glass transition temperature of 354°C and a melting point of 378°C.

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