THE EFFECT OF COPAL RESIN AND TURPENTINE OIL AS TACKIFIER ON THE ADHESIVES QUALITY BASED OF LIQUID RUBBER COMPOUND

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Abstract

This research aims to develop adhesives from liquid rubber compounds with a variety of tackifiers, a combination of copal resin, and turpentine oil. It also aims to characterize the adhesives to determine the quality of the adhesives produced. The research comprised several stages, including the preparation of rubber additive dispersions and solutions, formulation of liquid rubber compounds and adhesives, fabrication of test pieces, and subsequent characterization. The liquid rubber compound is made by mixing concentrated latex with rubber additives, which include KOH as a stabilizer, stearic acid and ZnO as an activator, BHT as an antioxidant, $CaCO_3$ as a filler, MBTS and TMTD as an accelerator, and sulfur as a vulcanizer. The adhesives are made by mixing liquid rubber compound with tackifier material with various combinations of copal resin/turpentine oil, namely F1 (0 gram/4 gram); F2 (1 gram/3 grams); F3 (2 grams/2 grams), F4 (3 grams/1 gram), and F5 (4 grams/0 grams). Adhesives characterization was carried out by testing pH, viscosity, adhesives strength, FTIR, XRD, and thermal analysis. The research results showed that the best conditions were obtained on F4 adhesives with an adhesives strength value of 1.7452 N/mm, viscosity of 1100 cP, and pH 4.6. In this condition, the resulting adhesives meet the requirements for adhesives in accordance with SNI 06-6049-1999 and SNI 12-7195-2006.

***Keywords**: adhesives, liquid rubber compound, tackifier material*

Introduction

Rubber plants are one of the plantation commodities that have an important role and are very reliable in supporting the economy of Indonesia. Indonesia, as a rubber producer, is able to increase rubber production every year, reaching 3.1 million tons in 2021 (Central Statistics Agency, 2022). Rubber plants produce sap, known as latex. Rubber plant latex has good bounce, elasticity, stickiness and grip. Therefore, its use as a raw material for preparing finished goods is quite widespread. Natural rubber latex is widely used as raw material for preparing various types of vehicle tires, rubber shoes, rubber pipes, cables, insulators, mattresses, carpets, gloves, adhesives and vibration barriers (Siregar & Suhendry, 2013)

In the process of preparing finished rubber goods, natural rubber latex must first be made into a compound, namely a mixture of natural rubber latex with chemical additives which include vulcanizing agents, accelerators, activating agents, fillers and protective agents (Anti & Ginting, 2020). As a raw material for preparing finished rubber goods, natural rubber compounds can be used as adhesives because of their flexibility. Natural rubber latex has a long polymer chain structure, namely polyisoprene. Apart from that, latex also contains protein compounds in which there are hydrophobic and hydrophilic groups, which will bind the same groups to other materials. This is what causes natural rubber latex to have the ability to act as an adhesive. Research conducted by Nurhayati (2018) shows that natural rubber compounds can be used as plywood adhesives.

In the adhesives manufacturing process, the material that plays an important role in formulating the adhesives is the tackifier, which is a material that increases adhesives strength which will later influence the bonding process. Tackifier materials commonly used in preparing adhesives are gondorukem resin, coumaron resin, copal resin, phenolic resin, petrosin resin, and pine tar oil. In research conducted by Herminiwati et al. (2008) regarding the manufacture of natural rubber compound as shoe sole adhesives, the tackifier material used was coumaron resin with variations of 5, 10, and 15 phr. The best formulation was obtained using 10 phr coumaron resin which produced adhesives with adhesives strength value of 1553 gr/cm and a viscosity of 2250 cP. Another research was conducted by Susilawati & Rahmaniar (2018) regarding the manufacture of rubber tile glue using gondorukem (siongka) resin as tackifier. The best results obtained in this study showed adhesives strength value of 4.17 kg/in and a viscosity value of 51.33 cP. Previous research regarding the manufacture of natural rubber compound-based adhesives was also carried out by Widayanti (2022). In this research, a natural rubber compound was used as adhesives for shoe soles with tackifier, namely gondorukem resin. The results obtained show adhesives strength value of 2.6482 N/mm and a viscosity value of 375 cP. In other research, which was carried out by Shobrina (2022) regarding the manufacture of shoe sole adhesives using coumaron resin as tackifier, it showed that the best conditions were the addition of 2.87% coumaron resin. The adhesives strength value obtained in this condition is 0.032 N/mm and the viscosity value is 200 cP.

Based on reported research, the use of gondorukem resin and coumaron resin as tackifier materials in the manufacture of natural rubber compound adhesives turns out to be quite a lot, but the process of applying them to the test material requires quite a long drying time as in research conducted by Widayanti (2022) and Shobrina (2022), which requires a drying time of 48 hours. Therefore, it is necessary to look for other tackifier materials that can be used in the process of preparing adhesives based on natural rubber compounds.

Copal is a sap (resin) whose main component is resinous acid, namely agatholic acid. Other compounds are neutral diterpene derivatives and monoterpene alcohols (Lukmandaru, 2017). Turpentine oil is a colorless liquid and is composed of the compounds α-pinene, d-champhene, β-pinene, myrcene, α-phellandrene, carene, p-cymene, and d-limonene (Wiyono et al., 2006). Based on their composition, copal resin and turpentine oil contain hydrophobic (hydrocarbon parts) and hydrophilic (carboxylic acid groups) compounds, so both have potential as natural tackifiers. Various studies regarding the manufacture of natural rubber compound-based adhesives, there has been no research combining copal resin and turpentine oil as tackifiers. Besides that, Indonesia is one of the largest producers of natural rubber in the world besides copal and turpentine as natural tackifier materials are easy to obtain so that the sustainability of the adhesive industrial process will not result in a shortage of raw materials.

Research Methods

Materials

The materials used are concentrated latex with a dry rubber content of 60%, ZnO, stearic acid, MBTS (2-2'-Dithiobisbenzothiazole), TMTD (Tetramethylthiuram disulfide), BHT (butyl hydroxy toluene), $CaCO_3$, KOH, distilled water, darvan (sodium naphthalenesulfonate), copal resin, and turpentine oil. All materials used in this research are of technical quality and obtained from Bogor, West Java.

Instrumentation

The tools used in this research include analytical balances, stirring rods, dropper pipettes, glassware, mortar and pestle, pH meter, tensile strength tester KT 7010A, Brookfield type RVT viscometer, Bruker alpha II Fourier Transform Infrared (FTIR) spectrophotometer, Thermo Graphimetry (TG) analyzer instrument, and X-Ray Diffraction (XRD) instrument.

Procedure

1) Preparation of dispersions and solutions

The composition of the dispersion and solution of materials for preparing the compound is presented in Table 1.

Table 1. Dispersion composition of materials for preparing compound (Putri, 2017)

Material Name	Amount (grams)	Darvan (grams)	Aquadest (mL)
Sulfur 50%	50	1	49
Stearic acid 50%	50	0	50
ZnO 30%	30	1	69
KOH 10%	10	0	90
BHT 50%	50	4	46
$CaCO_3$ 60%	60	2	38
MBTS 30%	30	1	69
TMTD 30%	30	1.5	68.5

2) Preparation of liquid rubber compound

The process of preparing liquid rubber compounds is carried out by mixing concentrated latex with chemical additives that have been made into a dispersion. The ingredients are added gradually and sequentially while stirring until homogeneous. The sequence of ingredients added in preparing the compound is KOH, stearic acid, ZnO, BHT, $CaCO_3$, MBTS, TMTD, and sulfur. The composition of the ingredients for preparing liquid rubber compounds can be seen in Table 2.

Table 2. Composition of ingredients for preparing liquid rubber compound (Sutanto, 2015 and Putri, 2017)

No	Material Name	Content (%)	Amount (grams)
1	Latex	60	166.7
2	KOH	10	7
3	Stearic Acid	50	4
4	ZnO	30	9
5	BH T	50	2
6	$CaCO_3$	60	8.3
7	MBTS	30	2.5
8	TMTD	30	0.6
9	Sulfur	50	3

3) Preparation of adhesives

The adhesives-preparing process is carried out by mixing 203.1 grams of liquid rubber compound with tackifier material. The addition of the tackifier material is done gradually (little by little) while stirring. Variations in experimental parameters to be carried out in this research can be seen in Table 3.

Table 3. Variation of experimental parameters

Formulation	Tackifier Material	
	Copal Resin (gram)	Turpentine Oil (gram)
F0	0	0
F1	0	4
F2	1	3
F3	2	2
F4	3	1
F5	4	0

4) Testing and characterization

The tests carried out in this research were pH testing, viscosity testing using a Brookfield type RVT viscometer (spindle 5 with a speed of 20 rpm), adhesives strength testing, functional group analysis with an FTIR instrument, thermal analysis, and crystal phase analysis with an XRD instrument. Adhesion strength testing is carried out based on SNI 0566-2009; namely, the test piece is opened to a length of 20 mm, as shown in Figure 1. Then, it is installed in the clamp of the tensile test tool. The pulling tool is run at a constant speed until the layer of the test piece is completely exposed.

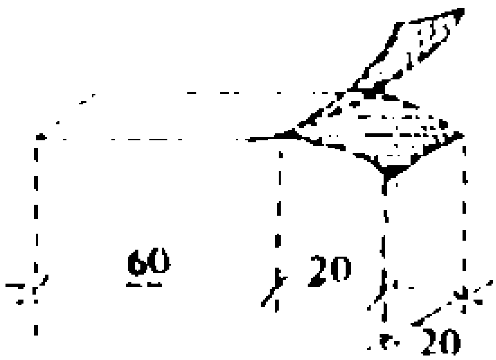

Figure 1. Parts withdrawn (National Standardization Agency, 2009)

Results and Discussion

Liquid rubber compound

A liquid rubber compound is a mixture of natural rubber latex and chemical additives that form the compound (Herminiwati et al., 2008). The aim of preparing a compound is to change the properties of rubber from thermoplastic to become heat-stable, elastic and strong. The result of preparing the compound is white-brown in color, has a strong ammonia odor, and there is foam on the surface of the compound, as shown in Figure 2.

Figure 2. Liquid rubber compound

Preparing the compound requires a curing process (maturation) for 72 hours (Sutanto, 2015). The maturation process is carried out with the aim of maximizing the vulcanization process so that a level of compound maturity is obtained which will affect the quality of the finished rubber product (Mayasari & Wirapraja, 2019). Liquid rubber compound that has been matured has a slightly thicker texture, does not clump, the smell of ammonia is not strong, and there is no foam on the surface of the compound.

Adhesives and application on test pieces

The adhesives have been produced by mixing the tackifier material with a liquid rubber compound that has been cured for 72 hours. The tackifier material used in this research was a combination of copal resin and turpentine oil. This combination of tackifier materials is intended to increase the adhesives strength of the resulting adhesives. The result of preparing the adhesives is liquid (semi-viscous), has a slight odor, and is milky white in color, as shown in Figure 3.

Figure 3. Liquid rubber compound adhesives

The adhesives produced in this research consist of 5 formulations, namely F1 (combination of 0 grams copal resin and 4 grams turpentine oil), F2 (combination of 1 gram copal resin and 3 grams turpentine oil), F3 (combination of 2 grams copal resin and turpentine oil 2 grams), F4 (combination of 3 grams of copal resin and 1 gram of turpentine oil), and F5 (combination of 4 grams of copal resin and 0 grams of turpentine oil).

The effect of copal resin tackifier and turpentine oil combination on adhesive pH

Based on the test results data shown in Figure 4, the effect of the combination of copal resin tackifier and turpentine oil on the pH of the adhesives can be seen. In Figure 4, it can be seen that the pH value of the adhesives from F1 to F5 has decreased, but in F2, F3, and F4, the pH value of the adhesives is stable at 4.6. The pH level decreases when more copal resin is used as a tackifier due to the presence of agatholic acid in copal resin. The degree of acidity (pH) is a test parameter that influences the shelf life of the adhesives. The higher the pH value of the adhesives, the shorter the shelf life, and vice versa. The lower the pH value of the adhesives, the longer its shelf life will be. This is because bacteria cannot live in an acidic atmosphere (Fadlurahman, 2018).

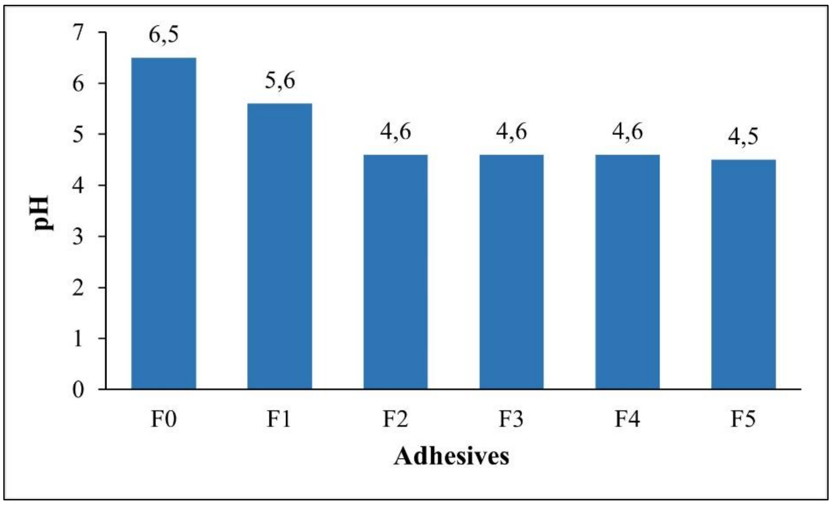

Figure 4. Influence curve of copal resin tackifier and turpentine oil combination on adhesives pH

The pH test results shown in Figure 4 show that the highest pH value is at point 5.6, namely adhesives F1, while the lowest pH value is shown in adhesives F5, which is at point 4.6. Judging from the manufacturing formulation, F5 adhesives are made from the highest variation of copal resin, namely 4 grams, where the composition of this copal resin is dominated by acid compounds, namely agatholic acid. This is what causes F5 adhesives to have a more acidic pH than other adhesives. Based on SNI 06-6049-1999, the pH value requirement for adhesives ranges from 3–8. This shows that the adhesive pH values (F1, F2, F3, F4, and F5) obtained in this study have met the adhesive quality requirements.

The effect of copal resin tackifier and turpentine oil combination on adhesive viscosity

Based on the test results data, the effect of the combination of copal resin tackifier and turpentine oil on the viscosity of the adhesives can be seen in Figure 5. It shows the relationship between adhesive viscosity and the tackifier material used. The viscosity (thickness) value of the adhesives increases gradually from F1 to F4, then increases rapidly at F5, namely 1400 cP. The increase in viscosity occurs due to the concentration of the tackifier material used. This situation is caused by the copal resin and turpentine used as tackifiers consisting of compounds with large molecular weights so that the more of this mixture of compounds used, especially copal resin, the greater the viscosity of the adhesives produced. F1 adhesives are formed from a combination of 0 grams copal resin tackifier and 4 grams turpentine oil, so it has a lower viscosity value compared to other adhesives, while F5 adhesives have the highest viscosity value because the tackifier material used is only copal resin with the highest variation, namely 4 grams.

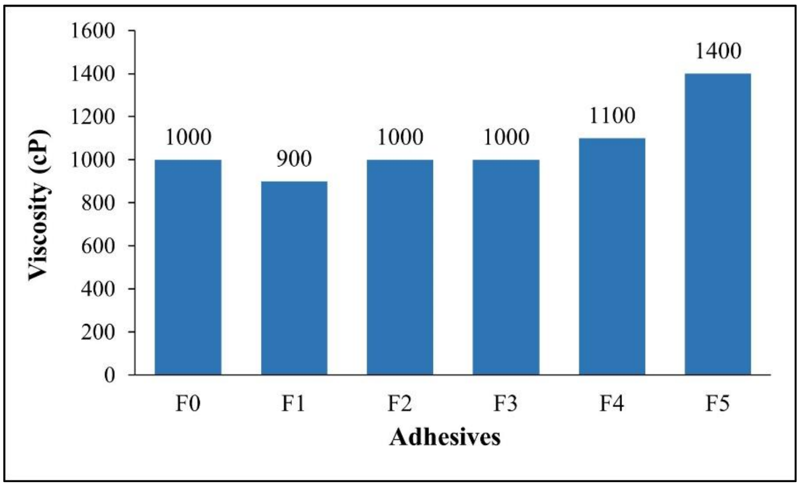

Figure 5. Influence curve of copal resin tackifier and turpentine oil combination on adhesives viscosity

The viscosity value of the adhesives affects its flow properties. The higher the viscosity value, the more difficult it is to wet and penetrate the pores of the surface of the material being glued, but the lower the viscosity value of the adhesives (the thinner it is), the more penetration of the adhesives into the pores of the material being glued will be excessive, causing adhesives lines between the surfaces of the materials being bonded will decrease. Therefore, Eskani et al. (2017) stated that a good adhesive is one that is not too thick and not too runny. According to SNI 06-6049-1999, the adhesive viscosity value requirement is a minimum of 1 poise or 1000 cP so that all adhesive formulations that have been made (F1, F2, F3, F4, and F5) comply with the adhesive quality requirements.

The effect of copal resin tackifier and turpentine oil combination on adhesive adhesion strength

Based on the test results data, it can be seen the effect of the copal resin tackifier and turpentine oil combination on the adhesive bond strength in Figure 6. In Figure 6, it can be observed that there is an increase in the adhesives strength value from adhesives F1 to adhesives F4, however, for adhesives F5, the adhesives strength value obtained has decreased. The increase in adhesive strength value is caused by viscosity. This is in accordance with the results of research conducted by Eskani et al. (2017) stated that the adhesives strength of adhesives is closely related to their viscosity, which indicates that adhesives that have low and high viscosity values have low adhesives strength values , which are caused by their flow ability to wet the surface of a material. This is also related to the adhesives's ability to wet the substrate surface and form intermolecular bonds with the substrate. Optimum conditions are obtained for F4 adhesives, this shows that the bond that occurs between the adhesives molecules and the substrate surface reaches a maximum. Judging from the manufacturing formulation, F1 adhesives, which is a combination of 0 grams of copal resin and 4 grams of turpentine oil, have the lowest viscosity value, while F5 adhesives have the highest viscosity because it is formed from a combination of 4 grams of copal resin and 0 grams of turpentine oil. Through this approach, it can be seen that the adhesives with the highest adhesive strength value have a viscosity value that is neither too low nor too high.

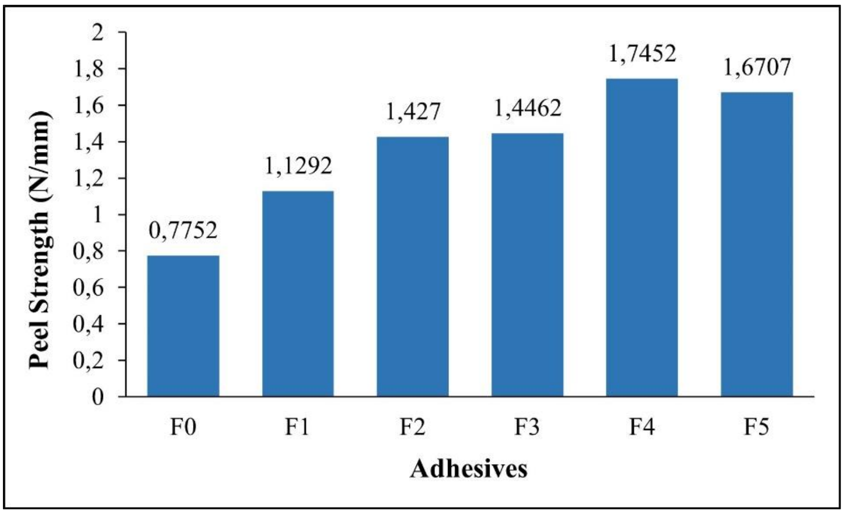

Figure 6. Influence curve of the combination of copal resin tackifier and turpentine oil on adhesives peel strength

The adhesives strength test results show that the best adhesives formulation is at point F4 which shows the highest adhesives strength value of 1.7452 N/mm. Based on the adhesives strength data from the adhesives that has been made, the adhesives strength value meets the adhesives strength quality requirements according to SNI 12-7195-2006, namely a minimum of 1.5 N/mm.

Functional group analysis results

The results of the FTIR testing of the liquid rubber compound and adhesives which was carried out in the wave number area 500–4000 cm^{-1} are visible in the spectrum in Figure 7. Based on the spectrum, as seen in Figure 7, shows that the peaks that appear have almost similar functional groups with different intensities. The complete functional groups contained in the compounds and adhesives made are shown in Table 4.

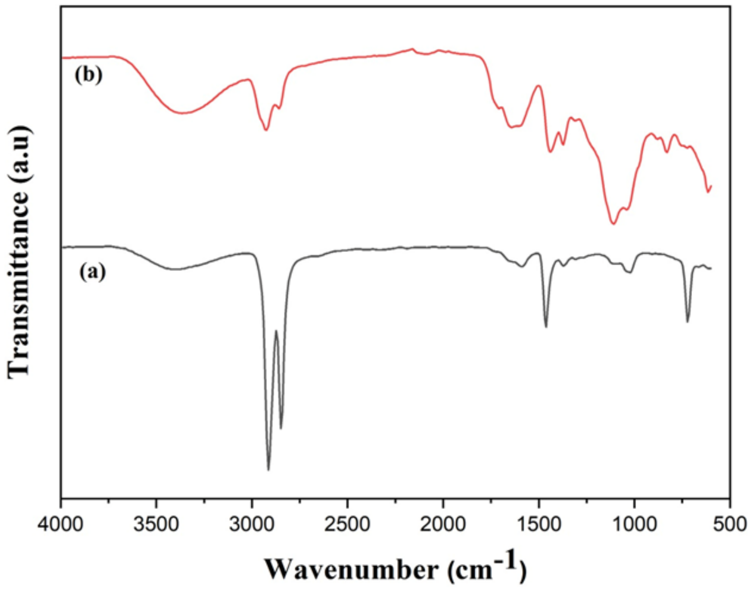

Figure 7. FTIR spectrum (a) liquid rubber compound; (b) adhesives

Table 4. Absorption peaks of functional groups of natural rubber compounds and adhesives

Absorption area (Dachriyanus, 2004)	**Functional Group**	**Absorption Peak (cm^{-1})**	
		Compound	**Adhesives**
3750–3000	-OH	3398.95	3366.11
3000–2700	-CH3 strain, -CH2, -CH	2914.56 2848.88	2922.77
1675–1500	Strain -C=C-	1584.53	1642.01
1475–1300	-CH bending	1461.38	1444.96
1300–1000	-C-O carboxylic acid	1018.04	1108.35
995–650	C-H alkene	722.48	837.42

The results of functional group identification in Table 4 show that the peak infrared absorption areas of F0 adhesives (without the addition of tackifier) and F4 adhesives (with the addition of tackifier) are similar. The absorption peak indicating the presence of a -C=C- bond can be seen as a shift from the F0 adhesives. This is influenced by the addition of tackifier material to F4 adhesives, the constituent components of which have quite a lot of -C=C- bonds. F0 adhesives have a sharper peak than F4 adhesives. Specific functional groups that many tackifier molecules have, namely -C=C- double bonds as well as a few -OH groups, cause changes and shifts in the spectrum at wave numbers 3750–3000 cm^{-1} and 1675–1500 cm^{-1}. This change in the position and appearance of the infrared absorption band is caused by hydrogen bonding. If the hydrogen bonds are less extensive, they will show a sharper peak (Fessenden and Fessenden, 1999). Based on the results of the IR spectrum analysis, it can be seen that the -C=C group of isoprene is still at the peak absorption area of 1675–1500 cm^{-1} (Dachriyanus, 2004).

XRD analysis results

Adhesives F0 (without the addition of tackifier) and adhesives F4 (with the addition of tackifier) were further analyzed to determine the crystalline, semi-crystalline or amorphous structure. The results of the XRD analysis are displayed in the diffractogram in Figure 8.

The results of XRD analysis of adhesives F0 and F4 in Figure 8 show the presence of amorphous and crystalline phases, indicating that both are semi-crystalline (Sudirman et al., 2004). The XRD diffractogram of F0 adhesives shows a diffraction pattern at peaks with angles of 2θ, namely 18.42°, 21.44°, 23.94° and 36.46°. In contrast to F4 adhesives, which shows many peaks with angles of 2θ, namely 18.66°, 30.98°; 31.82°; 34.47°; 36.28°; 41.17°; 47.60°; 56.66°; 62.89°; and 67.99°.

Based on XRD test data, the degree of crystallinity that affects the properties of a polymer can be determined. The greater the degree of crystallinity of a polymer, the greater the adhesive strength value and the better its thermal resistance. The results of determining the degree of crystallinity (Xc) of adhesives F0 and F4 show that adhesives F0 and F4 have a degree of crystallinity of 20.72% and 46.86%, which indicates that adhesives F4 have better adhesive strength and thermal resistance. This is because the components that make up copal resin, namely agatholic acid, and those that make up turpentine, namely abietic acid and pimaric acid, are crystalline compounds obtained from plant sap, so the addition of tackifier will increase the level of crystallinity of the resulting adhesives.

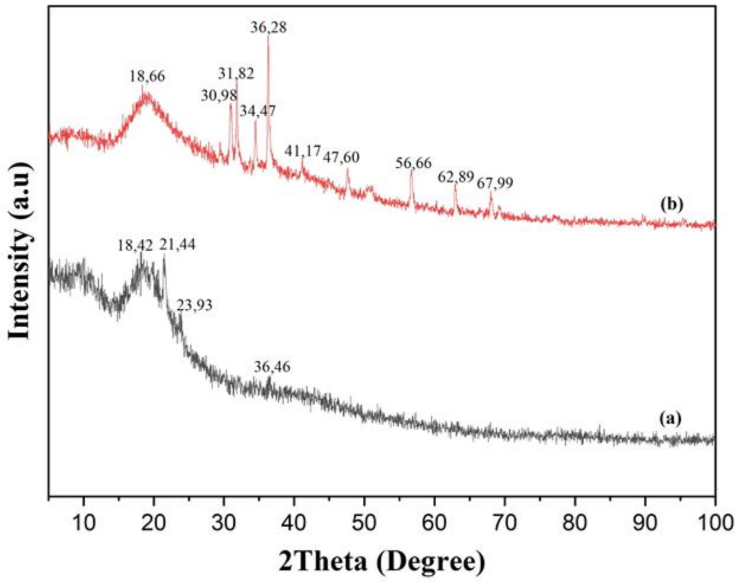

Figure 8. XRD diffractogram of adhesives (a) F0; and (b) F4

The XRD diffractogram of adhesives F0 and F4 also shows the crystalline size. The XRD peak that has a wide pattern indicates small-sized crystal material, while the diffraction pattern with a sharp pattern indicates large-sized crystal material (Abdullah and Khairurrijal, 2009). Based on the results of the XRD analysis, it can be seen that the use of tackifier material in the adhesives changes the shape of the diffraction pattern and peaks at an angle of 2θ.

Thermal analysis results (TGA-DSC)

Thermal analysis is studied through TGA and DSC testing. TGA testing is carried out to determine the degradation temperature of the adhesive sample so that when applied, the resistance of the adhesives to high temperatures can be determined. The TGA test results are displayed via the TG curve in Figure 9.

The TG curve in Figure 9 shows a single-stage decomposition process. This decomposition process is characterized by a decrease in sample mass. The initial stage of degradation is shown at a temperature of 100 °C. At this stage, both samples lost mass by 0.92%, which occurred due to the release of the -OH group (Sudirman et al., 2000). At a temperature of 314 °C, all samples decomposed with a weight loss of 2.43%. The process that occurs at a temperature of 100–314 °C is the evaporation of volatile additives such as TMTD and BHT. At a temperature of 314–470 °C, all samples decomposed with the largest percentage mass loss (F0 adhesives 95.12% and F4 adhesives 88.05%). The process that occurs at this temperature is breaking the sulfur cross-links and breaking the main chain of rubber (polyisoprene) (Kind and Hull, 2012).

At temperatures above 470 °C, no other decomposition processes were seen in all samples (adhesives), indicating only the presence of decomposed gas, as described in the research of Ribeiro et al. (2017). At this stage, there is no further decrease in mass (sample mass is constant). The TG curve indicates that F4 adhesives have the highest thermal stability because the mass loss is the lowest percentage compared to F0 adhesives. This is due to the presence of tackifier material, which contains a crystalline compound and is more heat resistant, which is added to the F4 adhesives so that at a temperature of 470 °C there are still more components remaining compared to the adhesives without using tackifier, namely F0.

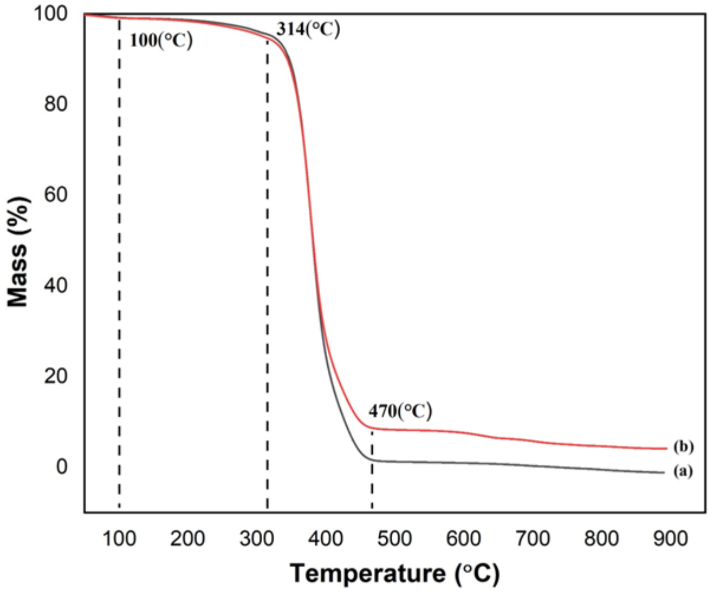

Figure 9. TG curve of adhesives (a) F0; and (b) F4

Thermal analysis studied through Differential Scanning Calorimetry (DSC) provides information regarding the glass transition (Tg), which will determine the thermal resistance of a material. The DSC test results are displayed in the curve in Figure 10. Figure 10 shows the DSC curve of F0 adhesives (without the addition of tackifier material) and F4 adhesives (with the addition of tackifier material). The DSC curve shows the presence of 1 (one) endothermic peak in all samples, which indicates the occurrence of an endothermic reaction in the sample. The endothermic peak appeared at a temperature of ±385 °C for all samples, indicating that adhesives F0 and adhesives F4 melted at that temperature.

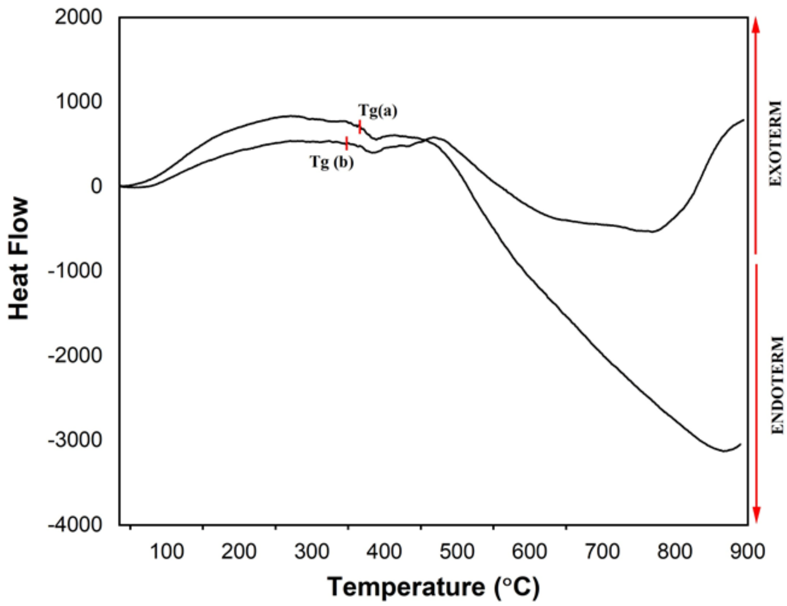

Figure 10. DSC curve of adhesives (a) F0; and (b) F4

The DSC curve in Figure 10 also shows the glass transition temperature (Tg) and melting temperature (Tm). The glass transition temperature (Tg) is the temperature when a change occurs from hard (rigid) to soft (rubber). The DSC curve shows that the glass transition temperature (Tg) of F0 adhesives occurs at 365 ℃ and the melting point (Tm) at 388 ℃, while the Tg of F4 adhesives occurs at 340 ℃ and Tm at 382 ℃. This shows that the glass transition temperature (Tg) of F0 adhesives is higher than that of F4 adhesives, indicating that F0 adhesives have lower thermal resistance because their structure is the most brittle. Based on these indications, it can be seen that the addition of tackifier material in the manufacture of adhesives affects the glass transition temperature (Tg). The high glass transition temperature causes the adhesives to require a longer reaction to change their state from glassy to rubbery, so the higher the Tg, the stiffer the adhesives (brittle), causing the lower the adhesiveness (Cifriadi, 2008).

Conclusion

Adhesives that have been made using a combination of copal resin tackifier and turpentine oil, namely F2, F3, F4 and F5 adhesives, meet the adhesives quality requirements based on SNI 12-7195-2006, namely a minimum of 1.5 N/mm. The more copal resin tackifiers used, the lower the pH value, the higher the peel strength and viscosity, and the better the thermal resistance. The best adhesives obtained were F4 adhesives, which showed a pH of 4.6, viscosity value of 1100 cP, and peel strength of 1.7452 N/mm. The made adhesives have advantages over existing adhesives because they use natural materials that are more easily degraded. The latex-based adhesive industry with copal resin and turpentine tackifiers is highly promising due to the abundant availability of raw materials in Indonesia.

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Conflict of Interest

The authors declare that there is no conflict of interest.

Author Contributions

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. All authors agreed to the final version of this manuscript.

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