Gutta-Percha-based Adhesive for Laminated Wood Production

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The characteristics of gutta-percha (*i.e.*, chemical compound and melting and glass transition temperatures) and the performance of laminated wood (*i.e.*, moisture content, density, shear strength, and delamination ratio) prepared from sengon wood *(Paraserianthes falcataria* L. Nielsen*)* bonded with a gutta-percha-based adhesive were investigated. The gutta-percha-based adhesive was prepared by modification of gutta-percha with 5% maleic anhydride (MAH) and 0.75% benzoyl peroxide (BPO) at various gutta-percha to toluene ratios (w/w) (*i.e.*, 15:85; 17.5:82.5; 20:80; and 22.5:77.5), followed by heating at 70 °C in a water bath for 10 min. Laminated wood was manufactured using both modified and unmodified gutta-percha-based adhesives at 250 gm^{-2} of glue spread and clamped for 24 h. Terpenes, especially 1,3 butadiene, 2-methyl (CAS)-isoprene (*trans* 1,4- isoprene) (polyterpene), were found to be the dominant chemical component of gutta-percha. The glass transition and melting temperatures of gutta-percha were -56.75 °C and 51.67 °C, respectively. The modification of gutta-percha with MAH and BPO as an initiator resulted in improved performance for the laminated wood. Infra-red spectrometry of the modified gutta-percha-based adhesive showed a new peak at 1720 cm^{-1}, indicating the C=O bond of MAH.

Keywords: Gutta-percha; Laminated wood; Maleic anhydride; Trans-1,4-isoprene

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INTRODUCTION

The use of synthetic adhesives for wood composites is unfavorable because of their non-renewable and non-biodegradable nature; they are thus considered unsustainable and less environmentally benign. Therefore, endeavors to search for renewable and environmentally friendly raw materials for adhesives for wood composites are needed. Various potential sources of adhesive materials, such as lignin, tannins, and elastomers, are abundantly available in tropical countries such as Indonesia.

Palaquium, a gutta-percha (trans-1,4-isoprene rubber)-producing tree, is naturally available and extensively developed in Indonesia. Various species of palaquium can be readily found in the forested areas of the Riau Archipelago, South Sumatra, South Sulawesi, Central Kalimantan, South Kalimantan, and West Papua. *Palaquium burckii* trees have been planted in a community forest at Lingga island Riau Archipelago (Purwanto 1997). A *Palaquium oblongifolium* plantation of 788.31 hectares and a seed orchard of 34.11 hectares have also been established by State Plantation XI Ltd. (now State Plantation VIII Ltd.) in Cipetir, Sukabumi, in the Province of West Java of Indonesia (PT

Perkebunan XI 1986). In 2011, *Palaquium oblongifolium* plantations in that area were approximately 283 hectares (PT Perkebunan Nusantara VIII 2011). Therefore, gutta-percha, a renewable elastomer, offers a promising source of sustainable and environmentally benign adhesive materials for the alternative synthetic resins that are increasingly imported by Indonesia. In 2010, Indonesia imported amino resins, phenolic resins, and polyurethanes, the weight of which amounted to 59.894 tons (Indonesia Ministry of Trade 2011).

Although the adhesive potential of gutta-percha (*trans*-1,4-isoprene rubber (TIR)) has not been investigated to a great extent, some works related to the modification of TIR have been conducted. In the absence of peroxide, maleic anhydride-modified synthetic TIR (MTIR) was successfully grafted to the OH group of wood flour through esterification (Febrianto *et al.* 1999; 2001). These authors found that the affinity between wood flour (filler) and polymer matrix was low. The addition of additives or a modifier such as maleic anhydride (MAH) was thought capable of improving the adhesion between wood flour and the polymer matrix. The addition of additives was also suggested by Youngquist (1999) to improve bonding between thermoplastic and wood components. MAH was introduced into a polymer through the addition of radical coupling in the presence of peroxide to form MAH-modified polymer (Felix and Gatenholm 1991; Gaylord and Mishra 1983). Furthermore, reaction of TIR with 5% MAH and 15% benzoyl peroxide (BPO) (based on MAH weight) in a kneader at 150 ºC and 30 to 70 rpm for 10 min produced MTIR useful for hot melt adhesive (Febrianto *et al.* 2006). However, the solid state of the adhesive complicates its application.

To solve these problems and reduce manufacturing costs, in the present study, a gutta-percha-based adhesive was developed using the dissolution technique. The quality of the resulting adhesive was evaluated by applying it to cold-pressed laminated wood product.

EXPERIMENTAL

Materials

Gutta-percha with 4.15% moisture content containing 95.15% gutta was supplied by a gutta-percha processing factory, PT Perkebunan Nusantara VIII Sukabumi, West Java (Fig. 1). Maleic anhydride (MAH), benzoyl peroxide (BPO), and toluene (boiling point of 110.6 °C, density of 0.867 g mL^{-1}) were purchased from Bratachem chemical, Bogor, West Java. Eight-year-old sengon wood (*Paraserianthes falcataria* L. Nielsen) with an average density of 0.34 g cm^{-3}(0.33 to 0.36 g cm^{-3}) was collected from Jatinangor, Bandung Institute of Technology, West Java, Indonesia.

Methods

Chemical compound and melting and glass transition temperature analysis of gutta-percha

The chemical makeup of gutta-percha was identified using a Shimadzu GCMS-QP 2010 pyrolysis gas chromatograph mass spectrometer (Shimadzu Corporation; Kyoto-Japan). The sample was inserted into the quartz chamber in the pyrolysis unit, followed by heating in oxygen-free conditions at a temperature of 400 °C. The temperature of the column was set at 50 °C, with 0.85 mL min^{-1} column flow and 60-min retention time. Melting and glass transition temperatures were measured using a differential scanning calorimeter (DSC) Mettler TA 400 Thermal Analysis System (Schweizenboch, Switzerland), in which 5 mg of sample (flour) was used in the measurement. The sample was put in a 40-µL crucible, and the analysis was performed with an alternate heating-cooling temperature program of 30 °C - 70 °C - (-100) °C - 75 °C. Nitrogen gas with a flow rate of 20 mL min^{-1} was purged during the analysis.

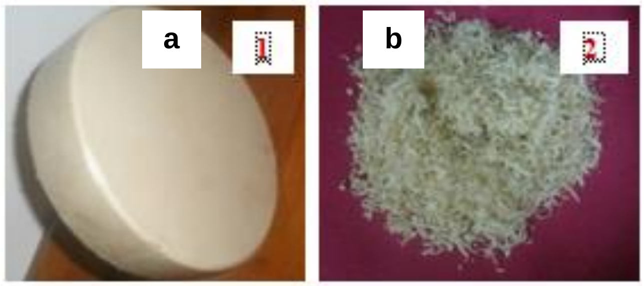


Fig. 1. (a) Gutta-percha solid and (b) gutta-percha granules

Preparation of gutta-percha-based adhesives

In the present work, toluene was used as a solvent for unmodified gutta-percha (UGP) and modified gutta-percha-based adhesives. Modified gutta-percha-based adhesives consisted of gutta-percha + 5% MAH (GPM) and gutta-percha + 5% MAH + 0.75% BPO (GPMB). During the adhesive preparation, solid gutta-percha was initially shredded into granules. Either with or without MAH, the granules were dissolved in toluene at gutta-percha to toluene ratios (w/w) of 15.0:85.0, 17.5:82.5, 20.0:80.0, and 22.5:77.5, and then heated in a water bath at 70 °C for 10 min with stirring and allowed to cool for 24 h. BPO was added to GPM adhesive before its application in laminated wood preparation.

Infrared spectroscopic measurement

Fourier transform infra-red (FTIR) spectroscopy ABB MB 3000 (Reliable FTIR Laboratory Analyser, Canada) was used to analyze the changes in functional groups due to treatment. A 2-mg sample of flour and 200 mg of potassium bromide (KBr) were mixed, homogenized, and put into the pelletizer tool. A pellet sample was placed in the holder and dissipated by infrared ray. FTIR spectra were observed in the wave number range of 2750 to 500 cm^{-1}, with 16 cm^{-1} resolution.

Measurement of the physical properties of gutta-percha adhesives

The physical properties of gutta-percha-based adhesives were characterized referring to the Indonesian standard (Badan Standardisasi Nasional 1998). This included the evaluation of the adhesive appearance, solid content, viscosity, and specific gravity.

Board manufacturing

Two-ply laminated boards of 200 mm x 80 mm x 20 mm were produced from sengon wood. Sengon laminae with moisture contents of 9 to 10% were glued with UGP, GPM, and GPMB with a glue spread of 250 gm^{-2} and assembled in a parallel pattern. The assembly was then clamped for 24 h at room temperature (20 °C and 65% RH) and finally conditioned for 7 d.

Evaluation of the properties of laminated wood

The performance of laminated wood (*i.e.*, moisture content, density, shear strength, and delamination ratio) was evaluated based on JAS 234 (Japan Plywood Inspection Corporation 2003). Block shear strength testing (shear area of 6.25 cm^2) was conducted in dry and wet conditions. The wet condition test was conducted by cold water immersion at 20 °C and 65% RH for 6 h, and by soaking in hot water at a temperature of 60 °C for 3 h. For the delamination test, the samples were first immersed in water at room temperature (20 °C) for 6 h and then dried at a temperature of 40 ± 3 °C for 18 h prior to being tested.

Data analysis

Data were analyzed using analysis of variance (ANOVA). The homogeneity of the mean among combinations was evaluated using Duncan's multiple range tests at a 95% confidence interval. Analyses were carried out with Statistical Analysis System (SAS) version 9.1 (SAS Institute; Cary, NC).

RESULTS AND DISCUSSION

Characteristics of Gutta-percha

The chemical components of gutta-percha were dominated by terpene groups consisting of 1,3 butadiene, 2-methyl (CAS) isoprene, geranyl linalool isomer, solanesol (polyisoprene), d-limonene, beta-elemene, gamma-elemene (monoterpenes), farnesene, and d-nerolidol (sesquiterpene and its derivatives) (Table 1, Fig. 2). The component with the largest concentration was 1,3-butadiene, 2-methyl-(CAS) isoprene. Gutta-percha has an isomer structure of *trans*-1,4-isoprene (Warneke *et al.* 2007) or *trans*-1,4-poly (methyl buta 1,3-diene (polyisoprene) (Cowd 1982; Rose and Steinbuchel 2005).

Table 1. Chemical Components of Gutta-percha

Compound	Concentration (%)
1,3 Butadiene, 2-methyl-(CAS) isoprene	24.61
Cyclohexene, 1-methyl-4-(1-methylethenyl)	2.89
d-Limonene (monoterpene)	16.00
Cloven n	3.33
1,5 cycloundecadiene, 8,8 dimethyl-9-methylene-(CAS)	5.56
Beta-elemene (monoterpene)	11.15
Farnesene (sesquiterpene)	5.37
Silane, Dimethyldi-1 propynyl	1.59
1-isopropenyl-3 propenyl-cyclopentane	1.05
Propane, 1,2-dibromo-(CAS) 1,2 Dibromopropane	2.34
Heptadiene	1.39
Geranyl linalool isomer (polyisoprene)	3.26
Cyclohexane	0.35
d-Nerolidol (sesquiterpene)	6.84
Cyclo heptane, 4-methylen-1-methyl-2	3.81
Gamma-elemene (monoterpene)	2.64
1,6,10,14,18,20-Tetracosahexane-3-OL (isoprene)	3.57
Tetracosahexane (isoprene)	1.78
Solanesol (polyisoprene)	2.47

Source: Center of Forest Product Research and Development (2013)

The glass transition (T_g) and melting temperatures of gutta-percha resin were -56.75 °C and 51.67 °C, respectively. They were different from those of synthetic trans-1,4-isoprene rubber reported by Nielsen and Landel (1994), *i.e.*, -60 °C and 74 °C, respectively. Glass transition and melting temperature differences between synthetic TIR and natural TIR are thought to be due to differences in the compound purity. Gutta-percha consists of various different compounds, whereas the synthetic TIR was a pure compound. In principle, melting is splitting of the crystalline regions of the polymer chain, thus allowing it to flow. Polymer properties may change as a result of glass transition change, a phenomenon related to the amorphous region (Cowd 1982).

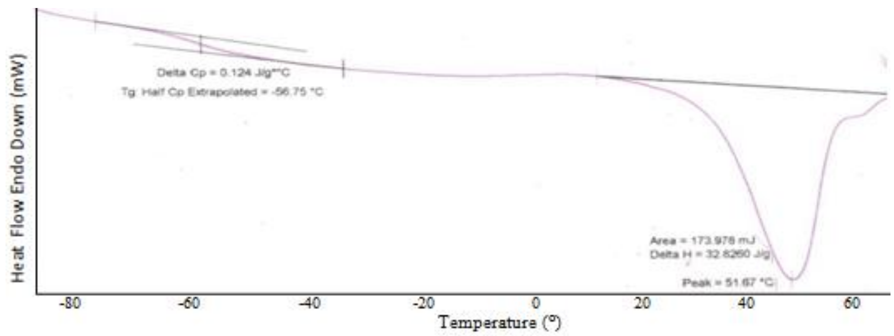


Fig. 2. The glass transition and melting temperatures of gutta percha

Characteristics of Gutta-percha-based Adhesive

The appearance (color), specific gravity, solid content, and viscosity of the resulting adhesives were examined in the present research. The color of UGP adhesive (Fig. 3a) was white to light beige. The GPM (Fig. 3b) and GPMB (Fig. 3c) adhesives were both beige.

Fig. 3. Appearance of gutta-percha-based adhesive at various ratios of gutta-percha to toluene; (a) UGP; (b) GPM; and (c) GPMB

The physical properties of gutta-percha-based adhesive, *i.e.*, specific gravity, solid content, and viscosity, are presented in Table 2. The specific gravity of the prepared gutta-percha-based adhesive was in the range of 0.86 to 0.89. Increasing the ratio of gutta-percha to toluene slightly increased the density of the resulting adhesives. The increase was thought to be related to the viscosity of the adhesive. The solid content of the adhesives was in the range of 13.58% to 24.10%. Increasing the amount of solvent certainly diluted the resulting adhesives. It can be seen from Table 2 that the viscosity of the adhesives was in the range of 18 to 80 poise. UGP prepared at the gutta-percha to toluene ratio of 15:85 retained the lowest viscosity, and the highest viscosity was obtained from GPM and GPMB prepared at a gutta-percha to toluene ratio of 22.5:77.5. It seemed that increasing the quantity of gutta-percha in adhesives increased the adhesive viscosity.

Table 2. Physical Properties (Specific Gravity (SG), Solid Content (SC), and Viscosity (Visc.)) of Gutta-percha-based Adhesive

Gutta-percha to toluene ratio (w/w)	Adhesive								
	Gutta-percha			Gutta-percha+MAH			Gutta-percha+MAH+BPO		
	SG.	SC. (%)	Visc. (poise)	SG.	SC. (%)	Visc. (poise)	SG.	SC. (%)	Visc. (poise)
15:85	0.86	13.58	18.0	0.87	14.41	22.0	0.87	14.70	21.5
17.5:82.5	0.87	17.92	28.5	0.87	18.10	35.0	0.88	18.05	36.0
20:80	0.88	21.30	42.0	0.88	21.19	45.0	0.88	21.25	46.0
22.5:77.5	0.88	23.32	72.0	0.89	23.27	75.0	0.89	24.10	80.0

Moisture Content and Density of Laminated Wood

The moisture content of laminated wood bonded with UGP, GPM, and GPMB produced in the present research was in the range of 12.90 to 13.37%, 12.97 to 13.13%, and 12.92 to 13.33%, respectively, as indicated in Table 3. The density of the produced laminated wood can also be seen in Table 3. The density values for laminated wood were in the range of 0.37 to 0.38 g cm^{-3}. Modification and the ratio of gutta-percha to toluene did not influence the moisture content and density of laminated wood. The density of the resulting laminated wood was higher than that of the original wood (*i.e.*, 0.33 to 0.36 g cm^{-3}). Adhesives have been reported to confer higher density to laminated wood compared to that of the original wood (Santoso 1995). Pressing load was also thought to increase the density of laminated wood made from low wood density, such as that of sengon wood used in the present experiments.

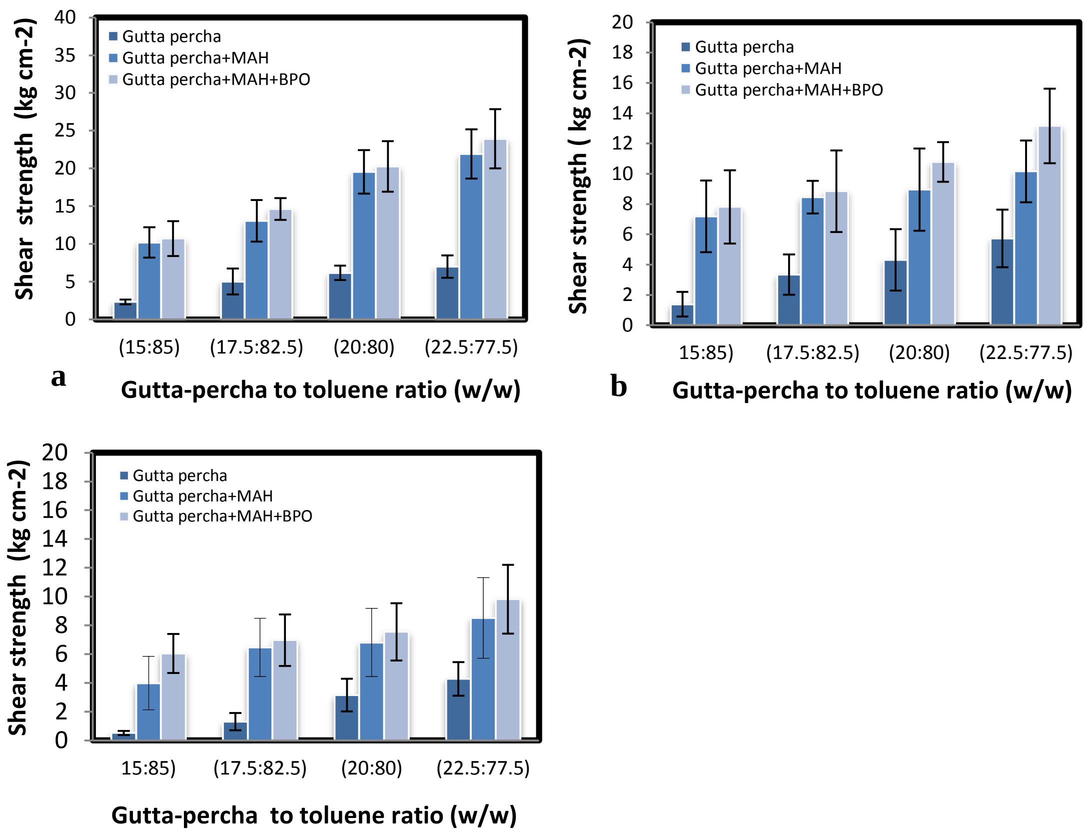
Table 3. Density and Moisture Content (MC) of Laminated Wood Glued with Gutta-percha-based Adhesive

Gutta-percha to toluene ratio (w/w)	Adhesive					
	Gutta-percha		Gutta-percha+MAH		Gutta-percha+MAH+BPO	
	Density (gcm^{-3})	MC (%)	Density (gcm^{-3})	MC (%)	Density (gcm^{-3})	MC (%)
15:85	0.37	12.97	0.38	13.13	0.37	13.00
17.5:82.5	0.37	12.90	0.38	13.10	0.38	13.33
20:80	0.37	13.09	0.38	12.97	0.38	12.92
22.5:77.5	0.38	13.37	0.38	12.99	0.38	12.96

Shear Strength and Delamination Ratio of Laminated Wood

The influence of the ratio of gutta-percha to toluene on the shear strength of laminated wood in dry conditions, soaked in cold water, and soaked in hot water is shown in Fig. 4. The shear strength of laminated wood in dry conditions (Fig. 4a), soaked in cold water (Fig. 4b), and soaked in hot water (Fig. 4c) was found to be in the range of 2.32 to 23.92 kg cm^{-2}, 1.39 to 13.16 kg cm^{-2}, and 0.52 to 9.82 kg cm^{-2}, respectively. For all measurement conditions, the highest shear strength was achieved by GPMB-bonded laminated wood at the ratio of gutta-percha to toluene of 22.5:77.5, and the lowest shear strength was achieved by UGP-bonded laminated wood at the ratio of gutta-percha to toluene of 15:85.

At the same ratio of gutta-percha to toluene, modified gutta-percha-based adhesives resulted in higher shear strength of laminated wood compared to that of unmodified gutta-percha. It was apparent that either modified or not, increasing the amount of gutta-percha in gutta-percha-based adhesives linearly increased the shear strength of the resulting laminated wood. It was also clear that the shear strength of laminated wood in dry conditions was higher than that soaked in cold water (which was higher than that soaked in hot water).



c

Fig. 4. Shear strength (kg cm^{-2}) of laminated wood (a) in dry conditions, (b) after soaking in cold water, and (c) after soaking in hot water

The present results indicate that modification of gutta-percha and the ratio of gutta-percha to toluene influenced the shear strength of laminated wood for all evaluation conditions. The shear strengths of laminated wood bonded with GPM and GPMB were significantly different from those of laminated wood bonded with UGP. At the same ratio of gutta-percha to toluene for all evaluation conditions, the shear strength of laminated wood bonded with GPMB was not significantly different to that of laminated wood bonded with GPM. The shear strength of cold water-soaked and hot water-soaked laminated wood at gutta-percha to toluene ratios of 22.5:77.5 and 20:80 were significantly different from that at gutta-percha to toluene ratios of 15:85 and 17.5:82.5. When tested in dry conditions, the shear strength of laminated wood at gutta-percha to toluene ratios of 22.5:77.5 and 20:80, with a gutta-percha to toluene ratio of 17.5:82.5, and with a gutta-percha to toluene ratio of 15:85 were significantly different from each other.

The influence of gutta-percha to toluene ratio on the delamination ratio of laminated wood bonded with UGP, GPM, and GPMB is shown in Fig. 5. The delamination ratios of laminated wood bonded with UGP, GPM, and GPMB were in the ranges of 1.60 to 10.19%, 0.98 to 5.59%, and 0.55 to 2.25%, respectively. It can be seen from Fig. 5 that the use of

UGP at a gutta-percha to toluene ratio of 15:85 resulted in the highest delamination ratio of laminated wood, and the lowest was that with the use of GPMB at the gutta-percha to toluene ratio of 22.5:77.5. It is obvious that at the same gutta-percha to toluene ratio, modification of gutta-percha with MAH and MAH + BPO decreased the delamination ratio of these adhesives bonded to laminated wood. In decreasing order (from the highest to the lowest), the delamination ratio of the laminated woods were those bonded with UGP, GPM, and GPMB. Furthermore, either modified or not, increasing the content of gutta-percha in the adhesives linearly decreased the delamination ratio. Smaller delamination ratios indicated better performance of laminated wood.

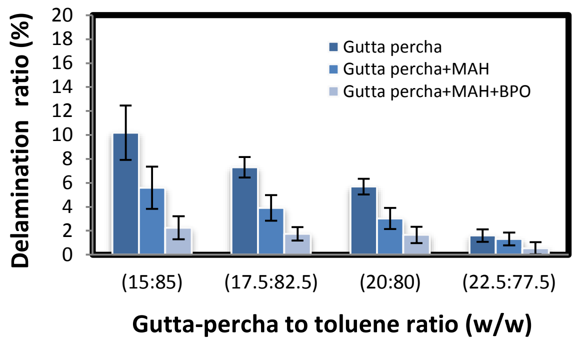


Fig. 5. Delamination ratio of laminated wood with gutta-percha-based adhesive at various ratios of gutta-percha to toluene

The present work indicates that modification of gutta-percha and gutta-percha to toluene ratio influenced the delamination ratio of laminated wood. The delamination ratios of laminated wood bonded with GPM and GPMB were not significantly different, but both of them were significantly different from that of laminated wood bonded with UGP. The delamination ratio of laminated wood bonded with adhesives prepared at gutta-percha to toluene ratios of 22.5:77.5 and 20:80 was significantly different, and that for adhesives between 15:85 and 17.5:22.5 was also significantly different.

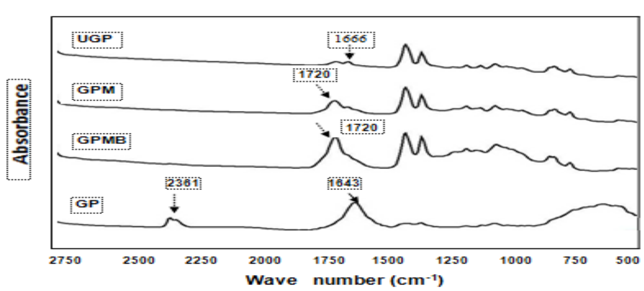


Fig. 6. FTIR spectra of gutta-percha-based adhesive (Gutta-percha solid (GP), UGP, GPM, and GPMB)

It is clear that laminated wood bonded with GPM and GPMB had higher shear strength and lower delamination ratio. These improvements can be attributed to the enhanced formation of maleated modified gutta-percha (trans-1,4-isoprene rubber) (MTIR). The infra-red spectra of gutta-percha, gutta-percha + toluene, gutta-percha + MAH, and gutta-percha + MAH + BPO is shown in Fig. 6. Two peaks occurred in gutta-percha with high absorbance at wave numbers 1.643 cm^{-1} (C=C stretching) and 2.361 cm^{-1} (O-H stretching vibration band of water molecule absorb on the surface of the sample due to the presence of moisture). The addition of toluene to gutta-percha reduced the absorbance of the 1.643-cm^{-1}peak and brought about the disappearance of 2.361-cm^{-1}peak. A peak at 1.720 cm^{-1} was found in both FTIR spectrographs of GPM and GPMB, although absorbance of the peak in GPMB was higher than that in GPM. This peak is indicative of the presence of the C=O group of MAH, in accordance with data previously reported by Creswell *et al.* (1972) that the C=O group appeared at 1.800 to 1.650 cm^{-1}; the C=O peak of TIR gutta-percha + MAH (MTIR) appeared at 1.716 cm^{-1} (Febrianto *et al.* 2006).

Modified gutta-percha-based adhesives effectively increased the shear strength of laminated wood by approximately 3 to 4, 2 to 6, and 2 to 12 fold when tested in dry conditions, after cold water soaking, and after hot water soaking, respectively (compared to these of UGP adhesives). Either bonded with modified or unmodified gutta-percha adhesives, the improvement in laminated wood shear strength at low gutta-percha contents was higher than that at high gutta-percha contents. Mechanical adhesion was thought to be responsible for the bonding strength of laminated wood glued with UGP adhesives (Febrianto *et al*. 2006). In mechanical adhesion, adhesive penetrates wood pores and the cell wall cavity and then hardens, anchoring the bonded wood (Tsoumis 1991). On the contrary, both mechanical and specific adhesion occurred when laminated wood was glued with GPM and GPMB adhesives. In specific adhesion, chemical bonding between the hydroxyl groups of wood with MTIR played an important role. The use of MAH as a modifier to improve composite properties has been reported by previous investigators. Febrianto *et al.* (1999, 2006) used MAH-grafted TIR (MTIR) as a compatibilizer and a hot melt adhesive. Lu *et al*. (2002), Kishi *et al.* (1989), and Kord (2011) used grafted MAH and poly propylene (MAPP). Barra *et al.* (1997) and Borggreve and Gaymans (1989) used MAH grafted to EPDM. The shear strength of laminated wood glued with GPMB adhesive was slightly higher than that bonded with GPM adhesive. This was assumed to be due to the role of BPO. Peroxide has an important role as an initiator through the free radical degradation mechanism (Bremner and Rudin 1993; Gaylord and Mishra 1983). The proposed reaction mechanism of grafting through esterification of MTIR by the OH group in wood is presented in Fig. 7.

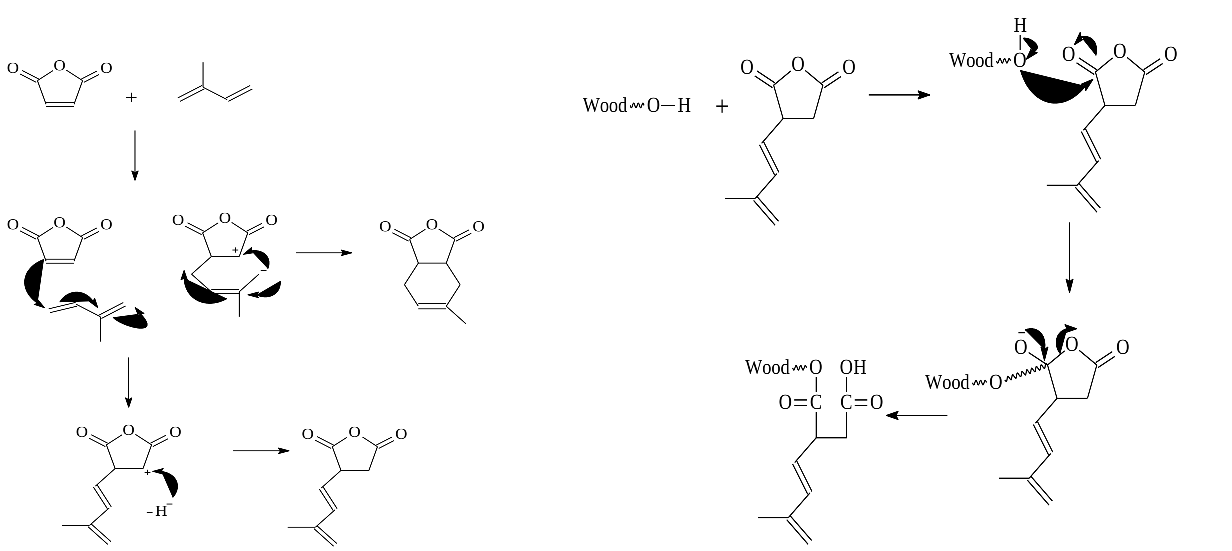


Fig. 7. Proposed reaction mechanism between MAH and TIR (left), and between wood and MTIR (right)

CONCLUSIONS

1. Terpenes, especially 1,3 butadiene, 2-methyl-(CAS) isoprene (trans-1,4-isoprene) (polyterpene), were the dominant chemical components of gutta-percha. The glass transition and melting temperatures of gutta-percha were -56.75 °C and 51.67 °C, respectively.
2. The modification and the ratio of gutta-percha to toluene did not significantly influence the moisture content or density of laminated wood.
3. The shear strength and delamination ratio of laminated wood bonded with gutta-percha-based adhesives were significantly influenced by the modification treatment and the ratio of gutta-percha to toluene. The modification of gutta-percha with maleic anhydride (MAH) and benzoyl peroxide (BPO) as an initiator resulted in an improved performance of the laminated wood. The higher the amount of gutta-percha in the adhesives was, the better the shear strength and delamination ratio of laminated wood.
4. Infra-red spectra showed that a new peak occurred at a wave number of 1.720 cm^{-1} in MAH-modified gutta-percha and MAH + BPO-modified gutta-percha-based adhesive, indicating the C=O bond of MAH.

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