Chapter 5

A Review of Isocyanate Wood Adhesive: A Case Study in Indonesia

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Abstract

The use of isocyanate adhesive for the binding of wood and wood products has been increasing in Indonesia particularly for research needs since wood products bonded by glue-based formaldehyde release formaldehyde emission that have been found to have carcinogenic effect and may lead to sick house syndrome. There are at least two types of isocyanate commonly used in Indonesia, namely isocyanate cross-linker and isocyanate alone. Isocyanate cross-linker is used together with polyvinyl alcohol (PVA) forming a water-based polymer-isocyanate emulsion; thus, its application using spreading technique for binding engineered wood products such as glue laminated timber (glulam) and laminated veneer lumber (LVL). For isocyanate alone, because its viscosity is adequate for spraying, it is preferably used for producing wood-based panels, especially particleboard and fiberboard. In this chapter, the characteristics of both types of isocyanate usually used in Indonesia are presented. Some research studies of the authors are also provided.

Keywords: isocyanate, wood adhesive, characteristics

1. Introduction

In order to reduce the formaldehyde emission originated from both adhesive-based formalin and wood products bonded by resin-based formaldehyde, such as urea-formaldehyde (UF), phenol-formaldehyde (PF), melamine-formaldehyde (MF), or a mixture of the two, i.e., melamine-urea-formaldehyde (MUF) or urea-melamine-formaldehyde (UMF), the use of either natural adhesive (bio-adhesive) or isocyanate adhesive has been slightly increased for research purposes in Indonesia. Bio-adhesives have been used by human for thousands of years [1] even though they have limitations in strength and durability [2–4]. On the contrary, isocyanate has the advantage of high strength and resistance and is also stable although it is applied on treated wood [5]. Therefore, the latter has been extensively used, particularly in research of wood products.

Isocyanates have been mainly used for wood adhesive in two ways: first, as a urethane prepolymers originated from isocyanate-polyol reaction products recently being used in the wood-laminating industry, and second, as the isocyanate currently being used in the particleboard industry [6]. The type of isocyanate generally used for binding wood laminated-based products is the water-based emulsion adhesive with isocyanate as the cross-linker [7], while the type of diisocyanate generally used for particleboard manufacture is MDI or 4–4′-diphenylmethane diisocyanate [6]. Further, fiberboard mills involved in particleboard manufacture use MDI as the binder [8, 9]. The method of mass production for particleboard and fiberboard using isocyanate adhesive has been patented in Europe since 2013 [10].

When isocyanate resins are used as a binder for wood materials, the resins react with the wood component [5] and water. However, if water is present in the wood materials, the isocyanate resins would react with the water in preference to the wood component. Therefore, the isocyanate resin-water reaction is considered as the one of the most important reactions when bonding wood composite materials with isocyanate resins.

In this chapter, a review of isocyanate applied on wood would be presented. Types of isocyanates including the chemical composition, their origin or history, properties with an emphasis on thermal behavior, application on the wood products, previous and on-going works of ours have been also explained.

2. History and application of water-based polymer isocyanate in Indonesia

According to our best knowledge, the use of isocyanate in Indonesia originated from the introduction of the product by two Japanese producers of water-based polymer isocyanate (WBPI), namely Koyo Sangyo Co, Ltd. and Oshika Corporation.

Koyo Sangyo then opened the branch factory (PT. Koyolem Indonesia) in 1993 and distributed the WBPI under the trade name Koyobond®(www.koyoweb.com) [12]. Likewise, a joint venture company (PT. Poly Oshika) was established in 1995 between Oshika Corporation and PT. Polichemie Asia Pacific for the production of aqueous polymer isocyanate (API) adhesive in Indonesia under the trade name of PI Bond®(www.polychemie.co.id) [13]. API glue is the same as WBPI, a common name for the adhesive system consisting of an aqueous PVA solution with an isocyanate cross-linker [7]; nowadays, it is also called emulsion polymer isocyanates (EPI) [14]. This information is in accordance with the statement of Grøstad and Pedersen [7], who mentioned from a business point of view that WBPI is naturally concentrated in the Asian markets, presumably including Indonesia. In contrast, non-Asian consumption of this type of adhesive is still limited, although it is slightly increasing.

Application of isocyanate adhesive for gluing wood in Indonesia is in the form of isocyanate for a cross-linker, which is mixed together with PVA, forming a WBPI system for binding

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engineered wood products such as glulam and LVL by the spreading technique, as shown in **Figure 1**. Glulam is a bonded wood product that is suitable for many applications because it can be utilized as a structural component for settlement construction or as a light structural component in buildings, replacing solid wood products from natural forests, which have been reduced in supply due to enormous logging, forest fire, forest conversion into plantation, and other factors. Furthermore, glulam is considered to be the best alternative material for larger structural components, because it can be manufactured from small laminated lumber.

The best example of Indonesian research on glulam is the work of Herawati et al. [15]. They investigated the quality of glulam made of two Indonesian wood species, namely African wood (*Maesopsis eminii*) and mangium (*Acacia mangium*), and bonded by WBPI with the trade name PI 3100® purchased from PT.Polychemie Asia Pacific, Indonesia. Before they are made into glulam, the sawn lumbers were graded into three classes of modulus of elasticity (MOE) using Panter/Plank-sorter of machine stress grading (MSR), namely E1, E2 and E3. With the composition as shown in **Figure 2**, the glulam was constructed of both balanced and unbalanced combination.

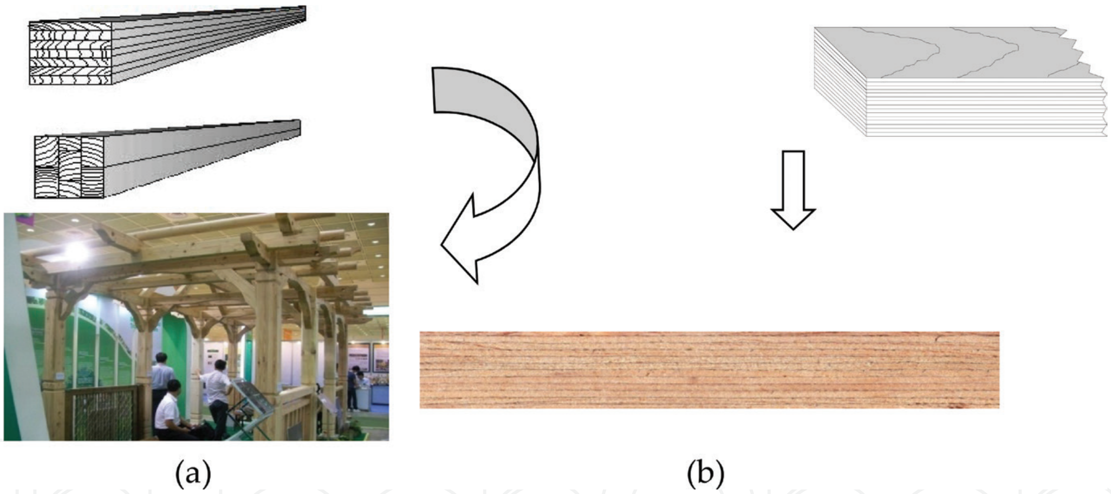

Figure 1. (a) Glulam and (b) LVL, both wood engineered products, were usually glued with a spreading method using an adhesive mixture consisting of PVA and isocyanate cross-linker in the ratio of 100:15 [11].

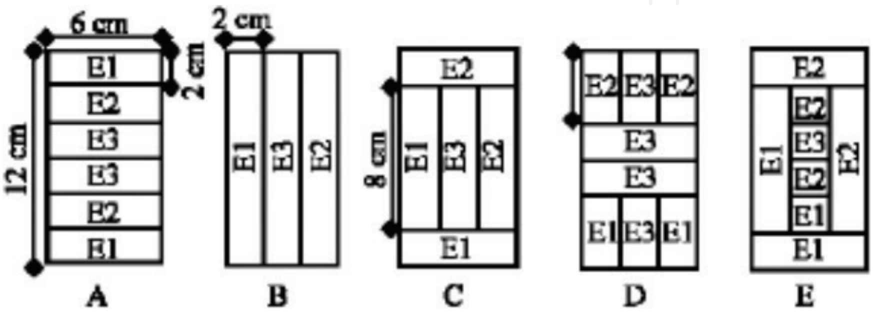

Figure 2. Composition of glulam, work of Herawati et al. [15].

Another product that is usually bonded by WBPI is LVL. It is composed of veneers glued with grain running parallel to each other for reducing the natural variability of wood. Usually the veneer of the higher quality is placed on the outside. Alamsyah et al.[16] investigated the quality of LVL made of three wood species from Indonesia, namely *Paraserianthes falcataria*, *Pinus merkusii*, and *A. mangium*, and bonded by WBPI (API with trade name of KR Bond-7800®, Koyo, mixed with 15 parts cross-linking agent) at 250 g/m^2 spread rate. They treated the LVL using various physical treatments such as boiling and soaking while the mechanical evaluation consisted of block shear test and contact angle test. Mixing of the glue was done in accordance with the supplier's instruction. Details of the main component of the API adhesive used in their study were presented. **Table 1** exhibits the properties of an aqueous polymer PVAc (polyvinyl acetate) as the resin containing a reactive aqueous polymer. PVAc was produced by hydrolysis of PVA [7].**Table 2** presents the characteristics of the isocyanate cross-linker.

Koyobond® is composed of two components: a resin containing a reactive aqueous polymer and an isocyanate cross-linker. The cross-linker reacts with active groups of not only aqueous polymer but also wood to produce strong chemical bonds.

Further, similar work of Alamsyah et al. [16] included lengthening using eight Indonesian wood species namely EX or *Eucalyptus cyclocarpum*, PF or *P. falcataria*, S or *Shorea* sp., TS or *Toona sinensis*, GA or *Gmelina arborea*, PM or *P. merkusii*, AM or *A. mangium* and AH or *A. hybrid* in order to understand the application and influence of WBPI on wood, the treatment and the testing. The treatment also referred to previous work namely normal or control (N), cyclic boiling (CB), vacuum pressure soaking (VPS) and vacuum pressure

No	Parameter	Properties
1	Appearance	White fluid
2	Viscosity at 25°C	60±20 poise
3	Solid content	58±3%
4	pH	7.5±1.0

Source: PT.LemindoAbadi Jaya [17].

Table 1. Typical properties of PVAc resin (KR-7800®).

No	Remarks	
1	Cross-linker	AJ-1®
2	Mix ratio w/w (resin/cross-linker)	100/15
3	Press time at 25°C	60–120minutes
4	Wood species	"Hard-to-bond wood species" such as teak, sungkai, red oak

Source: PT.Lemindo Abadi Jaya [17].

Table 2. Typical properties of isocyanate cross-linker.

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soaking dry (VPSD) treatment. **Figure 3** shows the results of the shear strength test of the lamina made from various Indonesian wood species bonded by Koyobond® while **Figure 4** shows the performance of the wood products specimen bonded by Koyobond® after treatment. In this study, shear strength percentages can be used to predict the bond quality of wood-bonded products such as laminated wood. De-lamination is an indicator of how well the bonded joint withstands severe swelling and shrinking stresses in the presence of high moisture and heat [18]. Adhesion using WBPI in this study was resistant to high moisture and heat.

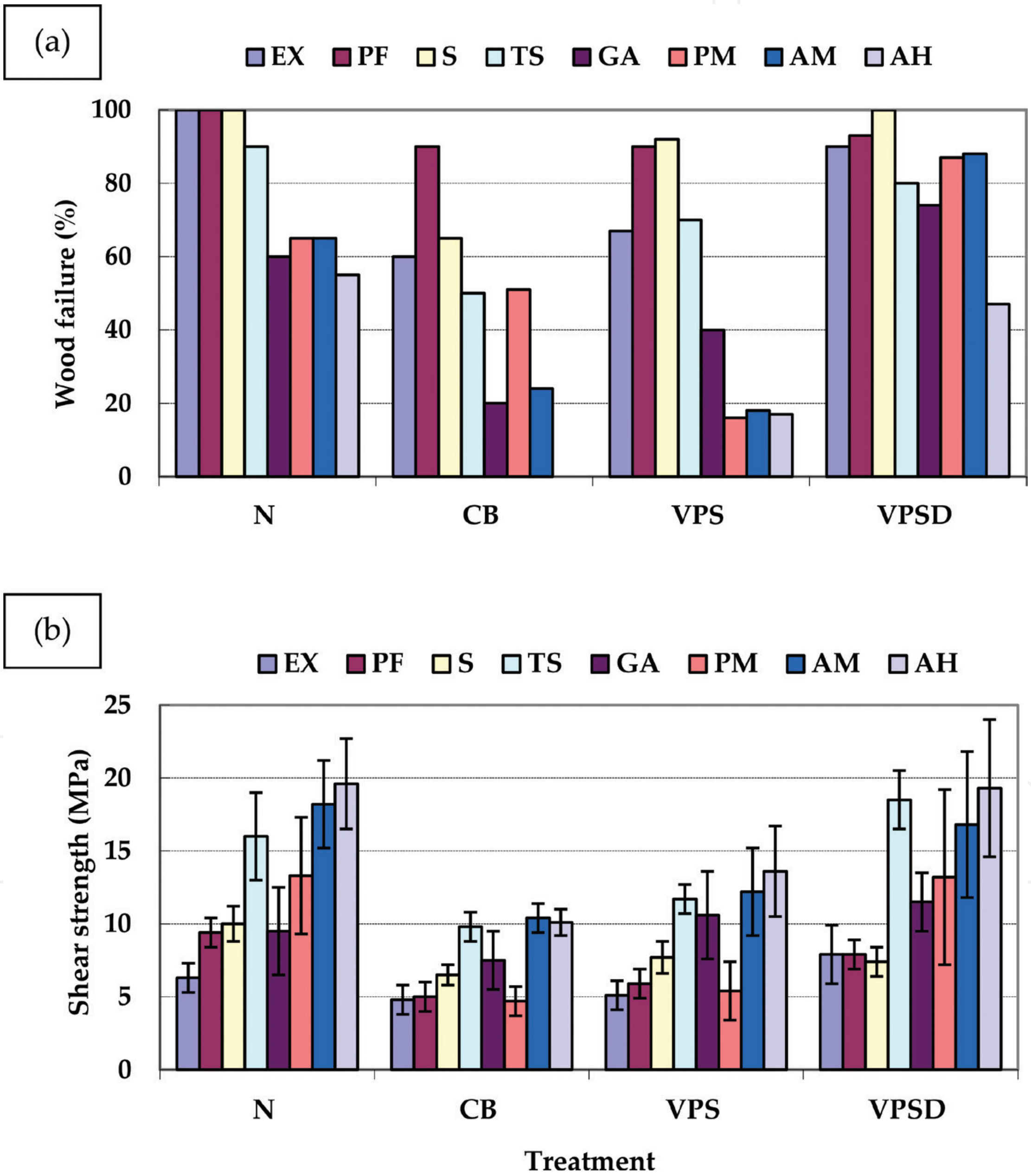

Figure 3. (a) Wood failure and (b) shear strength of laminates bonded with API made from various Indonesian wood species and in various treatments. Error bars indicated a standard deviation [19].

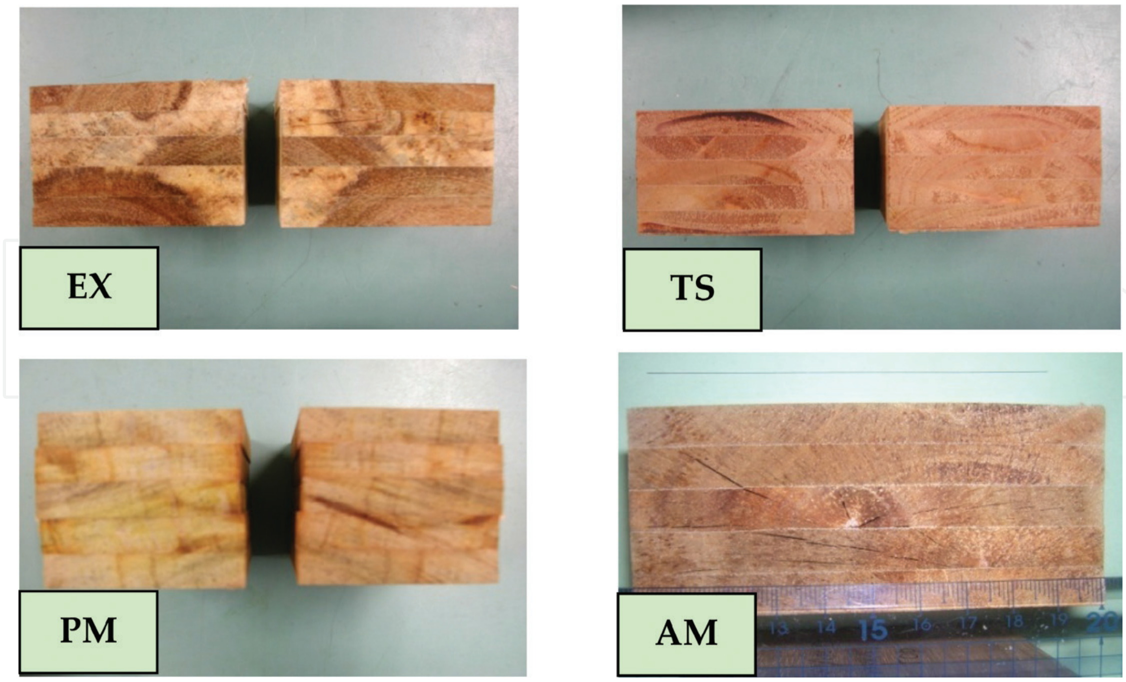

Figure 4. Specimen of wood laminates bonded with Koyobond®after VPS-2 treatment for de-lamination testing [19].

3. Isocyanate cross-linker and isocyanate alone

All isocyanates of industrial importance, including the isocyanate cross-linker used in WBPI, contain two or more isocyanate groups (–N=C=O) per molecule. Isocyanates have good bonding because their –NCO group can react with compounds having active hydrogen such as water, including wood with water therein (free and bonded water) [20–22]. An excellent review and an experiment result published more than three decades ago presented evidence that isocyanates also reacted with the wood component, particularly the cell wall part [5, 23].

To our best knowledge, isocyanate adhesive is a general term for a variety of esters, which rely mainly on phosgene synthesis in conventional synthesis industries. As of now, toluene diisocyanate (TDI) and diphenylmethane diisocyanate (MDI) are the most commercially important diisocyanates [24]; they are widely used and have the largest output [25]. TDI has quite a high vapor pressure; hence, increased health risks involved with the use of this isocyanate are significant [7]. MDI is produced from the reaction between aniline and formaldehyde with hydrochloric acid as the catalyst. A complex mixture of isomeric diamines and oligomeric polyamines is formed. The 4,4′-diamine predominates. This complex mixture is phosgenated to give polymeric diphenylmethane diisocyanate (pMDI), rather than a purified diisocyanate, an adhesive. At ambient temperature, pMDI is a clear brown liquid with a viscosity of about 0.5 Pas and a low vapor pressure. It has an excellent shelf life as long as moisture is not present [20].

pMDI is a mixture of MDI monomer and the related methylene bridged polyphenyl polyisocyanates. In the forest products industry, the binder is commonly referred to as MDI or simply as “isocyanates.” This confusion of names is further complicated by the fact that polymeric MDI is not at all polymeric. Approximately one-half of the resin is diisocyanate monomer, while the rest is a complex oligomeric mixture of polyisocyanates with a degree of polymerization less than 12 [9].

In this discussion, the term "isocyanate cross-linker" is used interchangeably with the term "isocyanate alone" because in fact isocyanate alone originates from isocyanate cross-linker as a component of WBPI. In Indonesia, because of the limitation of the producers and suppliers of the isocyanate adhesive, research on wood products bonded by isocyanate was carried out using isocyanate cross-linker purchased from PT.Koyolem Indonesia and PT.Polichemie Asia Pacific.

Figure 5(a) shows the properties of PF resin adhesive and MDI. The color of MDI is brighter than that of PF, but the viscosity is appropriate for applications using the spraying technique for producing wood composite products such as particleboard and fiberboard.

Figure 5(b) shows the packaging of isocyanate cross-linker sold and distributed in Indonesia. Although there is a "hardener" label in the brochure and packaging as shown in the picture, this terminology is wrong. In this context, hardener means cross-linker. Details of the properties are presented in **Table 3**, which contrasts the characteristics of H3M® and H7® (the trade name of the isocyanate cross-linker sold by PT.Polichemie Asia Pacific). Both types of isocyanate cross-linker were capable of being used as adhesive for making fiberboard in laboratory scale [28]. According to the supplier, H3M® is suitable for bonding any wood species; however, H7® is more appropriate for gluing of lamina wood.

Figure 5. (a) Color comparison between PF resin adhesive and MDI; (b) the isocyanate cross-linker distributed and most used in Indonesia [26, 27].

Characteristics	Trade name	
	H3M®	**H7®**
Performance (color)	Brown, viscous	Brown, viscous
Solid content (%)	Min 98–100%	Min 98%
Viscosity (cps/25°C)	180 cps	150–200, ± 175 cps
Source: Polyoshika [29].		

Table 3. Properties of the most used isocyanate cross-linker in Indonesia.

Many researches involving these types of isocyanate cross-linker have been carried out by Indonesian scholars, and the results have been presented or published in conference proceedings or journal papers. For example: Nuryawan et al. [30] mixed UF resin with isocyanate cross-linker purchased from PT.Polyoshika Company in the ratio 100:15 (w/w based on the solid content) for bonding particleboard made of sawdust from the residue of plantation forest of acacia (*A. mangium*) and eucalyptus wood (*Eucalyptus* sp.). Nuryawan et al. [31] used the isocyanate cross-linker of H7® for bonding sawdust's particleboard made of wood industrial waste. Febrianto et al. [32, 33] used commercial MDI (type H3M®) purchased from PT.Polichemie Asia Pacific as the adhesive for oriented strand board (OSB) made from an Indonesian fast growing tree species and betung bamboo, respectively. Recently, Iswanto et al. [34] used isocyanate resin (H3M®) obtained from PT.Polichemie Oshika for binding particleboard made of sorghum bagasse. From these examples, we can sum up that isocyanate cross-linkers are capable of bonding lignocellulose material such as wood particle, bamboo, and even sorghum bagasse.

4. Chemical composition

More than 270 isocyanates were synthesized between 1934 and 1949 [35]. But today, MDI (4,4′-diphenyl methane diisocyanate) is predominant worldwide; it is the generic name of a product used in industrial settings. pMDI or polymeric MDI, the primary technical/commercial form of MDI, is actually a mixture that contains 25–80% monomeric 4,4′-MDI as well as oligomers containing 3–6 rings and other minor isomers, such as the 2,2′-isomer. The exact composition of pMDI varies with the manufacturer [36].

Therefore, in this chapter, we had to emphasize and characterize the most used isocyanate in Indonesia, particularly in the research area of particleboard and fiberboard. Even though we already know the trade name and the supplier as well as the manufacturer, labeling, and writing, the chemical name is suggested. Indeed, MDI or 4,4′-diphenyl methane diisocyanate has at least 10 isomers as stated in the website of PubChem [37], the Open Chemistry Database. The isomers are as follows:

1,1-methylenebis(phenyl)diisocyanate;

4,4′-diisocyanatodiphenylmethane;

4,4′-diphenylmethane diisocyanate;

4,4′-methylene bisphenyldiisocyanate;

4,4′-methylenebis(phenylisocyanate);

4,4′-methylenediphenyl diisocyanate;

diphenylmethanediisocyanate;

diphenylmethane-4,4′-diisocyanate;

methylene diphenyl diisocyanate;

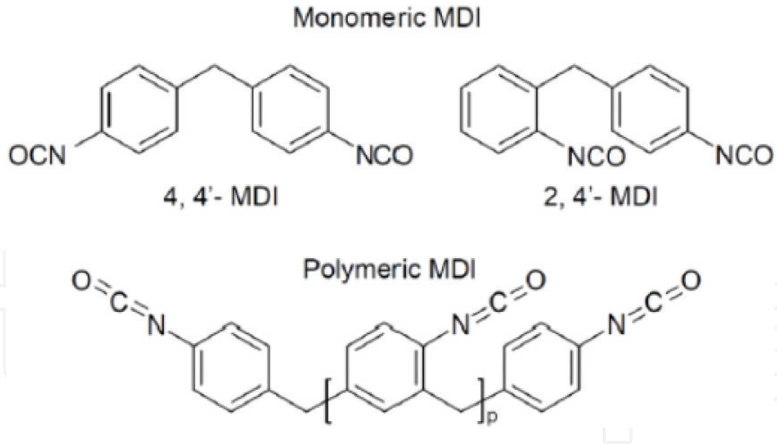

Figure 6. Monomeric MDI in 2 (ortho) position, in 4 (para) position, and its polymeric MDI [38].

p,p′-diphenylmethanediisocyanate.

The main effect of the isocyanate group (–NCO) on reactivity is in the 2 and 4 positions. The isocyanate group in the 2 (ortho) position is three times less reactive than the isocyanate group in the 4 (para) position as shown in **Figure 6**. In addition, pure 4,4′-MDI is solid at ambient temperature.

5. Isocyanate cross-linker applied on fiberboard under the cyclic test

Isocyanate cross-linker can be applied for bonding on either wood particle or wood fiber using spraying methods as shown in **Figure 7**.

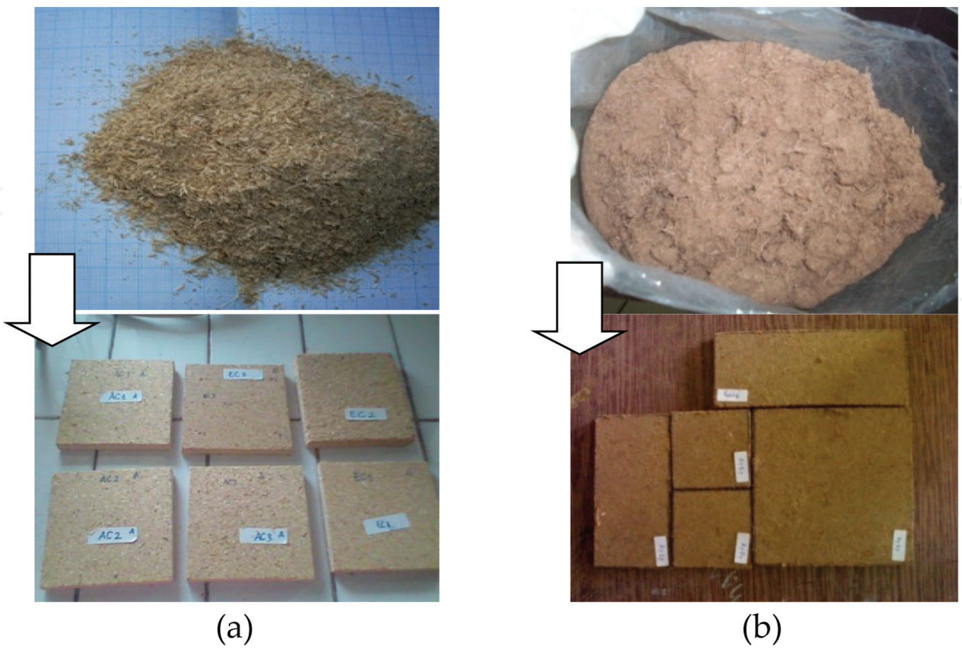

Figure 7. Typical wood composite products bonded with isocyanate alone using spraying method: (a) particleboard and (b) fiberboard [27, 39].

Nuryawan et al. [27] investigated the physical, mechanical, and performance properties of fiberboard bonded with H3M® and H7®. The result showed that the quality of both fiberboards was similar.

When cyclic evaluation was carried out, comprising water absorption and thickness swelling evaluation, surprisingly H3M® was much less compared to H7®. Moreover, the resistance of the fiberboard bonded by H3M® was stronger after cyclic test. This means that the fiberboard bonded with H3M® showed higher dimension stability compared to the fiberboard bonded with H7® as shown in **Figure 8**.

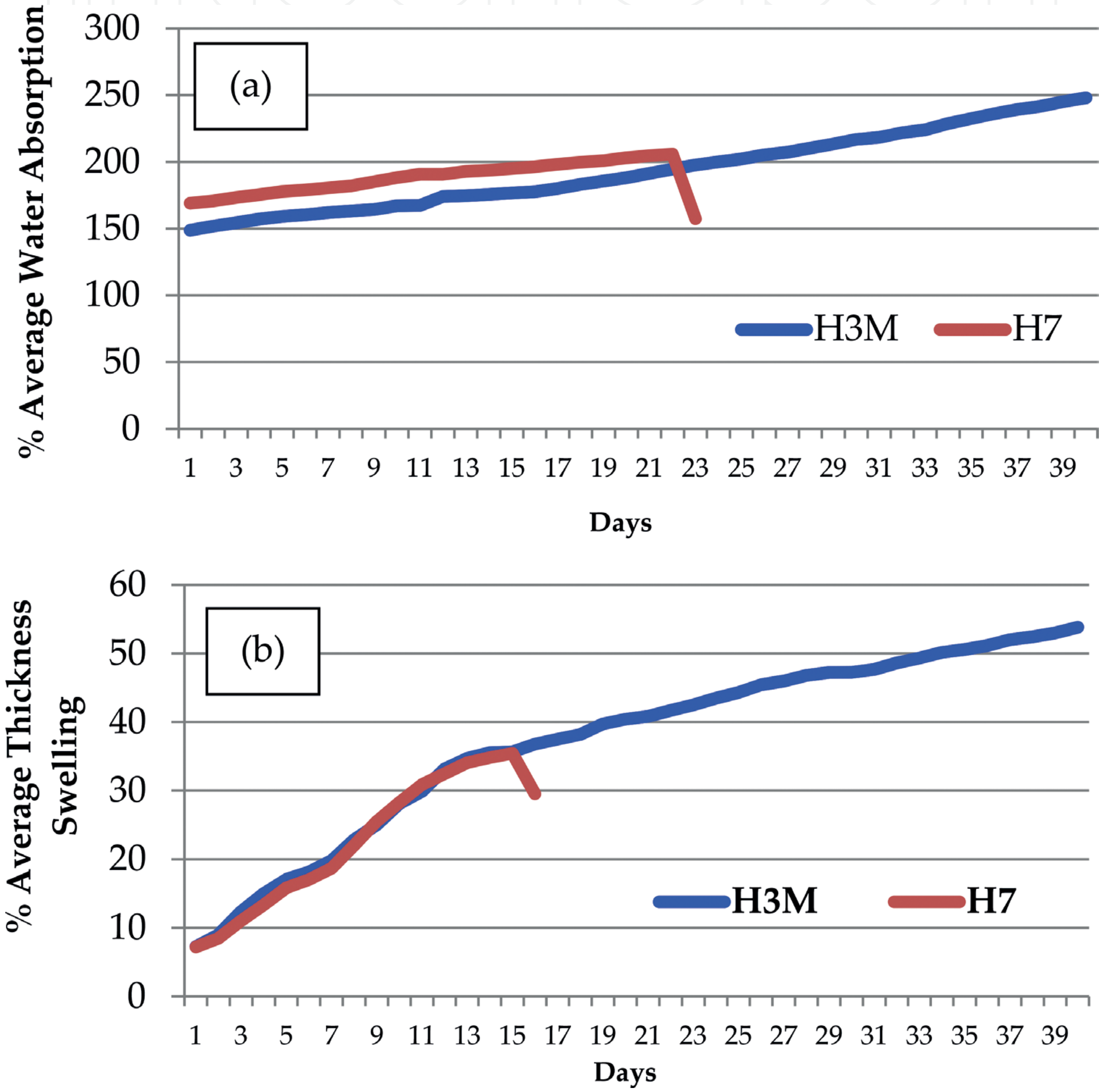

Figure 8. Cyclic test comprising (a) water absorption and (b) thickness swelling showed application of H3M®as binder was much longer in life use and stronger in resistance compared to H7®[27].

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6. Other properties of the isocyanate alone

The type of isocyanate alone adhesive used is emulsion polymer isocyanate (EPI). EPI can react with water. On the contrary, pMDI is not dispersible in water because it is oil-borne; therefore, it has to be blocked using blocking agents such as sodium bisulfite ($NaHSO_3$) [40]. A block isocyanate is formed through a reaction between an isocyanate group (–NCO) and a compound containing an active hydrogen atom to block the –NCO. This product has the advantage of a long shelf life because the active isocyanate groups are masked and protected. Furthermore, it has a small amount of the isocynate groups and requires a high temperature to de-block and to make the –NCO group free [41, 42].

On-going researches have been employing different scanning calorimetry (DSC) to analyze the thermal properties of the isocyanate [43]. Results of the analysis of polymeric isocyanate adhesive and its mixture with sawdust are shown in **Figure 9**.

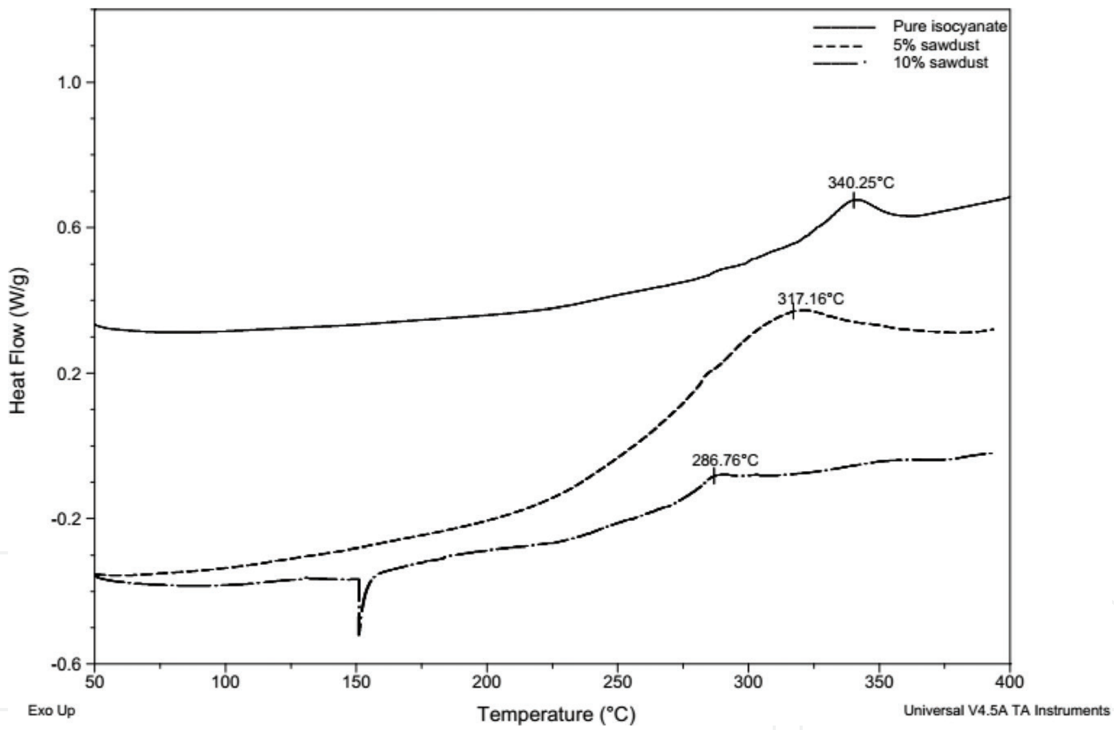

Figure 9. DSC thermograms of pure isocyanate and its mixture with sawdust at different addition levels [43].

$$R—N{=}C{=}O \; + \; O{=}C{=}N—R \longrightarrow R—N{=}C{=}N—R \; + \; CO_2$$

Figure 10. Self-polymerization of isocyanate (–NCO) to form polymeric carbodiimide upon elimination of carbon dioxide [45].

Sample	Mass (mg)	β (°C/min)	T_o (°C)	T_p (°C)	ΔH (J/g)
Pure isocyanate	7.0	10	325.86	340.25	7.31
5% sawdust	6.3	10	300.76	317.16	5.44
10% sawdust	8.5	10	280.98	286.76	1.69

Source: Nuryawan and Alamsyah [43].

Table 4. Results of DSC analysis of isocyanate and its mixture with sawdust at different addition levels.

The result showed that polymeric isocyanate adhesive had a peak temperature (T_p) at 340.25°C. That was probably the self-polymerization temperature of polymeric isocyanate. It is known that self-polymerization of polymeric isocyanate occurs at temperatures above 300°C [44]. The reaction forms polymeric carbodiimide upon elimination of carbon dioxide. It is an exothermic reaction and can accumulate heat in the products. The reaction equation is shown in **Figure 10** [45].

Incorporation of sawdust into polymeric isocyanate adhesive decreased the T_p. As can be seen in **Table 4**, 5% addition of sawdust into polymeric isocyanate adhesive decreases the T_p to 317.16°C. Further addition, up to 10%, of sawdust decreased the T_p drastically to 286.76°C.

Polymeric isocyanates are known to react readily with hydrogen atom (H) in water and alcohols. It seems an addition of sawdust to polymeric isocyanate provides more active H in the system and accelerates the cure of polymeric isocyanate adhesive. The active H obviously originates from the water in sawdust. The moisture content of sawdust used was 6.2%. An increase of sawdust content resulted in greater water content in the adhesive mixture, which eventually reacted with polymeric isocyanate adhesive and decreased the T_p. Moreover, it is known that the polymeric isocyanate adhesive also reacts with hydroxyl groups (–OH) from wood. This type of reaction produces urea or urethane bond as can be seen in **Figure 11** [46].

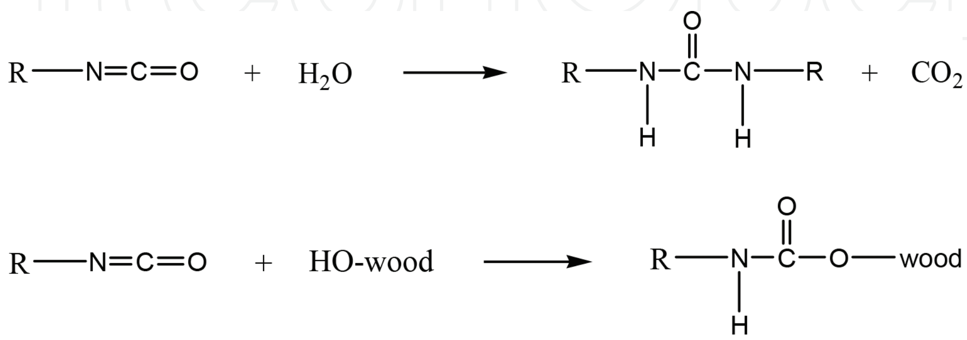

Figure 11. Reaction of isocyanate (–NCO) with water and wood containing water resulted in strong bonds and release of carbon dioxide [43].

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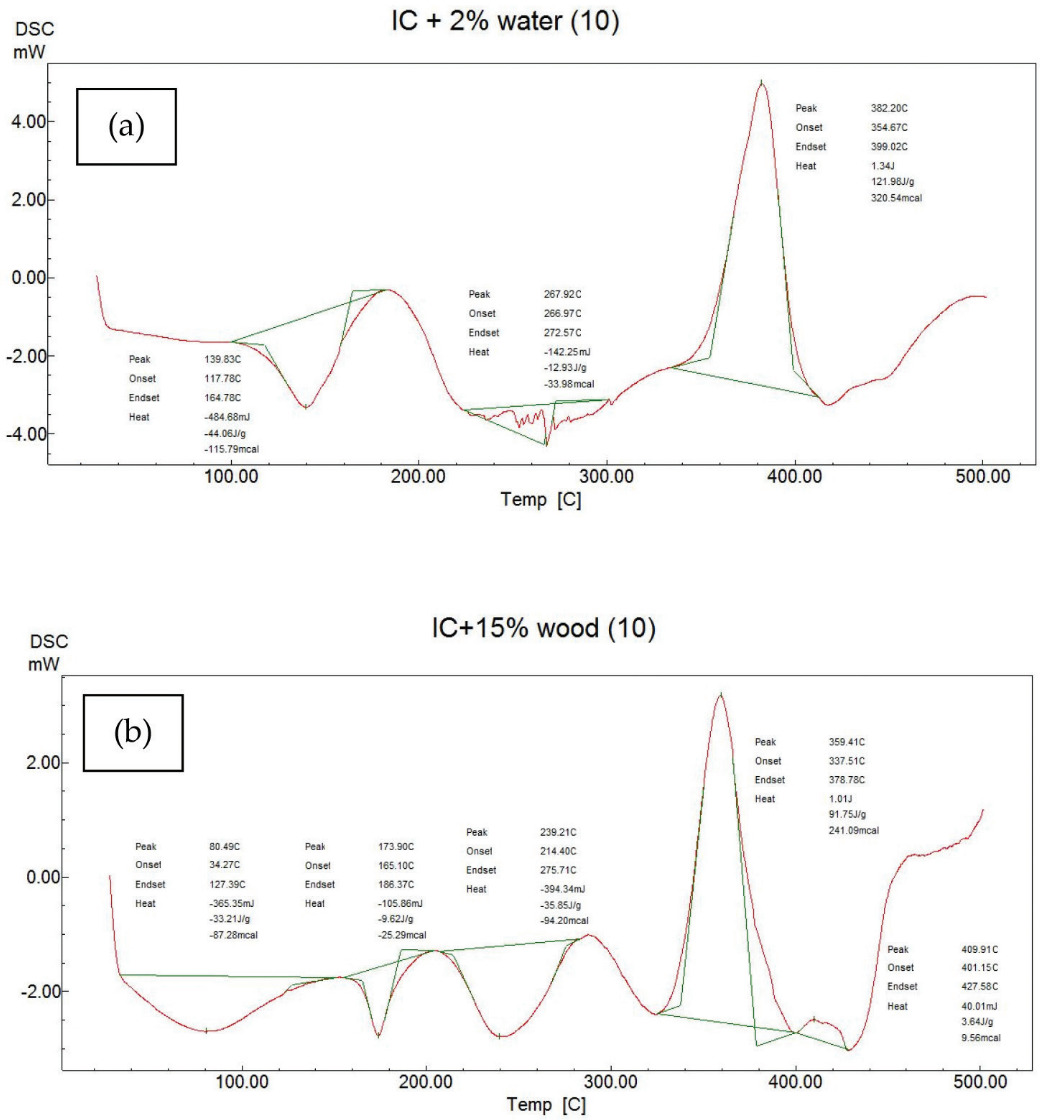

Figure 12. DSC thermogram of isocyanate films; (a) film cured with 2% water loading and (b) film cured with 15% wood particle loading [43].

When isocyanate is used as an adhesive in the particleboard system, the reactive –NCO group reacted with water (because wood is a hygroscopic material and contains free water and bound water) and also with the –OH group from wood (same as the reaction described earlier). Therefore, to study the curing behavior of the isocyanate curing using either water or wood in the form of sawdust, we scan film isocyanate curing using either water or wood under DSC as shown in **Figure 12**.

According to the peaks shown in the thermogram of the DSC scan (**Figure 12)**, there were other components within the isocyanate that have different molecular weight (**Figure 12a**); also wood component reacted with the isocyanate (**Figure 12b**).

Therefore, advance analysis such as gas chromatography-mass spectrometry (GCMS) is needed to clarify this phenomenon. Example of GCMS analysis of isocyanate H3M®

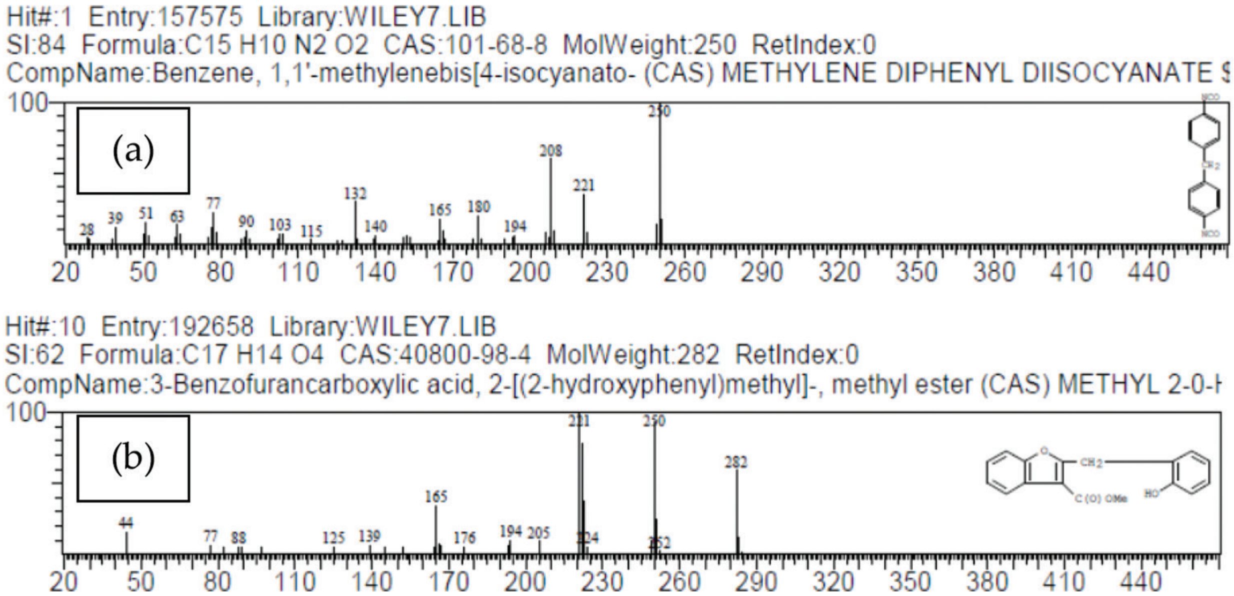

Figure 13. Analysis GCMS of isocyanate H3M® resulted in difference in species and molecular weight such as (a) MW = 250 and (b) MW = 282 [43].

resulted in not only difference in the molecular weight but also chemical species as shown in **Figure 13**.

7. Summary

According to the best of our knowledge, two manufactures in Indonesia, namely PT.Koyolem Indonesia and PT.Polychemie Asia Pacific, have been producing WBPI adhesive consisting of aqueous PVA and isocyanate cross-linker. For research needs, WBPI is used as an adhesive for glulam and LVL production while isocyanate cross-linker (alone) with the trade name H7® and H3M® has been used for bonding in OSB, particleboard, and fiberboard. For optimizing the properties of wood products bonded by isocyanate, the thermal properties have to be investigated such as curing behavior; peak temperature resulted in curing by either water or hygroscopic wood, or high heating for de-blocking isocyanate blocking. Another analysis such as GCMS is important for clarifying the molecular weight of species within the adhesive/glueline (film) system.

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