

Effects of Alkali Treatment and Polyisocyanate Crosslinking on the Mechanical Properties of Kraft Fiber-Reinforced Unsaturated Polyester Composites

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The effects of alkali treatment and polyisocyanate crosslinking on the mechanical properties of kraft fiber-reinforced UPE composites were investigated by means of tensile evaluation, SEM analysis, and XRD analysis. The results indicated that the alkali treatment decreased the tensile strength of the prepared composite before aging from 121 MPa to 97 MPa due to the decreased degree of crystallinity of the alkali-treated kraft fiber. Polyisocyanate crosslinking could apparently improve the mechanical properties and stability in terms of a 43% increase of non-aged tensile strength and 52% increase of hydrothermal-aged tensile strength compared with the controlled composite without crosslinking modification, which was attributable to the formation of strong chemical bonding between the interfaces of kraft fiber and polyester.

Keywords: Wood-plastic composites; Kraft fiber; Unsaturated polyester; Alkali pre-treatment; Polyisocyanate; Crosslinking; Mechanical properties

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INTRODUCTION

In recent decades, wood-plastic composites (WPCs) and reinforced thermoplastics with wood fiber or wood flour have become increasingly attractive due to their particular advantages, such as low density, high specific strength, no free formaldehyde release, better rigidity and stiffness than plastics, ability to reduce some plastic disposal, low equipment abrasion during processing, easy processing, resistance to cracking and splitting, and better renewability and bio-degradability of the reinforcements (Bledzki and Gassan 1999; Mohanty *et al.* 2002; Klyosov 2007). Therefore, these composites are considered to be a desirable alternative to solid wood; they are widely used in indoor applications (*e.g.*, furniture and interior paneling, vinyl facings on doors, and consoles and seat backs in automobiles) in addition to outdoor applications (*e.g.*, deck floors, railings, fences, landscaping timbers, cladding and siding, park benches, and windows) (Ashori 2008; Beg and Pickering 2008; Thompson *et al.* 2009).

However, the polymer matrices of WPCs are based on linear backbones, such as polyethylene, polypropylene, and polyvinylchloride, which have either slightly polar or nonpolar interactions. The linear polymer chains are ready to move slowly but permanently from each other under the applied load, making WPCs inclined to creep, leading to obvious deformations or deflections produced in a WPC structure under long-term loads that may be much lower than its yield strength (Bodig and Jayne 1982). Creep will subject the WPC structure to excessive distortion, which will cause a sharp reduction in its expected service life (Lee *et al.* 2004). Therefore, thermoplastic WPCs are restricted

from being employed for structural support, *e.g.*, in beams, columns, window frames, and roofing systems, because of their low mechanical stability and creep.

It has been confirmed that coupling agents are able to improve the mechanical properties and the creep resistance of WPCs by improving the compatibility and interfacial adhesion between the matrix and the wood (Bengtsson and Oksman 2006; Acha *et al.* 2007). For instance, the most popular coupling agent, maleic anhydride-polypropylene (MAPP), can economically increase the tensile strength, flexural strength, and creep resistance of WPCs because it sufficiently improves the polymer-wood matrix compatibility *via* the strong secondary interactions of polymer chains between the polar wood and the nonpolar matrices (Bledzki and Faruk 2004; Lee *et al.* 2004; Acha *et al.* 2007; Beg and Pickering 2008; Nygard *et al.* 2008). If the coupling agent contains active groups that can chemically graft to wood, such as silane-grafted high-density polyethylene (HDPE), then the resulting WPC will have better mechanical properties and creep resistance than WPC made with the MAPP coupling agent (Bengtsson *et al.* 2006; Nachtigall *et al.* 2007).

It has also been observed that many composite properties decrease with decreasing wood fiber length (Beg and Pickering 2008). However, WPCs are still commonly manufactured using wood flour rather than wood fiber for extrusion molding due to ease and costs. Although wood flour exhibits better and easier dispersion within a viscous melt matrix, it has less reinforcing efficiency when compared to wood fibers due to the flour's smaller aspect ratio and interfacial area; this factor results in poor mechanical properties of WPCs reinforced with wood flour than with wood fiber (Nygard *et al.* 2008). Commonly, kraft fibers have larger length-diameter ratio more than 100. In principle, this will contribute to good reinforcing efficiency and, therefore, to better mechanical properties of kraft fiber reinforced polymer composites.

Based on the literature, measures were taken in current study to prepare a wood-plastic composite that can be a candidate for load-bearing materials. Kraft fibers were substituted for wood flour to improve reinforcing efficiency, and a thermoplastic matrix was replaced with a thermosetting matrix (unsaturated polyester, UPE) to effectively reduce creep. In addition, polyisocyanate was used as the coupling agent to improve the interfacial adhesion between wood and UPE, and the kraft fiber was pre-treated to improve the chemical interfacial adhesion between wood and polymer.

EXPERIMENTAL

Materials

Bleached kraft paper (70.2 g/m² grammage) prepared from pine and fir used in this research was a commercial product from a local paper mill. The paper was cut into 240 mm × 200 mm pieces prior to use. Commercial UPE resin 189# and the commercial initiator methyl ethyl ketone peroxide (MEKP) were provided by the Harbin Fiber-Reinforced-Plastic (FRP) Institute (China). The UPE resin was polymerized from phthalic anhydride, maleic anhydride, propylene glycol, and neopentyl glycol, which had a solid content of 62.8% and a hydroxyl value of 9.86 mg KOH/g. Regent grade styrene (> 98%) and sodium hydroxide (> 99%) were purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (China). Polymeric diphenylmethane diisocyanate (pMDI; Suprasec[®] 5005), had an isocyanate group content of 31.4 wt% (or functionality of 2.8), and was

obtained from Huntsman Corporation (Shanghai, China). All of these chemicals were used as received.

Pre-treatment of kraft papers by alkali

Each composite was compounded from 19 sheets of labeled kraft papers that were soaked in 3 to 15 wt% sodium hydroxide (NaOH) solution for 0 (control), 3, 6, 9, or 12 h for alkali pretreatment. After soaking, the papers were rinsed using distilled water until the rinsing solution had a pH value of less than 7.5; afterwards, the treated sheets were oven dried at 98 °C until the mass did not change.

Compounding and processing

The 189# UPE resin was diluted with styrene to a solid content of 60%. To this mixture was added 0.5 parts per hundred (phr) of the MEKP initiator. Before the UPE resin was spread onto the kraft paper, various quantities of pMDI (0, 5, 10, and 15 phr, relative to the diluted liquid UPE resin) were added as a crosslinking agent. A total of 19 oven-dried kraft paper sheets with dimensions of 240 mm × 200 mm were weighed (W_1 , g). A rubber roll was then used to spread the modified UPE resin onto the kraft paper. This was left for 5 min to allow the resin to impregnate the paper. All 19 resin-spread paper sheets were stacked and cold-pressed at 10 MPa for 1 min to form a composite plate while squeezing out any excess resin. The cold-pressed plate was then placed between two Teflon films and hot pressed at 125 °C under 15 MPa for 400 s. Finally, the composites were placed into an oven at 125 °C for 2 h for post-curing prior to being cut into specimens for characterization. Two replicate composites were prepared with each formulation.

Density and resin content determination

After trimming approximately 20 mm off the length and width of the composites, the length (a , mm), width (b , mm), thickness (c , mm), and weight (W_2 , g) of the composite samples were measured. The composite density (kg/m^3) was calculated as $10^6 \times W_2 / (a \times b \times c)$. The resin content (%) of the kraft fiber-reinforced UPE composites was obtained by dividing the resin mass in the composite ($W_2 - W_3$) by the weight of the trimmed composite (W_2), *i.e.*, $(W_2 - W_3) / W_2 \times 100$, where the resin mass in the composite was obtained by subtracting the total mass of 19 kraft paper sheets with the same area as the composite ($W_3 = [W_1 / (240 \times 200)] \times a \times b$) from the trimmed composite weight W_2 .

Accelerated hygrothermal aging of the composites

According to the Canada Standard CSA 0112.9 (2004), an accelerated hygrothermal aging process composed of five continual cycles of “boiling in water for 4 h then drying at 63 °C for 20 h” was carried out to evaluate the mechanical durability of the obtained composites. During each aging cycle, the dried specimens were placed into a stainless wire cage, then immersed into boiling water and kept there for 4 h; afterwards, the boiled specimens were immediately transferred from the boiling water, placed into a pre-heated oven (63 °C) and kept at 63 ± 2 °C for 20 h.

Tensile strength and modulus

Tensile tests for the composites were conducted in accordance with ASTM Standard D638 (2000) using a Universal Testing Machine (SANS Co., Shenzhen, China) with a strain rate of 100 mm/min. Type IV tensile specimens were cut from the

composites before and after accelerated hygrothermal aging, respectively, by means of a machining center with following dimensions: $W = 3.18$ mm, $L = 9.53$ mm, and $G = 7.62$ mm, where W is the width of the narrow section, L is the length of the narrow section, and G is the gage length, according to ASTM D638 (2000). The thickness of the composites was approximately 2 mm.

XRD analysis

The kraft papers after alkali pre-treatments with various soaking times were analyzed by X-ray diffraction (XRD) using a D/max-2200 diffractometer (Rigaku; Tokyo, Japan) at room temperature. The XRD radiation was generated from a $\text{Cu-K}\alpha$ ($\lambda = 0.154$ nm) source, and the diffraction data were collected from the 2θ values of 2.5° to 12° with a step size of 0.02, an accelerating voltage of 40 kV, and a current of 30 mA. The degree of crystallinity of kraft fiber under various pre-treating conditions was determined according to Sun *et al.* (2008).

SEM analysis

The composite specimen (with dimensions of 50 mm \times 10 mm \times 2 mm) was immersed in liquid nitrogen for 5 min for quick freezing (quenching) and then immediately broken by an impact force. A fractured surface was selected from the quenched sample for scanning electron microscopy (SEM) examination. The surface morphology of kraft paper after alkali pre-treatment was also examined by SEM. All SEM samples were sputter coated with approximately 10 to 20 nm of gold before examination with a QUANTA-200 scanning electron microscope (FEI Co., USA) with a working distance of 10 mm and an accelerating voltage of 12.5 kV.

RESULTS AND DISCUSSION

In a fiber-reinforced composite system, the improved stress transfer from resin to fibers resulting from the good interfacial adhesion will increase the mechanical properties of the composite. A kraft fiber-reinforced polyester composite generally has poor interfacial compatibility due to ill-matched intermolecular interactions between the polar fibers and the non-polar polymer matrix. In addition, the cellulose crystals are compact and smooth, which prevents the kraft fiber from being wetted and penetrated by the liquid resin polymer and further leads to poor interfacial resin-fiber adhesion. The combination of alkali pretreatment of kraft fibers and interfacial chemical crosslinking is expected to improve the interfacial adhesion and mechanical properties of the composite.

Typically, unbleached kraft fibers consist of 65 to 75% cellulose, 17 to 32% hemicellulose, and 3 to 8% lignin, compared to bleached pulp with 70 to 80% cellulose, 20 to 30% hemicellulose, and less 1% lignin (Beg and Pickering 2008). In the NaOH solution, sodium ions can penetrate into the amorphous areas of the cellulose and cause the cellulose to swell (Voronova *et al.* 2003). During the cellulose swelling, some components of the wood fiber, such as lignin and hemicellulose, may also be dissolved (Ku *et al.* 2011). As a result, after alkali pretreatment, kraft fibers may have a coarse surface due to the dissolution and swelling and change the crystalline structure of cellulose as well. However, alkali soaking of the kraft fibers at low a NaOH concentration (1 to 9 wt%) and at 30 °C for 3 h caused the degree of crystallinity to

slightly decrease indicating that little lignin and hemicellulose dissolved, as confirmed by the XRD results in Table 1.

Table 1. Degree of Crystallinity of Kraft Fibers after Various Alkali Pretreatment Conditions

NaOH concentration (wt%)	Treatment time (h)	Degree of crystallinity (%)
0 (control)	3	75.35(0.12)
3	3	74.92(0.27)
6	3	74.39(0.23)
9	3	73.65(0.28)
12	3	71.27(0.45)
15	3	67.63(0.82)
15	6	64.48(0.67)
15	9	59.88(0.93)
15	12	53.71(1.36)

When the NaOH concentration reached 15 wt% and the treating time increased, the degree of crystallinity of the kraft fibers apparently decreased. The fiber surfaces became very coarse due to the obvious dissolution of wood components and the destruction of the crystalline area of cellulose by the penetration of sodium ions, as shown by the XRD results in Table 1 and the SEM images in Fig. 1. The higher NaOH concentration and longer pretreatment time resulted in a lower degree of crystallinity and a coarser fiber surface.

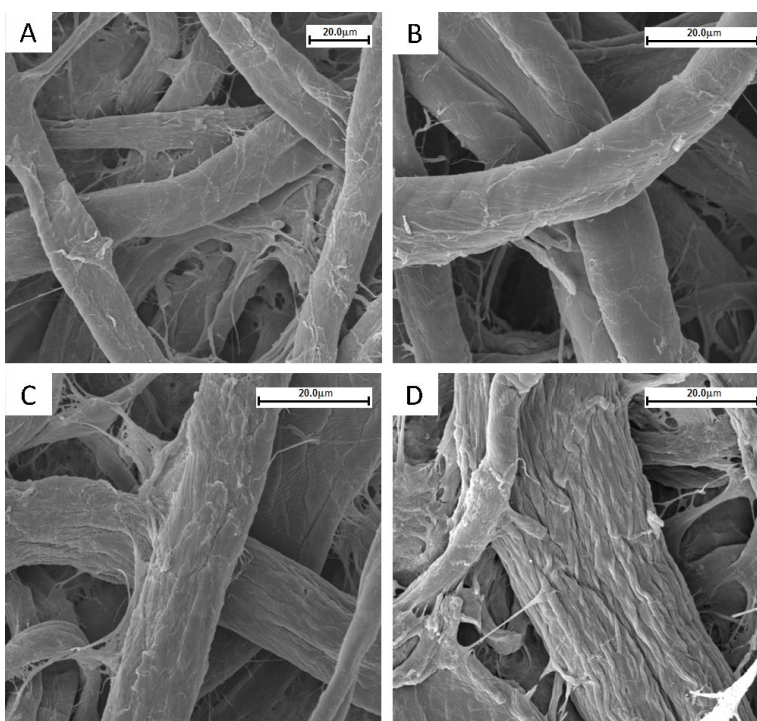


Fig. 1. SEM micrographs of alkali pretreated kraft fibers. (A) water only for 3 h (Control), (B) 6 wt% NaOH for 3 h, (C) 15 wt% NaOH for 3 h, and (D) 15 wt% NaOH for 12 h

The XRD analysis in Fig. 2 also indicates some new XRD peaks at 12.5° and 20.3° , which occurred after the kraft fibers were alkali-pretreated by 15 wt% NaOH solution for more than 9 h. This observation indicated that part of the cellulose changed from the crystalline mode of cellulose I into cellulose II. However, this transformation was not completely confirmed by the existence of the XRD peaks attributed to both cellulose I (14.8° , 16.6° , and 22.7°) and cellulose II (12.5° , 20.3° , and 23.1°). This transformation, called mercerization, is attributed to the dissolution of cellulose in NaOH solution and then the regeneration of the cellulose, during which the cellulosic chain rearranged intermolecularly to form cellulose II, which is more thermodynamically favored (Isogai and Atalla 1998; Ben-bassat *et al.* 1999).

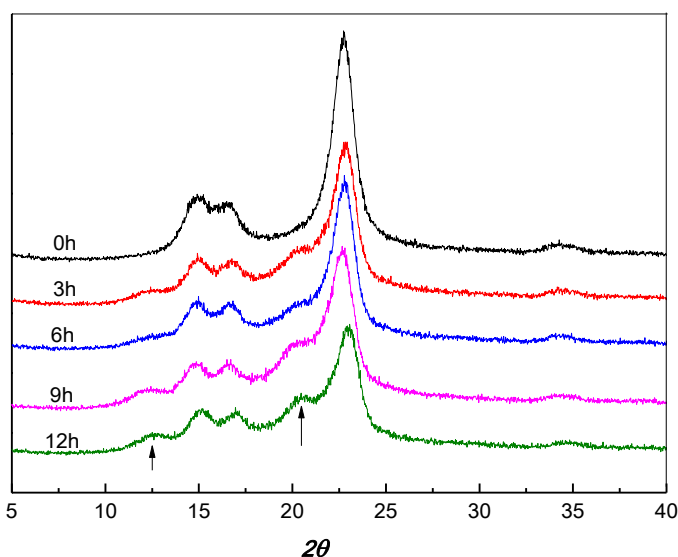


Fig. 2. XRD curves of kraft fibers after various alkali pretreating times

Test results shown in Table 2 indicate that alkali pretreatment of the fibers with 15 wt% NaOH solution had a negative effect on the tensile strength of the composites, showing an ever-decreasing tendency with an increasing alkali pretreatment time. This was attributed to the interior reinforcing efficiency of the alkali-treated kraft fiber due to the apparent decrease of cellulose crystallinity (as shown in Table 1). After the kraft fiber-reinforced polyester composite was subjected to hygrothermal aging, the composites' tensile strengths were significantly reduced, with a strength loss that ranged from 31.2% to 52.3%. This decrease could be caused from irreversible mechanical and physicochemical changes to the composites during hygrothermal aging, such as hydrolysis, chemical degradation, cracking, and/or interfacial debonding (Jedidi *et al.* 2006). However, the composites showed a slight increase of tensile strength or obvious decrease of tensile loss after hygrothermal aging with an increase in alkali treating time, which should be attributed to the improved interfacial adhesion after alkali treatment.

The coarse fiber surface (Fig. 1) could increase the effective fiber surface area available for contact with the polyester matrix, and hence the interfacial adhesion was improved, which is in accordance with many reported studies (Ku *et al.* 2011). In addition, the decreased degree of crystallinity (Table 1) meant that there was an increase of amorphous cellulose in the alkali-treated kraft fiber, which could allow the liquid unsaturated polyester resin to better penetrate the fiber, thus forming mechanical

interlocks between the cured polyester and the kraft fiber to further strengthen the interfacial adhesion.

The higher resin content of the composite with longer alkali pretreatment time in Table 2 confirmed that more liquid unsaturated polyester resin penetrated into the kraft fiber with the higher amorphous cellulose content.

Table 2. Physical and Mechanical Properties of Composites with Kraft Fibers Pretreated with Alkali at Various Times

Composite ID	Pretreatment time (h)	Density (kg/m ³)	Resin content (%)	Tensile strength ^{a*} (MPa)	Tensile strength ^{b*} (MPa)	Strength loss (%)
C0	0	1414	23.63	121.17 (3.36)	57.80 (2.72)	52.30
C1	3	1408	24.16	120.07 (3.84)	59.50 (3.10)	50.45
C2	6	1401	25.73	118.62 (4.16)	64.63 (3.57)	45.52
C3	9	1392	26.63	108.76 (2.72)	68.13 (3.29)	37.36
C4	12	1395	28.55	96.76 (2.02)	66.53 (2.34)	31.24

Note: ^{a*} and ^{b*} refer to the tensile strength before and after hygrothermal aging, respectively; all values in parentheses are standard deviations; the concentration of NaOH solution used was 15wt%.

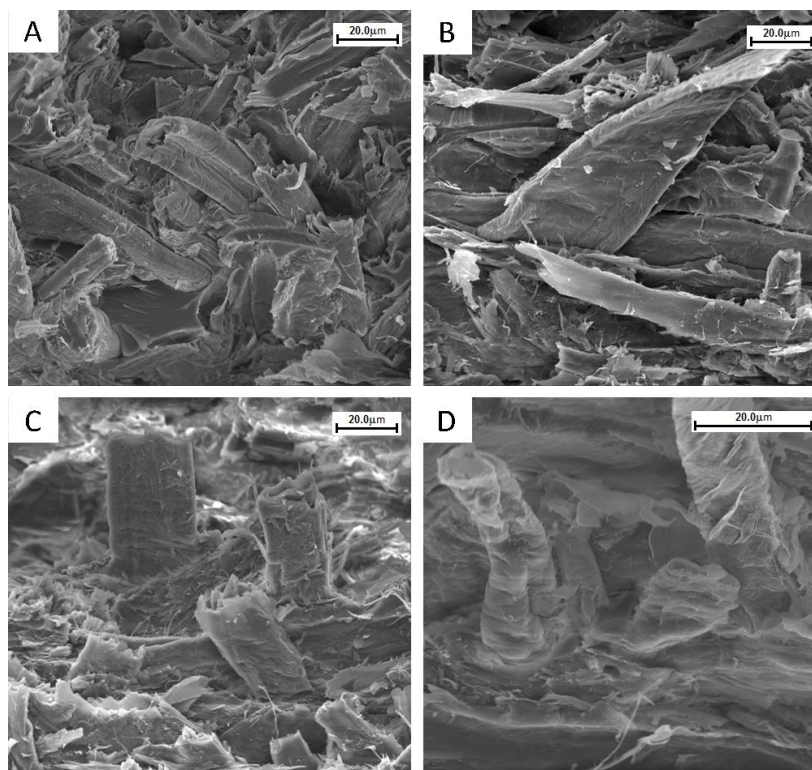


Fig. 3. SEM micrographs of the kraft fiber/unsaturated polyester composite. (A) and (B) are composite C4 before and after hygrothermal aging, respectively and (C) and (D) are composite C6 before and after hygrothermal aging, respectively.

The SEM micrograph in Fig. 3A showed some small cracks between the fiber and the cured polyester, which indicated lower interfacial adhesion due to the lack of compatibility between the polar hydrophilic kraft fibers with the nonpolar hydrophobic unsaturated polyester matrix. After the hygrothermal aging, the interfacial cracks and the debonding increased, as shown in Fig. 3B, resulting in an obvious decrease of tensile strength (from a range of 96.8 to 121.2 MPa to a range of 57.8 to 68.1 MPa (Table 2)). This implies that these composites had poor mechanical stability during their service lifetime after the effect by water or moisture during hygrothermal aging.

In an effort to overcome the problems just mentioned, pMDI was introduced to attempt to improve the interfacial adhesion or the mechanical stability of the composite *via* the formation of chemical bridges between the cured unsaturated polyester and the kraft fibers. The unsaturated polyester resin used in the current study had a hydroxyl value of 9.87 mg KOH/g (equivalent to 0.0176 moles of hydroxyl groups per 100 g liquid resin). Every molecule of the polyisocyanate pMDI has 2.8 isocyanate groups (on average), which can react not only with residual hydroxyl groups in unsaturated polyester resin but also with the hydroxyl groups of the kraft fibers to form a covalent bond, as illustrated in Fig. 4.

Table 3. Mechanical Properties of Composites with Polyisocyanate Crosslinking Agent

Composite ID	Polyisocyanate level (%)	Density (kg/m ³)	Resin content (%)	Tensile strength ^{a*} (MPa)	Tensile strength ^{b*} (MPa)	Strength loss (%)
C3	0	1392	26.63	108.76 (2.72)	68.13 (3.29)	37.36
C5	5	1405	27.22	142.15 (5.63)	91.52 (5.57)	35.62
C6	10	1428	27.09	153.43 (5.89)	98.02 (6.72)	36.11
C7	15	1475	27.81	155.21 (5.71)	101.16 (6.89)	34.82

Note: ^{a*} and ^{b*} refer to the tensile strength before and after hygrothermal aging, respectively. All kraft fibers were alkali-treated with 15 wt% NaOH for 9 h. All values in parentheses are standard deviations

As expected, the tensile strengths of the composite crosslinked with polyisocyanate were obviously improved when compared to the composites without modification, as shown in Table 3. In particular, the composite modified with 15% polyisocyanate had a very high tensile strength (101.16 MPa) after hygrothermal aging, which was 52.1% more than that of the control C3 after aging (66.53 MPa) and almost comparable to the strength of the control C3 prior to aging (108.76 MPa). This comparison confirmed the formation of chemical bridges between the cured polyester matrix and the kraft fibers, which was further evidenced by the improved interfacial adhesion before and after hygrothermal aging, as shown by the SEM micrographs in Figs. 3C and 3D. The interface between the polyester matrix and the kraft fiber was integrated without apparent cracks or debonding, even after undergoing six cycles of boiling-dry treatment; this indicated an improved interfacial stability in the composite, which resulted in good mechanical stability.

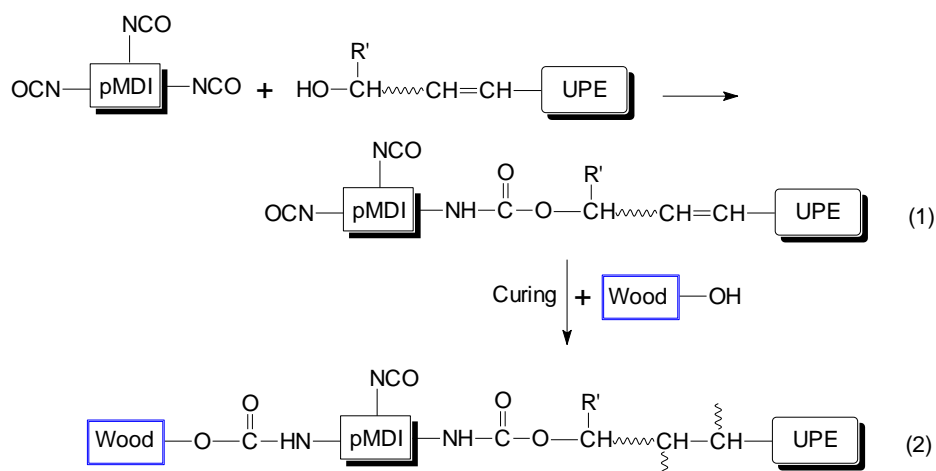


Fig. 4. Crosslinking scheme between polyester and wood fiber by polyisocyanate

However, it is interesting to note that there was an apparent increase in tensile strength before aging (30.7%) or after aging (37.6%) when the polyisocyanate level increased from 0% to 5%; a further increase in the amount of polyisocyanate from 5% to 15% only resulted in a 9.2% to 10.53% increase of the tensile strength. Therefore, the addition of 5% polyisocyanate as a crosslinking agent could result in a satisfactory increase in the tensile properties, whereas a further increase of polyisocyanate content did not bring about any improvements. This may be attributed to a saturation of the crosslinking efficiency of the polyisocyanate with the kraft fiber-polyester interfaces (Gao *et al.* 2013). Considering the higher cost of polyisocyanate crosslinker (about US\$2.90/kg) when compared to the unsaturated polyester resin (about US\$1.94/kg), the optimal polyisocyanate level as a crosslinking agent was determined to be 5%, which yields an acceptable mechanical improvement while maintaining a low composite cost.

CONCLUSIONS

1. Kraft fiber-reinforced unsaturated polyester composites with improved mechanical properties and stability were prepared *via* the fiber alkali pre-treatment and polyisocyanate crosslinking modification.
2. Polyisocyanate as a crosslinking agent can effectively increase the mechanical properties and stability of the composite due to the formation of covalent bonds between the kraft fibers and the polyester matrix.
3. Alkali pretreatment of the fibers yielded a negative effect on the mechanical properties of the composite (before hygrothermal aging) due to the apparent decrease of degree in cellulose crystallinity; however, the pretreatment slightly improved the mechanical properties after hygrothermal aging due to the improved interfacial adhesion between the fibers and the polymer matrix.

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