High pressure-assisted magnesium carbonate impregnated natural fiber-reinforced composites

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Abstract

The kenaf fiber/polyester composites, owning the comparable mechanical properties with commercial glass-fiber sheet molding compounds (SMC) for automotive application, were achieved through the high pressure-assisted magnesium carbonate impregnated fibers and the vacuum-assisted resin transfer molding process. The in situ inorganic nanoparticle impregnation (INI) process was performed by immersing the alkali retted fibers into magnesium chloride solution with various pressures and then treated by sodium carbonate to yield the INI-treated fibers. The magnesium carbonate content was increased by 180.3%, when a 13.79 MPa pressure was applied during the magnesium carbonate immersing process, resulting in lower porosity of fibers. Compared to the un-treated fiber/polyester composite, the mechanical properties of the high pressure-assisted INI-treated fiber/polyester composites were enhanced by 18.6–277.3%, and the 24-hour thickness swelling and water absorption were reduced by 64.4% and 62.2%, respectively. The mechanical properties and specific mechanical properties were comparable with or exceeded to that of glass-fiber SMC.

Keywords: Natural fiber; Magnesium carbonate; Composites; Porosity; Mechanical properties

1. Introduction

Driven by the ecological property, the interest has recently arisen in using natural fibers to replace synthetic fibers for fabricating fiber reinforced polymer composites (Bledzki et al., 1996; George et al., 2001; Jindal, 1986; Joshi et al., 2004; Kalia et al., 2009; Saheb and Jog, 1999). Because of the low density and outstanding mechanical properties, these composites are potential to serves as the next-generation of high-performance materials for the automotive applications (Marsh, 2003). As a type of abundant lignocellulosic fibers, based on the report of Food and Agriculture Organization of the United Nations (FAO), kenaf (*Hibiscus cannabinus* L.) and allied fibers are estimated the average yield of 288,000 tons/year over 2007–2012. Being known as the excellent mechanical properties, kenaf fibers have tensile strength comparable with that of glass fibers (Akil et al., 2011; Roger et al., 1999). Due to their excellent characteristics of low density, low cost, and availability, kenaf composites have attracted great attentions (Feng et al., 2002; Liang et al., 2013; Xia et al., 2016a; Xia et al., 2016b; Zampaloni et

al., 2007). In addition, some chemical methods, such as pre-treating kenaf fibers by adding CaCO_3 or $\text{Al}(\text{OH})_3$ nanoparticles, for improving the mechanical properties of kenaf composites were explored (Anuar and Zuraida, 2011; Xia et al., 2015c; Xia et al., 2016c).

There are some concerns about the production of natural fiber reinforced polymer composites, e.g. the matrix-fiber interfacial adhesion, fiber dispersion, and porosity of the fibers (Bledzki et al., 2005). Different types of fiber surface treatment procedures have been suggested to enhance the interaction between natural fibers and matrix. The alkali treatment is the easiest surface treatment technique for natural fibers among the reported treatment types (Gassan and Bledzki, 1999; Mohanty et al., 2000; Ray et al., 2002). It was reported that the alkali treatment increased the fiber adhesion with matrixes due to the fiber surface modification. Fiore et al. confirmed that the alkaline treatment improved mechanical properties of kenaf fibers and their epoxy composites (Fiore et al., 2015). Another promising surface treatment method is the inorganic nanoparticle impregnation (INI) treatment, which is an effective method to improve the compatibility between natural fibers and polymer matrix (Shi et al., 2011b; Xia et al., 2015c).

In the previous studies, calcium carbonate was used to increase the mechanical properties for kenaf fibers, as well as kenaf fiber/polymer composites (Liang et al., 2014). It can also be used as an adhesive between the kenaf fibers and polymer matrix, as well as the fillers filling the fiber voids. The tensile strength of the kenaf fibers was increased by more than 20% after the INI treatments. The INI treatments improved the compatibility between kenaf fibers and polypropylene matrix, resulting in the increased tensile modulus and strength of the composites reinforced with INI-treated fibers. The compatibility between bamboo fibers and polypropylene matrix materials was increased by the calcium carbonate treatment of the filled bamboo fibers was also reported (Cheng et al., 2014b). The tensile strength and modulus of composites reinforced with treated fibers increased by 14.58 and 19.66%, respectively. As the loading of calcium carbonate increased, an increased trend of the panel tensile strength and modulus was also observed. However, the pressures applied for increasing the calcium carbonate loading efficiency were caused by the internal pressures that were produced due to the high heating temperatures.

In the regular INI-treatment, the pressures were created by the heating temperature because no external pressure was introduced to the reactor, which limited the use of high pressures and high calcium carbonate loading amount (Cheng et al., 2014a; Xia et al., 2015c). For instance, the saturated water vapor pressure is only 0.60 MPa at 160°C (Shi et al., 2011a), which may not be high enough to produce a sufficient impregnation pressure for the nanoparticles. In order to improve composite properties by increasing the nanoparticle loading amount, the nanoparticles need to penetrate into fiber cell walls and increase the loading amount of inorganic particles. Since the pressure in the reactor played an important role in the INI process, external pressures were introduced in the impregnating process to improve the nanoparticle loading efficiency.

The pressured treatment has been proved to be the most effective method to force preservatives into wood cells for wood protection (Loferski, 2001). The amount of chemicals retained by the wood determines the preservation results and is regulated by the manufacturer's treating schedule (i.e. pressure and time). As a successful method used in the wood preservation practice, the external pressures were introduced in the loading of calcium carbonate nanoparticles for natural fibers in our

previous study (Xia et al., 2015b). Although the natural fiber composite has its unique advantage in environmental aspects, compared to glass-fiber or carbon-fiber composites, the drawback of relative low mechanical properties still limits its commercialization. Since the magnesium carbonate owns better mechanical properties than that of the calcium carbonate as the inorganic fillers (Edwards et al., 2005). It was expected that the impregnating of magnesium carbonate to kenaf fibers would enhance the mechanical properties of the composites.

This study was aimed at improving the mechanical properties of kenaf fiber/polyester composites, which were comparable with that of the commercial automotive glass-fiber sheet molding compound (SMC), by pressure-impregnating magnesium carbonate into kenaf fibers. During the INI-treatment, the effect of the applied pressure on the loading amount of magnesium carbonate and the resulted composite properties was investigated. Furthermore, a property comparison between the kenaf composites and glass-fiber SMC was carried out.

2. Materials and Methods

Kenaf bast with a length of approximately 50 mm was sourced from Kengro Corp., USA. The sodium hydroxide (NaOH) solution (5%, w/v) was prepared using NaOH beads ($\geq 97\%$, Acros Organics) and deionized (DI) water from Millipore Milli-Q Integral Water Purification System. The magnesium chloride hexahydrates ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 99.0-102.0%, Sigma-Aldrich) and sodium carbonates (Na_2CO_3 , $\geq 99.5\%$, Fisher Scientific) were used. The unsaturated polyester (AROPOL Q 6585, 30% styrene, Ashland Chemicals) and tert-butyl peroxybenzoate (t-BP, 98%, Acros Organics) were used for fabricating the composites.

2.1. MgCO_3 loading through INI processes

Three steps were taken place for loading MgCO_3 particles into kenaf fiber.

- 1) A mixture of 120 g kenaf bast fibers (moisture content of 9.1%, measured by Mettler-Toledo HB43-S Moisture Analyzer) and 1.8 L NaOH solution were added into a 2-litre hermetical reactor (251 M, Parr Instrument Company, USA). This alkali retting process was carried out at 160°C for one hour with the mechanical stirring. The saturated vapor pressure remained at 0.60 MPa in the hermetical reactor. After cooling, the retted fibers were washed using running water and then dried. The fiber yield was measured as $38.5 \pm 1.0\%$.
- 2) After being washed, but not dried, the retted fibers and 1.8 L MgCl_2 aqueous solution (0.1 mol L^{-1}) were added into the hermetical reactor. The reactor was then heated to 100°C and maintained for 0.5 hour. During the process, argon gas was injected into the reactor through a high-pressure cylinder to obtain pressures of 3.45 MPa (500 psi), 6.89 MPa (1,000 psi) and 13.79 MPa (2,000 psi). The saturated vapor pressure was 0.10 MPa without external pressure applied. After cooling, the excessive ionic solution was removed from kenaf fibers first by gravity, and then by hand-squeezing.
- 3) The retted fibers obtained from Step 2 were mixed into the 1.8 L Na_2CO_3 aqueous solution (0.1 mol L^{-1}) in the hermetical reactor. After the reaction at 100°C for 0.5 hour, the mixture was cooled down to room temperature, filtered, washed with running water, and then dried. The INI-treated fiber was called INI (pressure value) treated fiber, e.g. INI (13.79 MPa) treated fiber.

2.2. Determination of $MgCO_3$ contents

The $MgCO_3$ contents were calculated based on the difference in ash contents between the retted fibers and $MgCO_3$ impregnated fibers. The ash contents were determined by burning the samples in a muffle furnace at 675°C for 6 hours. From the thermogravimetric analysis (TGA data) in the literature (Paswan et al., 2013), it showed that $MgCO_3$ was decomposed into MgO when the temperature exceeded 675°C. The following reaction happened during the burning:

The $MgCO_3$ content was calculated by the approximate equation:

$$MgCO_3 \text{ content} = (Ash_{INI-fiber} - Ash_{fiber}) \times \frac{M_{MgCO_3}}{M_{MgO}} \times 100\% \quad (2)$$

where $Ash_{INI-fiber}$ and Ash_{fiber} were the ash contents of INI-treated fiber and un-treated fiber, respectively; M_{MgCO_3} and M_{MgO} were the molecular weights of $MgCO_3$ (84.3 g/mol) and MgO (40.3 g/mol), respectively.

2.3. Porosity determination

The porosity of un-treated fiber, INI (0.10 MPa) treated fiber, and INI (13.79 MPa) treated fiber were analyzed by mercury porosimetry. A mercury porosimeter (AutoPore IV 9500, Micromeritics Instrument Corp., USA) was employed. The procedure for the porosity measurement was described as follows: The samples were dried overnight at 105°C before measurements. The penetrometer for powdered samples was used, which had 3 cm³ capacity and 0.412 cm³ steam volume. The median pore diameter was calculated based on volume. The average pore diameter was calculated by:

$$D_m = \frac{4V_{tot}}{A_{tot}} \quad (3)$$

where V_{tot} is the total specific intrusion volume and A_{tot} is the total cumulative area.

2.4. Composites fabrication

The composites were fabricated using un-treated and INI-treated fibers though vacuum-assisted resin transfer molding (VARTM) process described in the previous research (Xia et al., 2015a). **Five replicates were used for each condition.** The unsaturated polyester resin (~150 g) with 1.5% of t-BP catalyst was used to fabricate the fiber/polymer composites. Briefly, the kenaf preform (~38 g), with a dimension of 25 × 25 × 10 mm (width × length × thickness), was placed on the mold and a flexible polyethylene vacuum bag was placed over the mold. The resin was infused by a vacuum pump (Vacmobile 20/2 System with Becker U4.20) with a pressure of 1.3–1.6 kPa. The vacuum force pulled the resin along the distribution layer into the fiber reinforcement preform. The resin-infused preform was cured at 100 °C for 2 h, and then 150 °C for 2 h, in the hot press with a pressure of 13 MPa. **During the hot-pressing process, the extra resin was squeezed out by the pressure.** Once the fabricated composite cooled down to room temperature, the vacuum bag and distribution layer were removed. **By weighing the resulted composite, the fiber and resin contents were estimated to be approximately 65% and 35%, respectively (The detailed information presented in Section 3.2).**

2.5. Mechanical property tests

From each composite, twelve specimens sized $25 \times 160 \times 3$ mm (width $\times$ length $\times$ thickness) were cut by a 9-in Skil benchtop band saw (Robert Bosch Tool Corp., USA), for examining the modulus of elasticity (MOE), modulus of rupture (MOR), and bending elongation at break (BEB) of the composites using a Shimadzu AGS-X universal testing machine in accordance with the procedure described in ASTM D790 standard. Three-point bending set-up was used with a span of 50 mm and a crosshead speed of 1.3 mm min^{-1} . The MOE, MOR and BEB were calculated by Eqs. (4), (5), and (6), respectively.

$$\text{MOE} = \frac{L^3 m}{4bd^3} \quad (4)$$

$$\text{MOR} = \frac{3PL}{2bd^2} \quad (5)$$

$$\text{BEB} = \frac{6Dd}{L^2} \quad (6)$$

where “L” is support span; “m” is slope of the tangent to the initial straight-line portion of the load-deflection curve; “b” is width of beam tested; “d” is depth; “P” is load at a given point on the load-deflection curve.

The tensile modulus (TM), tensile strength (TS), and tensile elongation at break (TEB) of the specimens were tested in accordance with the procedures described in ASTM D638 standard by the Shimadzu AGS-X tester with an axial extensometer (Epsilon Technology Corp., USA) with a 50.8 mm (2 inch) gauge length. The specimen dimensions were $19 \times 165 \times 3$ mm (width $\times$ length $\times$ thickness) and the cross section of the narrow section was 13×57 mm (width $\times$ length). A crosshead speed of 5 mm min^{-1} was used.

2.6. Microtopography analysis

The surfaces of un-treated fiber and INI (13.79 MPa) treated fiber were observed by a Quanta 200 environmental scanning electron microscope (SEM) with an accelerating voltage of 20 kV and a magnification of 2,000 $\times$. The size distributions of MgCO_3 particles on the surfaces of INI (13.79 MPa) treated fiber were analyzed using the software Digimizer with a counting of 150 data. Also, the fracture failure mode of the tensile specimens, including the composites of un-treated fiber/polyester and INI (13.79 MPa) treated fiber/polyester, were observed by SEM with a magnification of 500 $\times$.

2.7. Water-resistant tests

The composites made with un-treated and INI-treated fibers were used for the thickness swelling and water absorption measurements. The specimens with a dimension of $25 \times 25 \times 3$ mm (width $\times$ length $\times$ thickness) were cut using a 9-in Skil benchtop band saw. Five replicates were tested for each composite. Twenty four-hour water submersion tests were carried out in accordance with the ASTM D1037 standard for the determination of the thickness swelling and water absorption.

3. Results and Discussion

3.1. INI-treated fibers

The results of MgCO_3 loading at four different pressures during the INI process are shown in Table 1. With the increase of applied pressure, the impregnated MgCO_3 contents increased. When pressures of 3.45 MPa, 6.89 MPa and 13.79 MPa were applied to the reactor, the MgCO_3 contents increased to be 1.27%, 1.76% and 2.70%, which were 32.1%, 82.4% and 180.3% increases compared to that without external pressures, respectively. The results showed that the higher the applied pressure, the larger the impregnated MgCO_3 content. The high pressure may help the impregnated chemicals penetrating into the porous structure of the cells, resulting in an increase in MgCO_3 content.

Table 1. The effect of immersing pressures on the MgCO_3 loading.

Sample	Immersing pressure (MPa)	MgCO_3 content (%)	Increase ¹ (%)
INI (0.10 MPa) treated fiber	0.10	0.96	-
INI (3.45 MPa) treated fiber	3.45	1.27	32.1
INI (6.89 MPa) treated fiber	6.89	1.76	82.4
INI (13.79 MPa) treated fiber	13.79	2.70	180.3

Note: ¹ The increases were calculated based on the MgCO_3 content of INI (0.10 MPa) treated fiber without external pressure.

To examine the effect of the pressure on the INI process, the porosities of three different fibers, including un-treated fiber, INI (0.10 MPa) treated fiber without external pressure and INI (13.79 MPa) treated fiber with high pressure, were measured by the mercury porosimetry (Table 2). The INI (0.10 MPa) treated fiber owned 15.3% porosity, which was reduced by 31.5% compared with the un-treated fiber of 22.3% porosity. While, the porosity of INI (13.79 MPa) treated fiber reached as low as 10.6%, which was a reduction of 52.4% from the un-treated fiber. The results showed that the high pressure treatment resulting in larger MgCO_3 loading amount would accelerate the reduction of fiber porosity. Simultaneously, the median and average pore diameters were decreased (Table 2) from 21.7 μm to 13.5 μm and 10.6 μm , and from 10.0 μm to 6.7 μm and 5.5 μm , respectively. Fig. 1 shows the log differential mercury intrusion ($\text{mL/g}/\mu\text{m}$) as a function of pore diameter (μm). It was suggested that the pore sizes of fibers were reduced after the INI-treatment. The smallest pore reduced from approximate 0.3 μm to 0.2 μm . Fig. 1 also reveals that the MgCO_3 particles are mainly targeting to the pores with a diameter ranging from approximate 0.2 to 7 μm .

Table 2. Mercury intrusion porosimetry results of fibers.

Fiber	Porosity (%)	Median pore diameter (μm)	Average pore diameter (μm)
Un-treated fiber	22.3	21.7	10.0
INI (0.10 MPa) treated fiber	15.3	13.5	6.7
INI (13.79 MPa) treated fiber	10.6	10.6	5.5

Fig. 1. Pore size distributions of fibers measured by mercury intrusion porosimetry.

The micro topographies of un-treated and INI (13.79 MPa) treated fibers are shown in Fig. 2a-b. By comparing Fig. 2a (un-treated fiber) with Fig. 2b (INI (13.79 MPa) treated fiber), the MgCO_3 particles on the fiber surface were observable in Fig. 2b. The diameter of particle sizes on the INI (13.79 MPa) treated fibers were measured to be an average of 520 nm and a standard deviation of 179 nm. The probability distribution of the particle sizes was presented as a log-normal distribution with a R^2 (coefficient of determination) of 0.96 (Fig. 3). The median particle size was 490 nm with a standard error of 13 nm from the log-normal fitting.

Fig. 2. SEM examination of the fiber surfaces of un-treated fiber (a) and INI (13.79 MPa) treated fiber (b), and the tensile fracture surfaces of un-treated fiber (a) and INI (13.79 MPa) treated fiber (b) reinforced composites.

Fig. 3. Particle size distribution of MgCO₃ on the surface of INI (13.79 MPa) treated fiber.

3.2. Composite properties

The physical properties of the composites are summarized in Table 3. The results showed that the resin and fiber contents of the composites were 31.6–36.5% and 62.4–67.7%, respectively. After applying the INI-treatment to the fibers, the density of the composites, measured in accordance with the ASTM D792 standard, were increased approximately 10%, from 1,159.4 Kg m⁻³ to 1,265.4–1,318.0 Kg m⁻³. The MgCO₃ contents were increased from 0.7% to 1.8% when the pressure was increased from 0.10 MPa to 13.79 MPa during the INI-treatments.

Table 3. Physical and water-resistant properties of the composites.

Composite	Density (Kg m ⁻³)	Components (wt%)			Thickness swelling (%)	Water absorption (%)
		Resin	Fiber	MgCO ₃		
Un-treated fiber/polyester	1,159.4 (44.5)	33.3	66.7	-	19.7 (3.8)	18.3 (2.3)
INI (0.10 MPa) treated fiber/polyester	1,265.4 (20.2)	31.6	67.7	0.7	12.3 (1.5)	11.9 (0.5)
INI (3.45 MPa) treated fiber/polyester	1,316.3 (33.4)	34.1	65.0	0.8	10.0 (1.7)	8.7 (0.9)
INI (6.89 MPa) treated fiber/polyester	1,301.6 (15.2)	36.5	62.4	1.1	8.6 (0.3)	8.6 (0.3)
INI (13.79 MPa) treated fiber/polyester	1,318.0 (13.1)	35.1	63.2	1.8	7.0 (1.7)	6.9 (0.4)

The mechanical properties of the composites are presented in Table 4 as comparison. In general, all the mechanical properties from bending and tensile tests were dramatically improved after employing the INI-treated fibers instead of the un-treated fibers for the composite fabrication. The mechanical properties from bending tests, including MOE, MOR and BEB, were increased by 51.7–64.0%, 85.1–147.4%, and 70.1–103.7%, respectively. The results from tensile tests, including **TM**, TS and TEB, showed the increases of 6.8–25.2%, 36.6–93.1%, and 30.6–277.3%, respectively. The SEM images of tensile fracture surfaces of un-treated fiber/polyester and INI (13.79 MPa) treated fiber/polyester composites are presented in Fig. 2c-d. It was obviously showed a number of fiber bundles, long fibers, and the deep holes after fibers being pullout (Fig. 2c) on the composites of un-treated fiber/polyester. However, almost no fiber bundles, long fibers, and the deep holes on the fracture surfaces of the INI (13.79 MPa) treated fiber/polyester composite (Fig. 2d). These fiber pullouts were much shorter when the INI-treated fibers were employed, which could illustrate the improved interfacial bonding between fibers and matrix. The long pullout fibers indicated that the fibers did not well adhere to the resin matrix in the un-treated fiber/polyester composites. As a result, the mechanical properties were reduced. This phenomenon was also reported in the previous research for calcium carbonate-INI-treated fiber/PP composites (Shi et al., 2011b), and calcium carbonate-INI-treated fiber/polyester composites (Xia et al., 2015c). Furthermore, almost no fiber bundle was observed in these images. It was indicated that the interfacial compatibility between fibers and matrix was highly improved. The higher interfacial compatibility resulted in a stronger adhesion between the fibers and matrix, contributing to the improved mechanical properties.

267 **Table 4.** Mechanical properties of different composites.

Composite	MOE ² (GPa)	MOR ³ (MPa)	BEB ⁴ (%)	TE ⁵ (GPa)	TS ⁶ (MPa)	TEB ⁷ (%)
Un-treated fiber/polyester	6.9 (0.6)	68.3 (6.3)	1.1 (0.3)	10.7 (1.0)	44.5 (3.7)	1.1 (0.5)
INI (0.10 MPa) treated fiber/polyester	10.7 (1.0)	126.4 (12.7)	1.9 (0.7)	11.4 (1.2)	60.9 (2.3)	1.5 (0.2)
INI (3.45 MPa) treated fiber/polyester	11.4 (0.5)	168.9 (7.6)	2.2 (0.2)	13.2 (0.2)	72.1 (2.5)	2.8 (0.2)
INI (6.89 MPa) treated fiber/polyester	10.8 (0.3)	164.9 (4.9)	2.2 (0.1)	13.4 (0.4)	80.1 (0.5)	3.3 (0.1)
INI (13.79 MPa) treated fiber/polyester	10.5 (0.7)	151.9 (6.2)	2.2 (0.2)	12.7 (0.2)	86.0 (1.4)	4.3 (0.1)
Increment ¹ (%)	51.7%	122.4%	102.9%	18.6%	93.1%	277.3%

268 *Notes:* ¹ The increments were calculated based on the mechanical properties of un-treated fiber/polyester and INI (13.79
269 MPa) treated fiber/polyester composites; ² MOE = modulus of elasticity; ³ MOR = modulus of rupture; ⁴ BEB = bending
270 elongation at break; ⁵ **TM** = tensile modulus; ⁶ TS = tensile strength; ⁷ TEB = tensile elongation at break.

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272 To evaluate the contribution of pressure-treatments on the improvements of mechanical property,
273 the comparisons between INI (0.10 MPa) treated fiber/polyester and INI (3.45, 6.89 and 13.79 MPa)
274 treated fiber/polyester composites were conducted. After the pressure-treatments, MOE remained almost
275 unchanged; BEB and **TM** were slightly increased (17.3–19.3% and 11.0–17.2%, respectively); MOR,
276 TS, and TEB were significantly (Anova, $\alpha = 0.01$) improved by 20.2–33.6%, 18.5–41.4%, and 90.3–
277 188.8%, respectively. In summary, the mechanical properties were improved after introducing an
278 external pressure during the INI-treatments. Especially, the TEB was increased by 188.8% from 1.5% to
279 4.3%, indicating that the capability of the composite for resisting shape change without crack formation
280 was greatly improved.

281 Water-resistant properties of the composites are shown in Table 3. The results showed that the INI-
282 treatment dramatically reduced the thickness swelling and water absorption. Compared with the un-
283 treated fiber/polyester, the thickness swelling and water absorption of INI (0.10 MPa) treated
284 fiber/polyester were significantly (Anova, $\alpha = 0.01$) reduced by 37.5% and 35.1%, respectively. After
285 13.79 MPa external pressure being introduced into the INI-treatments, the thickness swelling and water
286 absorption were further reduced, and the reductions could be 64.4% and 62.2%, respectively. This could
287 be the major reason contributing to the dramatic improvement of the water resistance of the composites.
288 Owing to the hydrogen bonding between MgCO_3 and cellulosic fibers, MgCO_3 adhered to the fiber
289 surfaces and blocked the large amount of hydroxyl groups. The MgCO_3 dramatically reduced the
290 contacts between the water and fibers, resulting in better water resistance. The phenomena, that particles
291 on the fibers can block the hydroxyl groups so that improving the water-resistance, also could be found
292 when calcium carbonate particles were used (Hu et al., 2009; Xia et al., 2015c).

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3.3. The comparison with glass-fiber SMC

Glass-fiber sheet molding compound (SMC) is widely used in the automotive manufacturing. However, it is difficult to be recycled owing to its physiochemical composition, which will cause environmental pollution. Since kenaf fiber is lighter and available at a lower cost than glass fiber, the increasing attention is being devoted to the application. Based on the life cycle assessment (LCA) study (Wang et al., 2013), less negative environmental impacts were found in “cradle to gate” consideration of kenaf fibers compared to glass fibers. Additionally, from the viewpoint of energy consumption, kenaf fiber takes 15 MJ of energy to produce 1 kg of kenaf fiber compared to 54 MJ to produce 1 kg of glass fiber (Akil et al., 2011). Based on the treatment time and temperature, it is roughly estimated that less than 15% of energy consumption could be increased for the treatment of kenaf fibers. Even though the increase in energy consumption ($15 \text{ MJ} \times (1 + 15\%) = 17.25 \text{ MJ}$), it is still much lower than that for glass fiber.

Commercial glass-fiber SMC (SLI 323IF 2.5 mm, Meridian Automotive Systems Inc., USA) for the automotive application was used for the comparison. The glass-fiber SMC samples containing three components (from the company data sheet, in wt.%, 26.0% polyester resin, 28.0% glass fiber, and 46.0% mineral (mainly in CaCO_3)) were measured to be an average density of 1801.8 Kg m^{-3} with a standard deviation of 27.1. The density of INI (13.79 MPa) treated fiber/polyester composite was 26.9% lower than that of the glass-fiber SMC, which offers the benefit of reduced automotive weight. The resin contents of the INI (13.79 MPa) treated fiber/polyester composite and glass-fiber SMC were comparable (26.0% of glass-fiber SMC vs. 35.1% of INI (13.79 MPa) treated fiber/polyester composite). The fiber content of INI (13.79 MPa) treated fiber/polyester composite (63.2%) was much higher than that of glass fiber SMC (28.0%) owing to the lower content of filling minerals (1.8% of MgCO_3), while glass fiber SMC contained 46.0% minerals.

The mechanical properties of glass-fiber SMC were analyzed, including MOE ($10.2 \pm 0.1 \text{ GPa}$, mean $\pm$ standard deviation), MOR ($138.3 \pm 9.2 \text{ MPa}$), BEB ($2.5 \pm 0.5\%$), **TM** ($13.7 \pm 0.1 \text{ GPa}$), TS ($85.6 \pm 16.4 \text{ MPa}$), and TEB ($3.7 \pm 1.0\%$). A comparison of the mechanical properties of the INI (13.79 MPa) treated fiber/polyester composite and glass-fiber SMC is shown in Fig. 4a. After using high-pressure-INI-treated fibers, the mechanical properties (MOE, MOR, BEB, **TM**, TS, and TEB) of the composite were 102.7%, 109.9%, 90.5%, 92.8%, 100.5%, and 115.8% of the values of glass-fiber SMC, respectively. The results showed that the mechanical properties of the INI (13.79 MPa) treated fiber/polyester composite were very close to or even excess of glass-fiber SMC. Furthermore, if the influence of density was taken into account, the specific mechanical properties of the INI (13.79 MPa) treated fiber/polyester composite could excess the glass-fiber SMC. For this purpose, the specific mechanical properties, which are independent of the density influence, were calculated as follow:

$$\text{Specific mechanical properties} = \frac{\text{Mechanical properties}}{\text{Specific gravity}} \quad (7)$$

The specific gravities of INI (13.79 MPa) treated fiber/polyester composite and glass-fiber SMC were 1.3180 and 1.8018 according with their densities, respectively. The comparison results of specific mechanical properties of the INI (13.79 MPa) treated fiber/polyester composite and glass-fiber SMC are illustrated in Fig. 4b. The specific mechanical properties (specific MOE, MOR, **TM**, and **TS**) of the INI (13.79 MPa) treated fiber/polyester composite were 140.3%, 150.2%, **126.8%**, and **137.4%** of the values

335 of glass-fiber SMC, respectively, which showed better specific mechanical properties for the INI (13.79 MPa)
 336 MPa) treated fiber/polyester composite. In general, the mechanical properties and specific mechanical
 337 properties of INI (13.79 MPa) treated fiber/polyester composite were comparable with or even higher
 338 than the commercial automotive glass-fiber SMC.

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341 **Fig. 4.** Comparisons of mechanical properties and specific mechanical properties of INI (13.79 MPa)
 342 treated fiber/polyester composite and glass-fiber SMC.

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344 4. Conclusions

345 The INI process was improved by introducing an external pressure. The higher the pressure applied, the
 346 more the $MgCO_3$ was loaded to the fibers. The $MgCO_3$ content reached an increase of 180.3% after
 347 applying a pressure of 13.79 MPa. The mechanical and water-resistant properties were increased after
 348 employing INI-treated fibers, these properties were further improved after the pressure-INI-treatment.
 349 The INI (13.79 MPa) treated fiber/polyester composite owned the comparable mechanical properties
 350 with commercial automotive glass-fiber SMC. Moreover, due to the lower density of the INI (13.79
 351 MPa) treated fiber/polyester composite, the specific mechanical properties exceeded that of glass-fiber
 352 SMC. Due to their excellent mechanical properties, kenaf-fiber-reinforced composites are highly
 353 possible to replace glass-fiber SMC in the automotive industrial.

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