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Vacuum-assisted resin infusion (VARI) and hot pressing for $CaCO_3$ nanoparticle treated kenaf fiber reinforced composites

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ABSTRACT

This work was to apply the vacuum-assisted resin infusion (VARI) process and use calcium carbonate inorganic nanoparticle impregnation (INI) to improve the mechanical properties and water resistance of the kenaf fiber/polyester composites. The results show that the modulus of elasticity (MOE), modulus of rapture (MOR), tensile modulus (TE) and tensile strength (TS) of the composites made with INI-treated fibers are increased by 33.1%, 64.3%, 22.3% and 67.8%, respectively, compared with the composites made with un-treated fibers. The thickness swelling of 24-h water submersion is reduced from 19.7% to 1.9%. The moisture contents of the composites after the conditioning and water submersion are reduced from 5.8% to 1.5% and 18.3% to 2.2%, respectively, when INI-treated fibers are employed. The improvement makes the kenaf fiber/polyester composites possible to replace the glass fiber SMC for the automobile application.

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1. Introduction

Large quantities of potentially useful but largely underutilized kenaf (*Hibiscus cannabinus, L.* Malvaceae) fibers presently exist and are available for conversion into value-added products [1,2]. With both economic and ecological advantages, kenaf is well-known as a good cellulosic source. Interest has arisen in the manufacturing of kenaf composites due to their excellent characteristics of low density, low cost, and availability [3–6]. In addition, some studies were conducted for the improvement in mechanical properties of kenaf composites [7,8].

However, the interfacial bonding between the fiber and the matrix is crucial for the load transfer from the resin matrix to the fibers. Poor bonding at the interface of the composites reduced the efficiency of the load transfer from matrix to fiber, resulting in poor mechanical properties of the composites and weak to environmental attacks [9]. Different types of surface treatment procedures were reported to enhance the interaction between natural fibers and matrix. Among these treatment types, alkali treatment is the easiest surface treatment technique for natural fibers. The effect of alkali treatment on mechanical and thermal properties of composites was investigated by many researchers [10–12]. It was found a better adhesion of fibers with matrixes due to the surface modification by the alkali treatment. Fiore et al. confirmed that the alkaline treatment improved mechanical properties of kenaf fibers and their epoxy composites [13].

In comparison with conventional materials, various types of nanoscale materials, including carbon nanofibers, carbon nanotubes, nanoclay, and metal oxides, provide opportunities for improving the performance of composites. It was established that the addition of nanoparticles into a matrix can improve thermal and mechanical properties significantly without compromising the processability of the composites [14,15]. By adding nanoclay as filler particles, the moisture barrier, flame resistance, thermal, and mechanical properties of polymeric composites were improved [16]. Due to the good bonding in composites, the high surface area of nanoparticles was one of the most promising characteristics. Also the dispersion of nanoparticles in the matrix was another important parameter in fabricating nanophased composites. Thus, the processing technique, such as solution blending, shear mixing, in situ polymerization, ultrasonic cavitation, or high pressure mixing, played a critical role in the composite manufacturing [17,18].

The inorganic nanoparticle impregnation (INI) has been proved a promising method to improve the compatibility between natural fibers and polymer matrices [19–21]. 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

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tensile modulus and strength of the composites reinforced with INI-treated fibers by 25.9% and 10.4%, respectively [19]. Cheng et al. also reported that the compatibility between bamboo fibers and polypropylene matrix materials was increased by filling bamboo fibers using $CaCO_3$ [22]. The tensile strength and modulus of composites reinforced with treated fibers increased by 14.58% and 19.66%, respectively, compared with the composites reinforced with untreated bamboo fibers.

The vacuum-assisted resin infusion (VARI) technology is one of the well-established processes used to fabricate high-performance composites using synthetic fibers, such as glass or carbon fibers [23–26]. Interest has arisen in the manufacturing of natural fiber composites using the VARI process [27]. As an important driving force for the impregnation of the fiber tows, the capillary effect of natural fibers played an important role in the VARI process [28].

Water absorption is a drawback with natural fiber reinforced composites. Jawaid and Khalil reported that water content of natural fiber reinforced composites varies between 5 and 15% [29]. Whereas, the synthetic fiber reinforced composites absorb water around up to 4% depending on type and amount of fiber, type and amount of resin, and environmental conditions [30,31]. The jute fiber reinforced polyester composites fabricated by the VARI process exhibited 8.9% water gain at room temperature having fiber volume fraction of about 40%. Vilay et al. fabricated alkali treated and untreated baggage fiber reinforced polyester composites using the vacuum bagging process [32]. It was reported that 7% and 12% water were absorbed by the treated and untreated fiber reinforced composites, respectively, under the same condition. Any natural fiber reinforced composite to be effective needs to bring the water absorption rate down to 4% or below.

The effect of alkali treatment and nanoclay on thermomechanical properties of jute fabric reinforced polyester composites (JPC) fabricated by the VARI process was investigated by Dewan et al. [33]. The dynamic elastic/plastic responses and glass transition temperature (T_g) in treated jute polyester composites (TJPC) and nanoclay infused TJPC were enhanced compared with the untreated samples. Alkali treatment and nanoclay infusion also resulted in enhancement of mechanical properties of JPC. The maximum flexural, compression, and interlaminar shear strength properties were found in the 1% nanoclay infused TJPC. The enhanced thermo-mechanical properties in the composites were obtained due to the strong interaction between the organoclay and polyester. Lower water absorption was also observed due to surface treatment and nanoclay infusion in the TJPC. Crystalline properties of poly (L-lactic acid) composites were improved by filling nanometer calcium carbonates [34]. Compressive properties of composites were enhanced using nano-calcium carbonate/epoxy [35]. Furthermore, to understand the reasons of the enhancement of composite properties, the nano-structural interfaces were examined using computational micromechanical methods [36].

No report in the literature was found on the manufacturing of kenaf composites by means of calcium carbonate nanoparticle impregnated fibers using the VARI process. Hence, the objectives of this study were to fabricate composites using calcium carbonate nanoparticle impregnated kenaf fibers through VARI process and explored the effect of the calcium carbonate on the mechanical properties and water resistance behavior of the composites.

2. Materials and methods

2.1. Materials

Obtained from Kengro Corporation (MS, USA), the kenaf bast fibers were chopped into approximately 50.8 mm in length. Sodium hydroxide (NaOH) solution (5%, w/v) was prepared using NaOH beads (≥97%, Acros Organics) and deionized water that was from the Millipore Milli-Q Integral Water Purification System. The 0.1 mol/L solutions of calcium chloride ($CaCl_2$, ≥96%, Fisher Scientific) and sodium carbonate (Na_2CO_3, ≥99.5%, Fisher Scientific, USA) were prepared. Unsaturated polyester AROPOL Q6585 (30% styrene, Ashland Chemicals, USA) and tert-butyl peroxybenzoate (t-BP, 98%, Acros Organics, USA) were used to fabricate the fiber/polymer composites. Each specimen had a resin content of 33.4 ± 3.6%.

2.2. INI process

A three-step INI process was carried out. 1) A mixture of 120 g kenaf bast fibers (9.11% moisture content measured by Mettler-Toledo HB43-S Moisture Analyzer) and 1.8 L NaOH solution were added into a hermetical reactor (Parr Instrument Co. 251 M). This alkali retting process was carried out at 160 °C for one hour with a 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 ± 1.0%. 2) The retted fibers and 1.8 L $CaCl_2$ solution were added into the hermetical reactor. The reactor was then heated to 100 °C, and maintained the temperature for 0.5 h. The saturated vapor pressure was 0.10 MPa. 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 solution in the hermetical reactor. After the reaction at 100 °C for 0.5 h, the mixture was cooled down to room temperature, filtered, washed with running water, and then dried to form a mat preform with dimensions approximate $100 \times 165 \times 20\ mm^3$ (width, length and thickness).

2.3. Determination of $CaCO_3$ content

The $CaCO_3$ contents were calculated based on the difference in ash contents between the retted kenaf fibers and $CaCO_3$ impregnated kenaf fibers. All the contents were calculated in mass fractions. The ash contents were determined by burning the samples in a muffle furnace first at 400 °C for 30 min, and then at 850 °C for 45 min [19]. After the heating, $CaCO_3$ was decomposed into CaO and CO_2 [37,38]. Based on the CaO amounts in the residuals, $CaCO_3$ contents in the preforms were calculated to be 2.9 ± 0.8%.

2.4. Composites manufacture through VARI process

After applying a mold-release agent on the surface of the mold, the preform was placed on the mold. A flexible, gas impervious polyethylene sheet (commonly called a vacuum bag) was placed over the mold. After vacuum tubes were inserted in the bag, resin infusion was carried out by a vacuum that was created between the mold and the bag. As a result, the catalyzed resin was supplied to the infusion tubes. The vacuum pulled the resin along the distribution layer into the fiber reinforcement preform. A vacuum of 1.3–1.6 kPa was applied to the infusion system by a vacuum pump (Vacmobile 20/2 System with Becker U4.20). It took about 20 min for applying the vacuum and transferring the resins. The resin curing conducted in the hot press with a pressure of 13 MPa. The resin-infused preforms were cured in two temperature steps, i.e. 100 °C for 2 h, and then 150 °C for 2 h. Once the resin cured and cooled down to room temperature, the vacuum bag and distribution layer were removed.

Twelve panels of INI-treated fiber/polyester and un-treated fiber/polyester composites with a size of $100 \times 165 \times 3\ mm^3$ (width, length and thickness) were prepared by the VARI technology. Before the mechanical property tests, all the specimens were conditioned approximate 30 days to a constant weight and moisture content in a conditioning chamber maintained at a relative humidity of 65 ± 5% and a temperature of 20 ± 3 °C.

2.5. Mechanical property tests

From each composite, two specimens sized 25 × 160 × 3 mm^3 were cut for examining the flexural strength and modulus of the composites by means of 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 tensile strength and modulus of the specimens were tested at a room temperature 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. Twenty four replicates (12 for the specimens made using the INI-treated fibers and 12 for the specimens made using untreated fibers) were used. The specimen dimensions were 19 × 165 × 3 mm^3 and the cross section of the narrow section was 57 × 13 mm^2 (length, width). A crosshead speed of 5 mm min^{-1} was used. The density of each panel was measured in accordance with the ASTM D792 standard.

2.6. Water resistance tests

Six composites with un-treated fibers and INI-treated fibers (three for each) were used for the thickness swelling and moisture content measurements. The specimen dimensions were 25 × 25 × 3 mm^3. Two water resistance test specimens were cut from each composite, counting twelve replicates for the un-treated and INI-treated fiber composites. Two-hour and 24-h water submersion tests were carried out in accordance with the ASTM D1037 standard for the determination of the thickness swelling. In addition, the moisture contents after equilibrium in the conditioning chamber at a relative humidity of 65 ± 5% and a temperature of 20 ± 3 °C for 10 days and water submersion for 24 h were determined in according with the ASTM D4442 standard.

3. Results and discussion

3.1. Mechanical properties

The results of bending tests (MOE and MOR) and tensile tests (TE and TS) are summarized in Table 1. Compared to the un-treated fiber/polyester, MOE, MOR, TE and TS of the composites made by INI-treated fibers are increased by 33.1%, 64.3%, 22.3% and 67.8%, respectively. ANOVA tests were carried out to examine the significance of the increase. If the p-value is less than α, say 0.001, the null hypothesis is rejected. From the ANOVA tests, the p-values of MOE, MOR, TE and TS are 1.29×10^{-6}, 1.40×10^{-9}, 7.87×10^{-4} and 3.22×10^{-9}, respectively. The test results indicate that the increments are significant at the Probability Level of $\alpha = 0.001$. The previous research reported that TE and TS of the composites made from INI-treated fibers and polypropylene (PP) are increased by 5.3–10.4% and 10.6–25.9%, respectively, compared with the untreated fiber/PP composites [19]. Two reasons are proposed for the incensements: 1) the mechanical properties of fibers are enhanced since the fiber voids were filled by the nanoparticles, 2) the interfacial compatibility between the fiber and polymer matrix is improved, which can be confirmed by the scanning electron microscope (SEM) observation.

3.2. Water resistance

The water resistance properties of un-treated fiber/polyester and INI-treated fiber/polyester composites are presented in Table 2 for a comparison. The results of thickness swelling tests show the INI-treatment dramatically reduces the thickness swelling. The thickness swellings of 2-h and 24-h water submersion are dramatically reduced by 88.6% and 90.5%, respectively, when INI-treated fibers are employed. The statistical analysis (ANOVA, $\alpha = 0.001$) shows that the p-values of 2-h and 24-h water submersion are 4.73×10^{-9} and 5.07×10^{-7}, respectively, which indicates that the increases in thickness swelling of the INI-treated fiber composites are significant at the level of $\alpha = 0.001$. The 24-h thickness swelling of INI-treated fiber/polyester is as low as 1.9%. The results indicate that the $CaCO_3$ nanoparticles impregnation dramatically improve the water-resistant property of kenaf fiber/polyester composites.

The moisture contents after the conditioning and water submersion are listed in Table 2. The results show that the moisture contents of INI-treated fiber/polyester composites after the conditioning and water submersion are reduced from 5.8% to 18.3%–1.5% and 2.2%, which counts a reduction of 73.9% and 88.1%, respectively, compared to that of the un-treated fiber/polyester composites. The ANOVA analysis ($\alpha = 0.001$, p-values $= 4.42 \times 10^{-6}$ and 2.18×10^{-4}) reveals that the reductions in moisture contents of the INI-treated fiber composites are significant at the level of 0.001.

There could be two reasons making contributions to the dramatic improvement of the water resistance of the INI-treated fiber/polyester. The first one is that $CaCO_3$ protected the fibers from absorption of water because $CaCO_3$ is able to adhere onto the fiber surfaces to block the large amount of hydroxyl groups [39]. These $CaCO_3$ nanoparticles dramatically reduce the contact between the water and nature fibers, resulting in better water resistance. The second one is $CaCO_3$ itself. The reported contact angle of ground $CaCO_3$ was 0° [40], designating 100% hydrophilic, while the $CaCO_3$ precipitates from water at ambient conditions with a solubility of 47 mg/L and none swelling in water. The precipitate and none swelling behaviors of $CaCO_3$ will only absorb little water on the surface of nanoparticles. This finding supports our results and

Table 1
Mechanical properties of kenaf reinforced composites.

Composites	Density (kg m^{-3}) Mean (SD[a])	MOE[b] (GPa) Mean (SD)	MOR[c] (MPa) Mean (SD)	TE[d] (MPa) Mean (SD)	TS[e] (MPa) Mean (SD)
Un-treated fiber/polyester	1159.4 (44.5)	6.9 (0.6)	68.3 (6.5)	10.7 (1.0)	44.5 (3.8)
INI-treated fiber/polyester	1286.4 (9.0)	9.2 (0.9)	112.2 (13.4)	13.1 (0.7)	74.7 (6.3)
Increment (%)	–	33.1	64.3	22.3	67.8

[a] SD = standard deviation.
[b] MOE = bending modulus of elasticity.
[c] MOR = bending modulus of rupture.
[d] TE = tensile modulus of elasticity.
[e] TS = tensile strength.

Table 2
Thickness swelling and moisture contents of kenaf reinforced composites.

Composites	Thickness swelling (%)		Moisture content (%)	
	2-h Submersion Mean (SD)	24-h Submersion Mean (SD)	After conditioning Mean (SD)	After submersion Mean (SD)
Un-treated fiber/polyester	3.3 (0.3)	19.7 (3.8)	5.8 (0.2)	18.3 (2.3)
INI-treated fiber/polyester	0.4 (0.2)	1.9 (0.5)	1.5 (0.1)	2.2 (0.2)
Decrease (%)	88.7	90.5	73.9	88.1

explains the reason of adding CaCO3 could increase water resistance. The positive effects of the both situations help improve the water resistance of the composites dramatically.

3.3. *Micromorphology*

The surfaces of un-treated fiber and INI-treated fiber were examined by SEM (Fig. 1). The $CaCO_3$ are clearly seen on the surfaces of INI-treated fibers (Fig. 1b), comparing to the smooth surfaces of un-treated fibers (Fig. 1a).

The SEM images of tensile fracture surfaces are presented in Fig. 2. The composites of un-treated fiber/polyester obviously show a number of fiber bundles and long fibers (Fig. 2a1–a3). These pullout fibers are much longer compared with that of INI-treated fiber/polyester, indicating that the pullout fibers do not well adhere to the resin matrix. As a result, the mechanical properties are reduced.

After the tensile examinations, the broken sample surfaces of INI-treated fiber/polyester composites are shown in Fig. 2b1–b3. Though there still have a number of fibers pullout, these fibers are much shorter and more simultaneous failure. This phenomenon was also reported in the previous research for INI-treated fiber/PP composites [19]. Furthermore, almost no fiber bundle is observed in these images. It is indicted that the interfacial compatibility between fibers and matrix is highly improved. The higher interfacial compatibility results in a stronger adhesion between the fibers and matrix, contributing to the higher mechanical properties.

3.4. *A comparison of kenaf fiber composites and glass fiber SMC*

Glass fiber sheet molding compound (SMC) is widely used in the automotive manufacturing due to its lightness and strength. However, it is difficult to recycle owing to its physiochemical composition, which will cause environmental pollution. An increasing attention is being devoted to the application of kenaf fibers because they are lighter and available at lower cost than glass fibers. Furthermore, 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 [3]. Based on the treatment time and temperature, it was roughly estimated that less than 15% energy consumption could be increased for the treatment of kenaf fibers. Even though the increase in energy consumption ($15MJ + 15\ MJ \times 15\% = 17.25\ MJ$), it was still much lower than that for glass fibers. Based on the life cycle assessment (LCA) study [41], less negative environmental impacts were found in “cradle to gate” consideration of kenaf fibers compared to glass fibers.

The comparisons of physical properties and water resistance between glass fiber SMC (SLI 323IF 2.5 mm, Meridian Automotive Systems Inc., MI, USA) and INI-treated fiber/polyester composites are shown in Table 3. The results show that the density of INI-treated fiber/polyester composite is 28.6% lower than that of the glass fiber SMC, which is a great benefit in reducing automotive weight. The resin contents of glass fiber SMC and INI-treated fiber/polyester are comparable (26.0% of glass fiber SMC *vs.* 33.4% of INI-treated fiber/polyester). The fiber content of INI-treated fiber/polyester composites (64.7%) is much higher than that of glass fiber SMC (28.0%) owing to the lower content of filling minerals (1.9% of $CaCO_3$).

The mechanical properties of the INI-treated fiber/polyester composites and glass fiber SMC are compared in Fig. 3. After using INI-treated fibers, the mechanical properties of MOE, MOR, TE and TS of the INI-treated fiber/polyester composites reach 84.6%, 81.2%, 95.7% and 87.3% of the values of fiber glass SMC. According to the water resistance and density results, the INI-treated fiber/polyester composites have a great possibility to replace glass fiber SMC in the automotive manufacturing.

4. Conclusions

The composites made with $CaCO_3$ nanoparticles impregnated kenaf fibers through VARI process significantly improve the interfacial compatibility between fibers and resin matrix based on the

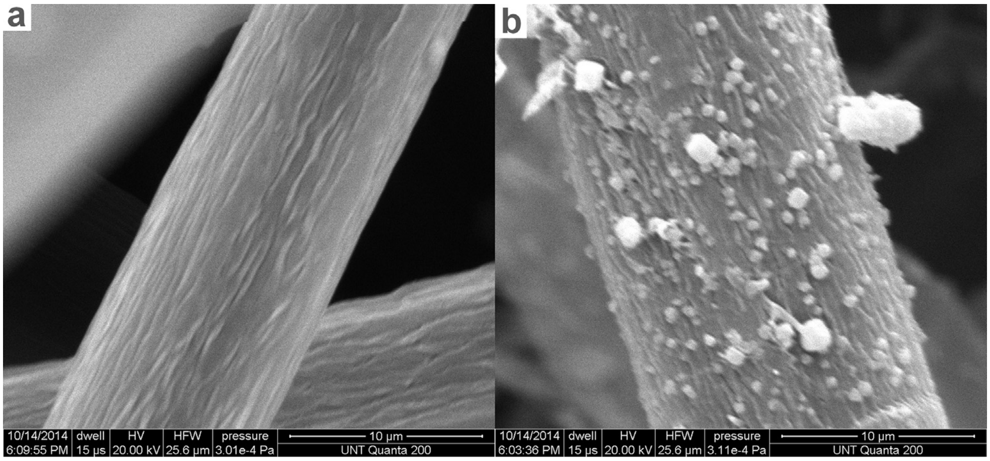

Fig. 1. SEM examination of the surfaces of un-treated fiber (a) and INI-treated fiber (b).

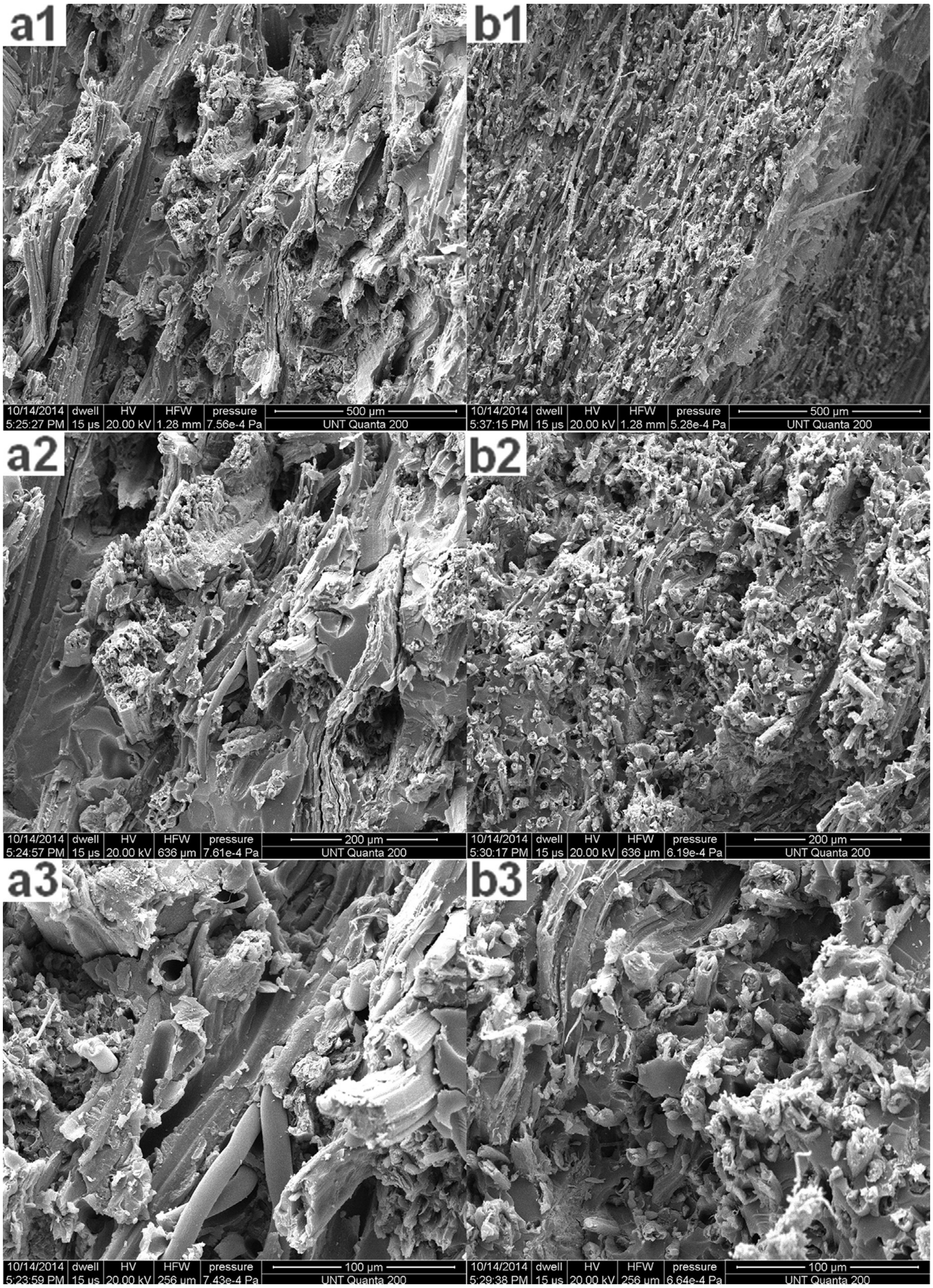

Fig. 2. A comparison of composites of un-treated fiber/polyester (a1–a3) and INI-treated fiber/polyester (b1–b3) at three different magnification levels by SEM.

Table 3
Physical properties and water resistance comparisons of glass fiber SMC and INI-treated fiber/polyester composites.

Sample	Density	Contents (%)[d]			Moisture content (%)[e]	
	($Kg\ m^{-3}$) Mean (SD)	Resin[a] Mean (SD)	Fiber[b] Mean (SD)	Mineral[c] Mean (SD)	Conditioning Mean (SD)	Submersion Mean (SD)
Glass fiber SMC	1801.8 (27.1)	26.0	28.0	46.0	0.3 (0.0)	1.1 (0.3)
INI-treated fiber/polyester	1286.4 (9.0)	33.4 (3.6)	64.7 (4.1)	1.9 (0.5)	1.5 (0.1)	2.2 (0.2)

[a] Unsaturated polyester resin.

[b] Glass fiber and kenaf fiber, respectively.

[c] $CaCO_3$ and other minerals for glass fiber SMC, and $CaCO_3$ for INI-treated fiber/polyester.

[d] All the contents calculated in the mass fractions, the contents of glass fiber SMC come from the original datasheet determined by ASTM D4000 standard.

[e] The moisture after condition and after submersion (24 h) were determined in according with ASTM D1037 and D4442, which was described in "2.6 Water resistance tests".

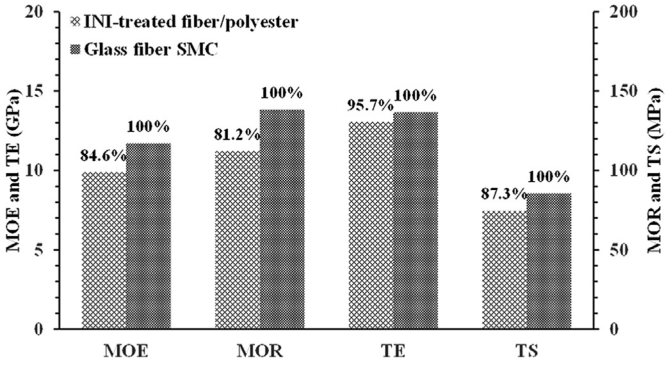

Fig. 3. Comparison of mechanical properties of INI-treated fiber/polyester composites and glass fiber SMC.

comparison of the mechanical properties. Compared to the non-impregnated fibers, the mechanical properties, MOE, MOR, TE and TS, are improved by 33.1%, 64.3%, 22.3%, and 67.8%, respectively. The thickness swelling of 24-h water submersion is reduced from 19.7% to 1.9%. The moisture contents of the composites after conditioning and 24-h water submersion are reduced from 5.8% to 1.5% and 18.3% to 2.2%, respectively. In additional, the comparisons of mechanical properties, density and water resistance between glass fiber SMC and $CaCO_3$ nanoparticles impregnated kenaf fibers/polyester, indicate a high possibility of kenaf fiber reinforced composites replacing automotive glass fiber SMC.

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