Effect of natural fiber types and sodium silicate coated on natural fiber mat/PLA composites: Tensile properties and rate of fire propagation

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Abstract. In this study, 3 types of natural fibres, i.e. jute, sisal and abaca, were plain weaved to fibre mat. Before weaving, the fibres were treated with 5% NaOH to remove hemi cellulose and lignin. The weaving was performed by hand using square wooden block fit with nails for weaving using one and two types of natural fibres as weft and warp fibre to produce natural fibre mat. The fibre mat was also impregnated in sodium silicate solution extracted from rich husk ash. The pH of the solution was adjusted to pH 7 using H₂SO₄ before impregnation. After predetermined time, sodium silicate was gelled and deposited on the mat. The fabric mat and sodium silicate coated mat were then impregnated with PLA solution to produce prepreg. Dried prepreg was laminated with PLA sheet using compressing moulding machine to obtain natural fibre mat/PLA composite. The composite containing abaca aligned in longitudinal direction with respect to tension force enhanced Young’s modulus more than 300%. Fibre mat composites with abaca aligned in longitudinal direction also showed tensile strength enhancement nearly 400% higher than neat PLA. After coating with sodium silicate, the tensile modulus of the composites was found slightly increased. The silicate coating was disadvantage on tensile strength of the composite due to the effect of sodium hydroxide solution that was used as solvent for silicate extraction from rice husk ash. However, sodium silicate could retard rate of fire propagation about 50% compare to neat PLA and about 10% reduction compared to fibre mat composites without sodium silicate coated fibre mat.

1. Introduction

Natural fiber reinforced composites have been paid high attention to extend their applications all over the world. Composites base on natural fiber and biopolymer [1-4] have received much attention as they are environmental friendly. The applications of natural fiber reinforced biopolymer mostly are on non structural part such as some part in the building or automotive parts [5]. Generally short natural fibers are added to bioplastic, as matrix, due to its processing ability using general thermoplastic processing techniques such as twin screw extruder and injection molding machine. The fiber length is one of the factor controlling mechanical properties of composites [6-9]. Fiber added for compounding...
the composites was normally in the range of 0.1-13 mm. The length which is longer than 20 mm may cause agglomeration during processing. The content of fiber incorporated into matrix was also limited to the highest content of 30-40% by weight. The fiber could be degraded physically, length shortening, during processing [10]. Misra and coworker [11] studied laminated biocomposites which prepared from stacking 4 layers of poly (lactic acid) sheet and 3 layers of kenaf fibers alternatively. They were laminated by using compression molding machine. The fiber content used was highest at 40% content. They found that the flexural modulus was found about 4 times higher than neat PLA. Khonder et al. [12] prepared unidirectional fiber composite using microbraid yarns wrapped around the metal frame and followed with compression mold. The microbraid yarns prepared from jute fibers being braided around with polymer matrix yarn. Memon et al. [13] had also adapted the same technique to fabricate unidirectional composite of jute yarn hybrid with PLA yarn. The composites were prepared by using compression molding machine. They prepared composites with fiber volume fraction of 37-53%. The impregnation of PLA into the fibers was dependent upon temperature used for compression. Skrifvars and coworker [14] studied thermoplastic composites manufactured using PLA/hemp co-wrapped hybrid yarn prepreg. The manufacturing was carried out by wrapping PLA filaments around a core that prepared from a 400 twists/m of 25 tex hemp yarn and 18 tex PLA yarn. They vary hemp yarn in the range of 10-45% by weight. The prepreg was prepared by compression molding. They found that Young’s modulus and tensile strength were increased about 2 times of neat PLA. The fiber mat made from woven and knitting has also been paid attention [15-25]. They are advantage in term of mechanical performance. Among them, Rajini and coworker [25] studied unidirectional natural fiber unsaturated polyester composites having banana and kenaf fibers and hybrid fibers, i.e. banana fiber hybrid with kenaf fiber, mat. The mat was prepared by weaving fiber and hybrid fiber in different pattern. The composites were prepared by hand lay-up technique. They report that the mat prepared by plain weaving hybrid composites can improve mechanical strength better than other pattern. Maranon and coworker [17] prepared laminate composite from bamboo fabric reinforcing in PLA. They found that composite with fiber in weft direction is greater than warp direction. Khan and coworkers [21] prepared woven jute fabric reinforced PLA composite using also unbalanced plain weave. In this research they found composites in warp direction are higher than those in weft direction. They also reported that tensile strength and tensile modulus were improved about 103% and 211%, respectively.

Prepreg is a class of premix composites or compound of composite that consists of fiber impregnated with polymer resin. Brouwer explained the possibility of preparation natural fiber prepreg for automotive parts [22]. Thermosets, thermoplastic and biopolymer matrix have potential for prepreg preparation. Flammability and thermal properties of natural fiber composites have been also very important for automotive and other applications [23, 24].

It was widely known that natural fibres are various in their properties [25, 26]. Their properties were also dependent upon their size and morphology [26-31]. In this study, we selected three types of natural fibers which are abaca jute and sisal fibers. Jute fiber is from as bast of Corchorus plant whereas abaca and sisal are from leaf of banana and Agave sisalana leaves respectively. These three types of fibers are commonly available in Thailand. This preliminary work was intended to prepare natural fiber mat by plain weave using the same size of fiber in warp and weft direction. Type of fibers used in weaving could be the same type and hybrid of fiber types. The prepreg was prepared impregnation of fiber mat in PLA solution. This study was also aimed to improve flame resistance of natural fiber mat by coating silica on fiber via so-gel process where the silicate solution was extracted from rice husk ash.

2. Materials and method

2.1. Materials
Abaca, sisal and jute fibers were supplied by OTOP centre from Hup Krupung Market, Prachuap Khiri Khan Province, south of Thailand. PLA 4043D was purchased from NatureWork LLC. Rice Hush ash was kindly supplied from local electric power plant.
2.2. Method

2.2.1. Preparation of fiber mat. Before plain weaving, all fibers used in this study were treated with 5% NaOH at 80°C for 4 hours. The fibers were cooled to room temperature before neutralization with 5% HCl to pH 7. The fiber was left overnight for thoroughly neutralization. Water was squeezed out, before being washed several times with distilled water. Treated fibers were dried in a hot air oven at 60 °C until totally dried. The dried fiber was then aligned and plain weaved in the square wooded block with the dimension of 30x30 cm in figure 1. The balance fiber mat, of 56x56 (56 yarns in warp and 56 yarns in weft directions per 150 mm) and average weight of 230 g/m², was obtained. The fiber mat and hybrid fibers mat was designed and designated as stated in table 1. In order to study the effect of silicate coating, fiber mat was impregnated in sodium silicate solution in which its pH was adjusted in order to gel sodium silicate within predetermined time. The sodium silicate solution was obtained from extraction of rice husk ash, 10 grams with 300 ml 1 M NaOH at 100-110°C for 16 hours. After predetermined time sodium silicate gelled and was deposited on the fiber mat surface with average weight gained about 20% by weight of dried fiber mat.

Table 1. Code represent fiber types used for weaving to natural fiber mat. A=Abaca, J=Jute and S=Sisal.

| Warp Weft | Sisal Fibre | Abaca Fibre | Jute Fibre |
|-----------|-------------|-------------|------------|
| Sisal Fibre | S + S       | S + A       | S + J      |
| Abaca Fibre | A + S       | A + A       | A + J      |
| Jute Fibre | J + S       | J + A       | J + J      |

Figure 1. Wooden block used for plain weave to produce natural fiber mat.

2.2.2. Molding of natural fiber mat/PLA composites. The preparation of fiber mat composites was conducted in 2 steps. Firstly, the fiber mat obtained from section 2.2.1 was impregnated in PLA solution prepared from dissolution 20 g of PLA in 100 ml chloroform. Fiber mat was then left to dry in air. 20 grams of dried PLA was compressed in a compression molding machine at 170°C with the hydraulic pressure of 500 psi giving rise to PLA sheet with the thickness of 0.8-1 mm. The PLA sheet was cut into the same size as prepreg and laminated with PLA sheet using compression mold at 170°C with the pressure of 500 psi. The composite sheet was obtained with the thickness of 1-2 mm. Weight fraction of fabrics in composites was about 25 % in all fabricated composite.

2.2.3. Mechanical measurements. Tensile test was performed in warp direction (as longitudinal direction) according to ASTM D 638 using Universal Testing Machine (Instron 3300). The specimens for tensile were cut into dumbbell shape with 50 mm length and 35 mm gauge length. The test was performed at the cross head speed of 5 mm/min. At least 15 specimens were used for investigation.

2.2.4. Fire resistance. Flame rating was performed according to UL-94 flammability standard. The specimen was cut using laser cutter (Laser Engraving Cutting Machine model 6040 N) to a rectangular shape with the length, width and thickness of 125 mm, 13 mm and 0.8 mm respectively. The specimen
was clamped vertically after conditioned in 50% RH at 25°C for 48 hours. Blue flame of Bunsen burner was applied to the edge of the specimen for 10 minutes. The flaming time was recorded with respect to 10 mm length on the specimen. Plastic drip was allowed to fall on surgical cotton that placed at 30 cm. below the burning specimen. Burning rate was calculated from burning length divided by flaming time.

3. Result and discussion

3.1. Stress-strain behavior

Plain weave prepared in this study was balanced plain weave where fibers in warp and weft directions have the same size. Warp direction was tensioned as longitudinal direction (parallel) with respect to tensile force. Weft fibre was in transverse direction (perpendicular) with respect to tensile force. The first letter is short abbreviation of fibre such as A which stood for abaca fibre, J stood for jute fibre and S stood for sisal fibre. For labelling the composite system, the first alphabet was designated for fibres in transverse direction and the second one was designated for fibres in longitudinal direction. The fiber mat composites were prepared by compression prepreg with PLA sheet. The content of fabric was kept at the content of 20% by weight.

Stress-strain curves of PLA reinforced with natural fiber mat and hybrid natural fiber mat composites is shown in figure 2. It was shown that natural fiber mat/PLA composites are brittle than neat PLA which has strain extension at 4.13%.

| Fiber system | Fabric | A+A | J+A | S+A | A+J | J+J | S+J | A+S | J+S | S+S |
|--------------|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| % weight gained | 28.76 | 10.46 | 9.18 | 10.29 | 18.17 | 19.68 | 9.42 | 8.64 | 9.60 |

Table 2. Percentage weight gained after sol-gel process.

![Figure 2](image-url) Tensile stress-strain of natural fiber mat/PLA and hybrid natural fiber mat/PLA with (a) abaca fiber (second alphabet) A, (b) jute fiber, J, and (c) sisal, S in longitudinal direction.

Figure 2. Tensile stress-strain of natural fiber mat/PLA and hybrid natural fiber mat/PLA with (a) abaca fiber (second alphabet) A, (b) jute fiber, J, and (c) sisal, S in longitudinal direction.
Figure 3. Young’s modulus (a) and tensile strength (b) of natural fiber mat/PLA and hybrid natural fiber mat/PLA composites.

Figures 3(a)-3(c) show Young’s modulus and tensile strength of neat PLA, natural fibre mat/PLA compared with hybrid natural fibre mat/PLA composites. It was shown that Young’s modulus and tensile strength of natural fibre mat/PLA and hybrid natural fibre mat/PLA were improved about 200-400% dependent on fiber system used for reinforcement.

Young’s Modulus of the composites having abaca fiber aligned in longitudinal direction was found to be achieved about 258-299% compared to neat PLA, figure 3(a). It could also be seen that if the composite have abaca fiber aligned in longitudinal direction and jute fiber aligned in transverse, the composite would be enhanced about 299%. This result showed the synergism of two fiber types where network structure of abaca fiber [27] induced good penetration of PLA, seen by fracture surface examined by SEM, in figure 4(a), led to good adhesion at the interface. Jute fiber also has similar structure as abaca which jute is produced from stem. The small filament of jute, as shown in figure 5, could be responsible for its higher modulus [32]. The finding in this present study was found to have modulus achievement very much higher than those reported by Memon [13] where they found the achievement in modulus about 95% at optimum moulding temperature. Although the content of fabric in this study was lower, 20% by weight. This could be by the impregnation of PLA into fabric prepared in this study and the network structure of abaca, as mentioned previously.

The natural fibre mat having jute fibre aligned in longitudinal direction was found to achieve slightly lower modulus than abaca. SEM micrograph of fractured surface, shown in figure 4(b) confirms the poorer adhesion between jute fiber bundle and PLA and this resulted in lower in modulus compared to composite reinforced with fiber mat having abaca fiber aligned in longitudinal direction. Not only the poor adhesion but jute fiber also has low cellulose content than abaca fiber [33]. Sisal fiber also shows poor adhesion at the interface, seen figure 4(c). It was explained by Smole et al. [33] that sisal fiber is fairly coarse and inflexible. Although sisal contains percentage of cellulose similar to jute fiber, its tensile properties are not uniform along its length. Sisal fibre also has lower Young’s modulus than jute fibre [27]. These could be the reasons that affect natural fiber mat composite having sisal fiber in longitudinal direction to have inferior modulus than abaca and jute fibers.

Tensile strength of natural fibre mat composites are shown in figure 3(b). It was generally seen that natural fibre mat/PLA composites having abaca fibre in longitudinal direction shows tensile strength achievement about 306–385%. The network structure and good adhesion at the interface was responsible for high tensile strength. In fibre mat composite system having jute fibre aligned in longitudinal direction, their tensile strength achievement was found in the range of 237-307% compared to neat PLA, which were slightly lower than that of abaca fibre system. As explained earlier, jute showed adhesion between fibre and matrix inferior to abaca fibre. This was indicated by SEM micrograph in figure 4(b). Moreover, Jute has cellulose content (61-71%) lower than that of abaca fiber (76.6%) [33]. It could be another reason for their tensile strength. Again natural fiber mat/PLA composites having sisal fiber aligned in longitudinal direction generally showed the lowest tensile
strength, 210-293% achievement which resulted from non uniform tensile properties along fiber length and also from the cellulose content in the fiber (70%) [33].

Figure 4. Fractured surfaces of natural fiber/PLA mat composites focusing on longitudinal fiber fractured showing (a) abaca fiber, (b) jute fiber and (c) sisal fiber.

3.2. Effect of Sol-gel Sodium silicate on fabric surface

The fiber mat that was coated with sol-gel of sodium silicate solution shows weight gained after impregnated in pH adjusted sodium silicate solution for predetermined time. The coated fabric was then dried at 80°C in a hot air oven until dry. The weight gained was tabulated in table 2. Percentage weight gained was varied depending on fiber and fabric structure. As abaca fiber has network structure, it could absorb sodium silicate solution and hence sol-gel occurred much more than other fiber fabric systems.

Figure 5. Natural fiber mat/PLA composites with various fiber mat system (a) Young’s Modulus and (b) tensile strength compared between sodium silicate untreated and treated fiber mat.

Tensile properties, i.e. Young’s modulus, tensile strength of fiber mat/PLA composite compared with sodium silicate treated fiber mat/PLA composites are shown in figures 5(a) and 5(b). The silicate coated on the fiber mat caused Young’s modulus of the composites to be increased due to silicate stiffness. However, natural fiber fabric having abaca aligned in longitudinal direction showed small increment in Young’s modulus. Due to the network structure of abaca caused absorption of sodium silicate solution more than jute and sisal fibers. Zhang [34] found that under long time NaOH treatment, natural fiber could be swollen and fragmented. It can affect the mechanical behavior of the fiber. For sisal fiber, the fiber morphology is quite different from abaca and jute. It could be seen from figure 6. The diameter of sisal fiber seems to be larger than that of abaca and jute fibers. Silicate could gel only on the outer surface of fiber fabric. The content of silicate coated on the fabric shown in table 2 also showed the lowest weight gained hence less fiber swollen and fragmented. Its mechanical properties could still be sustained.

Figure 6. Virgin (a) abaca fiber, (b) jute fiber and (c) sisal fiber; fractured surface of silicate treated natural fiber mat (d) abaca, (e) jute and (f) sisal aligned in longitudinal direction.
3.3. Fire resistance

Figure 7 shows average rate of flame propagation obtained from 10 specimens of each composite compared to neat PLA, using UL-94. PLA was burning very fast with dripping and the dripping carry on flaming. Whereas natural fiber fabric/PLA composites decreased rate of fire propagation evaluated in term of fire propagate distance per second about 40%. The decreased in fire propagation rate could be caused by lower in amount of polymer and also by the presence of natural fibers. Dripping was fond decreased due to the fabric protecting molten plastic to be dripped. Type of fibers did not show significant difference of the fire propagation. After treatment, sodium silicate was deposited on the fabric surface. The improvement was about 17% compared to untreated fabric. This was due to silicate which is widely known for their heat resistance [35-36].

4. Conclusions

Plain weave of natural fibre fabric was prepared using both one type of fibre and hybrid natural fibres. The fabric prepreg was prepared by impregnation the fabric in PLA solution. Prepreg was laminated with PLA sheet using compression moulding machine. The content of the fibre fabric was fixed at 20% by weight. It was found from this work that fabric having abaca aligned in longitudinal direction exhibited modulus improvement up to 399%. Tensile strength was found 385% improvement. The achievement was dependent upon type of fibre aligned in transverse direction. It was found that fabric having abaca aligned in longitudinal and jute aligned in transverse direction exhibited highest Young’s modulus. Tensile strength was also found showed the same trend. The morphology of fibre (network structure and size of fibre bundle are the factors affecting mechanical properties of natural fibre fabric/PLA composite as it control matrix and fabric adhesion at the interface. This work tried to apply sodium silicate extracted from rice husk silica as thermal insulator and hence flame retarder. It was found that after impregnation fabric in sodium silicate solution and allowed to gel for predetermined time sodium silicate was deposited on the fabric. The weight of silicate gained was dependent upon morphology of fibre. Fabric having abaca aligned in longitudinal direction was found to gain the highest silicate deposition. But the effect of alkaline on fibre resulted in slightly increased of modulus. Although less sodium silicate was deposited on the surface of sisal but it was not as swollen as abaca and jute fibre. The mechanical properties of fabric containing sisal aligned in longitudinal direction then clearly exhibited significantly increment in both Young’s modulus and tensile strength. Flammability of composites is crucial for their applications. The fire propagation rate of natural fibre fabric composite was found decrease about 40%. Whereas fabric treated with sodium silicate was found to improve flammability about 50% compared to neat PLA. This finding could give an idea of applying natural resources for composite materials and gave the some basic information for future researches.
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