

Preparation and characterization of jute- and flax-reinforced starch-based composite foams

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Abstract

Starch-based composite foams (SCFs) were successfully prepared by baking starch-based batters incorporating either jute or flax fibers inside a hot mold. The effect of moisture content on the mechanical properties of SCFs was investigated. Both the flexural strength and the flexural modulus of elasticity appeared to be markedly improved with addition of 5–10% by weight of the fibers. At a fixed fiber content of 10% by weight, both the flexural strength and the flexural modulus of elasticity were found to increase with increasing aspect ratio of the fibers. The improvement in the mechanical properties of SCFs was attributable to the strong interaction between fibers and the starch matrix, as evidenced by a series of scanning electron micrographs being taken on SCF fracture surface. Between jute- and flax-reinforced SCFs, jute fibers had a greater reinforcing effect than flax fibers did. Orientation of fibers was shown to have a strong effect on both the flexural strength and the flexural modulus of elasticity of SCFs, with the highest values being observed on specimens having fibers oriented in the longitudinal direction (fibers oriented perpendicularly to the crack propagation direction).

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1. Introduction

In recent years, much progress has been achieved in the development of biodegradable products using agricultural materials as basis. Various approaches have used starch for the production of functional materials. For examples, native starch has been chemically transformed into an easier-to-process thermoplastic starch. Unfortunately, properties of products derived from such a material still do not meet requirements for certain applications, such as in packaging (Averous, Fringant, & Moro, 2001). Nowadays, expanded polystyrene (EPS) foams are used extensively in single-use packaging applications, due largely to their low bulk density, good thermal insulation property, reasonable strength, and low cost. In spite of these favored properties,

there have been continual concerns about the impact of the manufacturing and the disposal of EPS foams on the environment. These concerns have led to an interest in developing alternative foam materials from renewable resources which are readily degradable (Glenn & Orts, 2001a).

Starch is an alternative material for making foams. Previous work (Tiefenbacher, 1993) showed that batters of starch and water can readily be baked in a closed, heated mold where the starch granules gelatinize and the evaporation of water causes the starch to foam out and take up the shape of the mold. Foams made from pure starch have major drawbacks on their brittleness and sensitivity to moisture and water. Further treatments are, therefore, necessary to impart the required properties (i.e. reasonable strength, good flexibility, and water resistance) to the products. Mineral fillers and wood fibers were added to improve strength, while surface coating with waxes or other materials was carried out to improve water resistance, of starch foam products (Andersen, Kumar, & Hodson, 1999).

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Table 2
Batter formulations for preparing starch-based and starch-based composite foams

Component	Weight (g)			
	SF ^a	SCF ^b with 1% fibers	SCF ^b with 5% fibers	SCF ^b with 10% fibers
Tapioca starch	100	100	100	100
Natural fibers	–	1	5	10
Distilled water	85	85	85	85
Magnesium stearate	2	2	2	2
Guar gum	1	1	1	1

^a Denotes starch-based foam.

^b Denotes starch-based composite foam.

varied as summarized in Table 2; in order to evaluate the effect of fiber aspect ratio on mechanical properties of SCFs, the length of the fibers was varied as summarized in Table 1 (for a fixed fiber content of 10 wt%); and finally, in order to evaluate the effect of fiber orientation on mechanical properties of SCFs, continuous flax fibers were pre-oriented in the longitudinal, random, and transverse directions in a mold before the application of the batter (for a fixed fiber content of 10 wt%). It should be noted that the pre-orientation of the continuous flax fibers in both the longitudinal and the transverse directions was carried out very carefully by fixing the fibers at both ends to the mold and care was taken during the application of the batter so that the orientation of the fibers was least-affected.

2.2.3. Baking process

Eighty-five gram of the as-prepared starch-based batters (see Table 2) was applied to a mold having a rectangular-parallelepiped cavity. The size of the mold cavity was 167.3 mm in length, 130.1 mm in width, and 3.1 mm in depth, respectively. A compression press (Wabash, V50H) was used to prepare SCFs from the as-prepared starch-based batters. Each molding was placed between the platens, the temperature of which was fixed at 220 °C. Clean poly(ethylene terephthalate) (PET) sheets were used to cover the molding on both sides to facilitate mould release. Since the apparent melting temperature for PET is around 245 °C (Supaphol, Dangseeeyun, Thanomkiat, & Nithitanakul, 2004), the integrity of the PET sheets at the molding condition was attained. Evaporation of water in the batter formulations began shortly after the mold came into contact with the hot platens and venting of the water vapor occurred around the edge of the mold. In order to achieve maximum venting, very small clamping force was used just to facilitate the closing of the mold. After 150 s, the mold was cooled down to 50 °C at a cooling rate that was fitted well with an exponential decay with a time constant of around 3 min and the as-prepared SCF was demolded.

2.3. Determination of moisture content

Conditioning jars having specific RH levels of 11.3, 32.8, 43.2, 52.9, and 75.3% were prepared by filling the jars with saturated, aqueous solutions of LiCl, MgCl₂, K₂CO₃, Mg(NO₃)₂, and NaCl salts, respectively (according to ASTM E104-85 standard test method). Pre-dried SCF specimens were then conditioned on wire grids over these solutions in these jars at a fixed temperature of 25 °C for a total observation period of 1 week. The weight of each specimen was recorded every 24 h period. The percentage of moisture content of SCFs was then taken as the difference between the weight of the specimen recorded after being conditioned in a respective conditioning jar for a specified observation time and the initial weight, divided by the initial weight and multiplied by 100. The results were reported as an average value from measurements of at least five specimens.

2.4. Physical and mechanical property measurements

The SCF specimens used to investigate the effects of moisture content and fiber content on their physical and mechanical properties were conditioned at 11.3, 32.8, 43.2, 52.9, or 75.3% RH for 3 days, while the SCF specimens used to investigate the effects of fiber aspect ratio and fiber orientation on their physical and mechanical properties were conditioned at 43.2% RH for 3 days. Bulk density of SCF specimens was calculated from the ratio between weight and volume. Prior to mechanical property measurements, the percentage of moisture content on some of the pre-conditioned SCF specimens was determined so that the effect of moisture contents on mechanical properties of SCFs can be reported. Flexural properties for SCF specimens were determined using a mechanical testing machine (Lloyd, LRX series), with the maximum load of 500 N and the span of 50 mm. The specimens were rectangular in shape, cut according to ASTM D790-92 standard test method. The probe was lowered onto each specimen until a load of 0.5 N was reached and then was further lowered at a speed of 1.3 mm/min. Flexural strength, flexural strain at maximum force, and flexural modulus of elasticity were determined. The results were reported as an average value from measurements of at least five specimens.

2.5. Microstructural observation

The morphology of the SCFs was examined using a scanning electron microscope (SEM) (JEOL, JSM 520-2AE). The operating voltage used was 10 kV. Selected, fractured specimens obtained after mechanical property measurement were cut about 2 mm below the fractured surface and mounted on aluminum stubs. Prior to examination, the surface of the specimen was coated with a thin layer of gold under vacuum for 3 min in order to

improve the conductivity of and to prevent electron charging on the surface.

3. Results and discussion

The foaming process of a starch batter inside a hot mold can be divided into several steps. First, the temperature of the batter increases to the point at which the temperature is equal or above the gelatinization temperature of the starch. Upon gelatinization, the viscosity of the starch increases dramatically. This causes starch to turn from an easy-flowing slurry into a thick paste. The high temperature of the batter mixture leads to a rapid evaporation of the entrapped water to evaporate, which, in turn, causes the paste to expand dramatically. The starch paste must have sufficient strength in order to withstand the force of the rapid expansion without a permanent, structural damage. Once the starch paste fills up the mold (as a result of the rapid expansion), the viscosity of the starch paste further increase expeditiously to stabilize the foam structure and to prevent the molding to collapse as residual water further evaporates. The evaporated water vents out around the edge of the mold. In the final and longest step of the baking process, the starch foam gradually dries to obtain the foam having the residual moisture content of ca. 2–4% (Shogren, Lawton, Doane, & Tiefenbacher, 1998c).

Because starch is naturally hydrophilic, the derived pure starch-based foams (SFs) are hygroscopic materials (Glenn & Hsu, 1997). Fig. 1 illustrates the effects of storage RH and storage time on the moisture content of the SFs prepared. For a fixed RH level, the moisture content was found to be constant after 3 days of conditioning. For a fixed storage time, the moisture content was found to increase with increasing storage RH level. Specifically, the resulting moisture content after 3 days of conditioning at 11.3, 32.8, 42.3, 52.9, and 75.3% RH was found to be ca. 3.6, 7.9, 9.4,

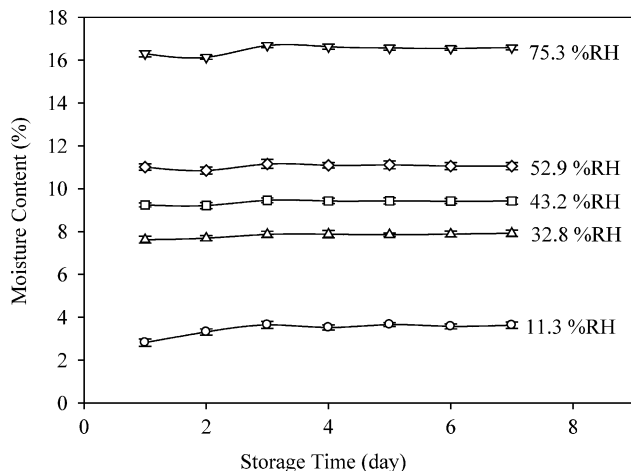


Fig. 1. Effects of storage relative humidity and storage time on moisture content for pure starch foams, which were conditioned at (○) 11.3, (△) 32.8, (□) 42.3, (◇) 52.9, and (▽) 75.3% RH.

11.1, and 16.6%, respectively. Lourdin, Coignard, Bizot, and Colonna (1997) reported the moisture content for cast potato starch films after 7 days of conditioning at 33, 43, 52, 57, and 70% RH to be ca. 11.1, 13.5, 13.9, 14.8, and 17.8%, respectively. Obviously, our results seem to agree fairly well with those reported by these authors.

The effects of moisture content and fiber content (with no preferred orientation) on the flexural strength, flexural strain at maximum force, and flexural modulus of elasticity for SFs, jute-reinforced, and flax-reinforced SCFs are illustrated in Figs. 2–4, respectively. It should be noted that the aspect ratios for jute and flax fibers used in these investigations were around 28.8 and 28.5, respectively. In Fig. 2, the flexural strength for all of the foams prepared exhibited a similar dependence on the moisture content, in that it increased with increasing moisture content up to around 7–9% where the flexural strength reached a maximum and then decreased with further increase in the moisture content. Since the SFs and the SCFs were conditioned at five RH levels, the results shown in Fig. 2 clearly suggested that the moisture uptake, for a given RH, for the SCFs was found to decrease from that of the SFs with

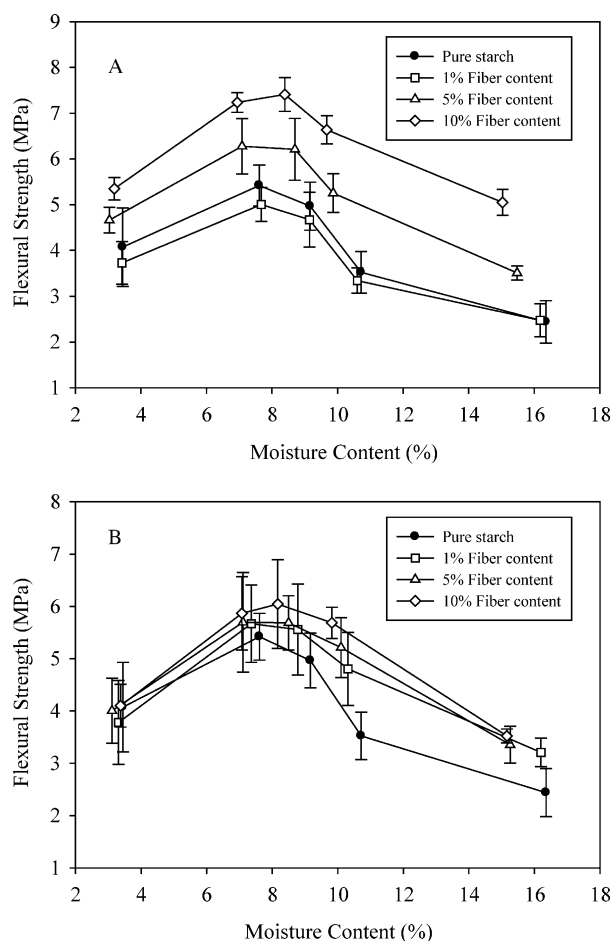


Fig. 2. Effects of moisture content and fiber content on the flexural strength for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

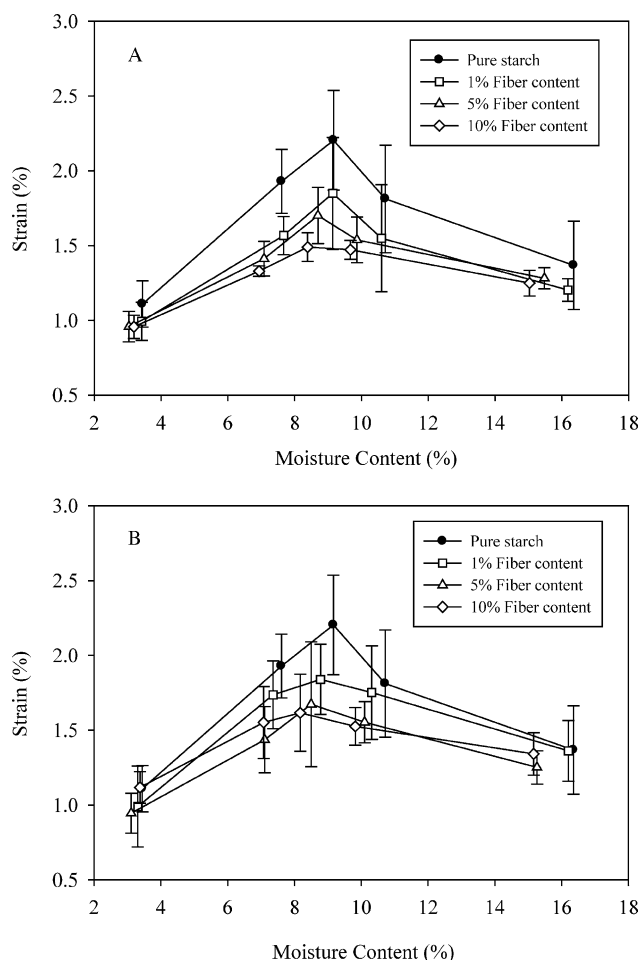


Fig. 3. Effects of moisture content and fiber content on the flexural strain at maximum force for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

increasing fiber content. For a given moisture content, most of the SCFs exhibited greater flexural strength than did the SFs, with an exception on the 1 wt% jute-reinforced SCFs which exhibited lower flexural strength than the SFs at all fiber contents. The flexural strength for SCFs was found to increase with increasing fiber content.

In Fig. 3, the flexural strain at maximum force for all of the foams prepared exhibited a similar dependence on the moisture content, in that it increased with increasing moisture content up to around 8–9% where the flexural strain at maximum force reached a maximum and then decreased with further increase in the moisture content. For a fixed moisture content, all of the SCFs prepared exhibited lower strain at maximum force than did the SFs. With an increase in the fiber content, the flexural strain at maximum force was found to decrease monotonically. In Fig. 4, the flexural modulus of elasticity for all of the foams prepared showed a similar dependence on the moisture content, in that it monotonically decreased with increasing moisture content. An exception to the observed trend was observed for the 10 wt% flax-reinforced SCFs in which flexural modulus of elasticity initially increased, reached

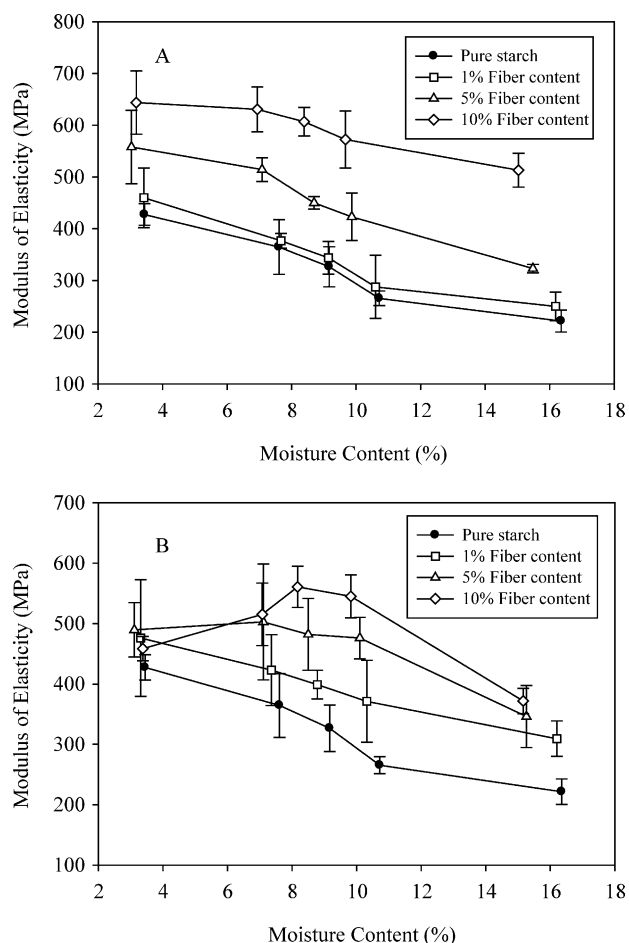


Fig. 4. Effects of moisture content and fiber content on the flexural modulus of elasticity for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

a maximum value at the moisture content of around 8–9%, then decreased with further increase in the moisture content. For a fixed moisture content, most of the SCFs showed greater flexural modulus of elasticity than did the SFs and the flexural modulus of elasticity was found to increase with increasing fiber content.

Similar results were also reported in the literature (Glenn, Orts, & Nobes, 2001b). Glenn, Orts, and Nobes (2001b) studied the effect of moisture content on mechanical properties of starch-based panels having the moisture contents of 3.4, 7.5, 11.1, and 14.5%. For flexural strength and flexural strain at maximum force, the starch-based panels behaved very similarly to what was observed in the present study in that these property values increased initially with increasing moisture content, reached a maximum at the moisture content of around 7.5%, and then decreased with further increase in the moisture content. For flexural modulus of elasticity, they reported that it decreased monotonically with increasing moisture content, which is in general accord with our results. In the food literature, it is known that a brittle-to-ductile transition or the loss of crispness for starch-based foams occurs at the moisture

content of about 9% (Li, Kloeppel, & Hsieh, 1998). This could explain the observed maxima in both the flexural strength and the flexural modulus of SFs and SCFs at the moisture content of around 7–9%.

Possible explanation for the low values of the observed flexural strength and flexural strain at maximum force for SFs and SCFs at low and high moisture contents may be the brittleness of the materials at low moisture contents and the plasticizing effects due to the presence of large amount of absorbed moisture at high moisture contents (Dufresne et al., 1999; Lourdin et al., 1997; Shogren, Lawton, Tiefenbacher, & Chen, 1998b). The observed monotonous decrease in the flexural modulus of elasticity with increasing moisture content may be explained mainly based on the plasticizing effect, in which the increasing amount of absorbed moisture caused the foams to be less stiff (Dufresne et al., 1999; Glenn, Orts, & Nobes, 2001b).

The results also showed that, generally, addition of jute or flax fibers was responsible for the much improvement in the flexural strength and flexural modulus of elasticity for SCFs as compared with SFs, at the expense of the flexural strain at maximum force. However, 1 wt% jute-reinforced SCFs showed lower flexural strength than the SFs at all fiber contents. This may be because, at low fiber contents, short fibers added may act as defects which can promote crack propagation, hence reducing the strength (Lodha & Netravali, 2002).

The reasons for the much improvement in the flexural strength and flexural modulus of elasticity for SCFs due to the addition of jute or flax fibers as compared with SFs may be two-fold. The first is the reinforcing effect. The scanning electron micrographs of fracture surface for both jute- and flax-reinforced SCFs, as shown in Fig. 5, reveal that interfacial interaction between fibers and starch matrix

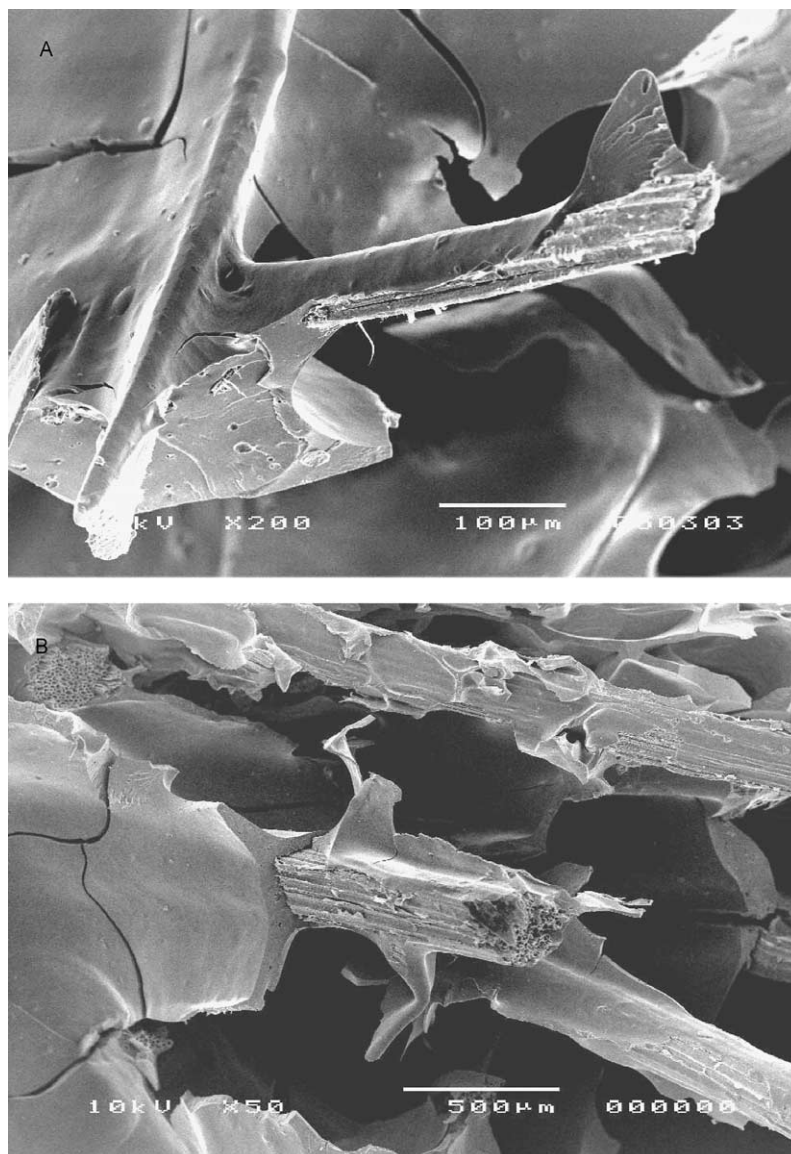


Fig. 5. Scanning electron micrographs for fracture surfaces of (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

Table 3
Effect of fiber content on densities of jute- and flax-reinforced starch-based composite foams (reported with the standard deviation in parentheses)

Foam type	Fiber aspect ratio (<i>L/D</i>)	Fiber content (%)	Average density (g/cm ³)
SF ^a	–	–	0.214 (0.014)
Jute-reinforced SCF ^b	28.8	1	0.223 (0.018)
		5	0.276 (0.017)
		10	0.323 (0.015)
Flax-reinforced SCF ^b	28.5	1	0.248 (0.017)
		5	0.295 (0.019)
		10	0.336 (0.021)

^a Denotes starch-based foam.

^b Denotes starch-based composite foam.

was very good, most likely a result of both having similar chemical functional groups. Good interfacial interaction suggests that stress can transfer from the starch matrix to the fibers very effectively during deformation, hence giving rise to higher strength (Averous et al., 2001; Lodha et al., 2002). Secondly, the presence of fibers in a batter formulation is responsible for an increase in the viscosity of the batter.

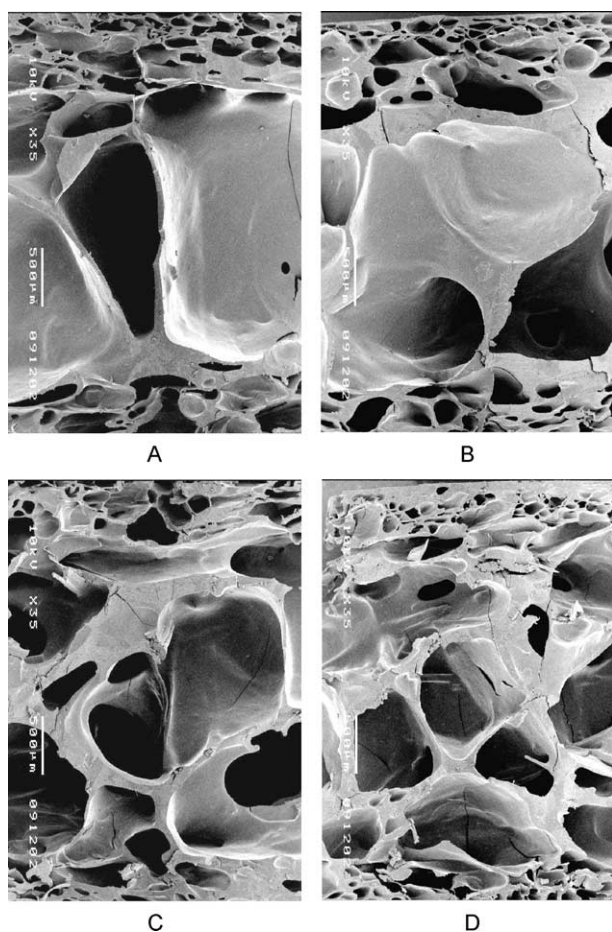


Fig. 6. Scanning electron micrographs for cross-sections of (A) pure starch-based foam and jute-reinforced starch-based composite foams at (B) 1, (C) 5, and (D) 10% fiber content, respectively.

The increase in the viscosity causes the batter to be less expandable, giving rise to smaller average cell size, thicker cell wall, and higher density (Shogren et al., 2002). According to Table 3, the density for all of SCFs prepared was greater than that for SFs and the density for SCFs increased with increasing fiber content. Figs. 6 and 7 verify that the average cell size for all of SCFs prepared was smaller than that for SFs and the average cell size for SCFs decreased with increasing fiber content. As a result of the smaller average cell size, thicker cell wall, higher density, and the presence of reinforcing fibers, fiber-reinforced SCFs appeared to exhibit much improvement in the flexural strength and flexural modulus of elasticity over the SFs, at the expense of the flexural strain at maximum force (Anderson et al., 1999; Shogren, Lawton, Doane, & Tiefenbacher, 1998c).

Between jute- and flax-reinforced SCFs, jute-reinforced SCFs showed much greater flexural strength than flax-reinforced ones did (see Fig. 2). The discrepancy may lie on the differences in the specific surface area and the stiffness (i.e. the tensile modulus) between these two fibers. On the first account, it is evident from Table 1 that the average diameter and the average density of flax fibers were greater than those of jute fibers (see Table 1). In the same weight

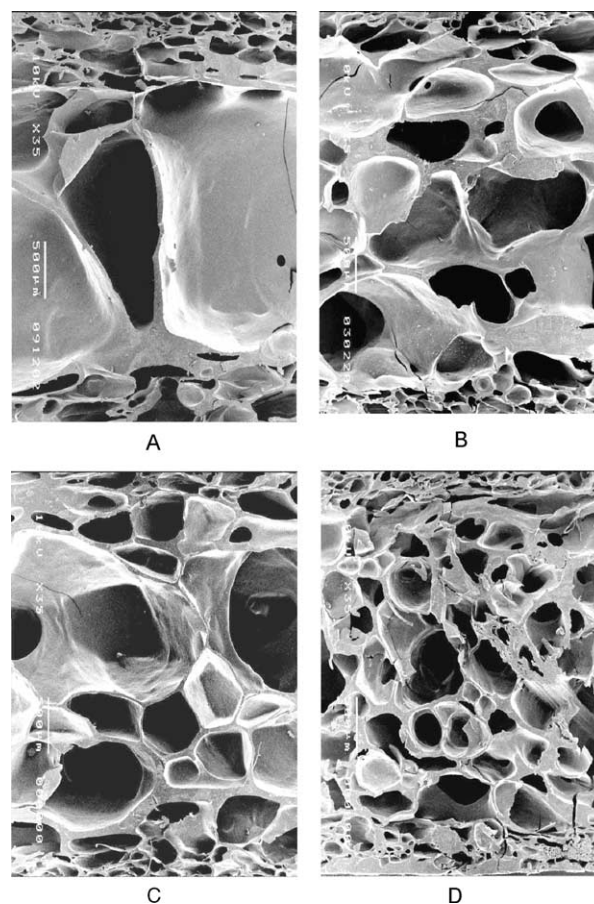


Fig. 7. Scanning electron micrographs for cross-sections of (A) pure starch-based foam and flax-reinforced starch-based composite foams at (B) 1, (C) 5, and (D) 10% fiber content, respectively.

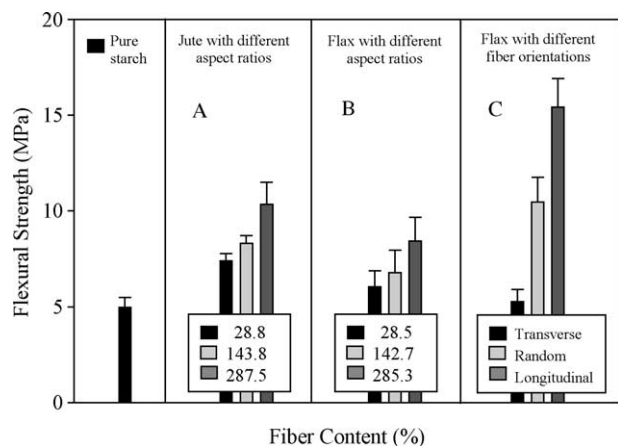


Fig. 8. Effects of fiber aspect ratio and fiber orientation on flexural strength for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

proportion and fiber aspect ratio, it is logical that jute fibers had higher specific surface area, hence more surface area to interact with the starch matrix, than flax fibers did. Good fiber-matrix interaction translates into the ability for the matrix to transfer stress to the reinforcing fibers very effectively, hence imparting higher strength to the composites (Anderson et al., 1999; Karnani et al., 1997). On the other account, the fact that the jute fibers used were stiffer (i.e. greater tensile modulus) than flax fibers did suggests that jute fibers could provide better reinforcing effect than flax fibers could (provided that all other factors are essentially similar). Since it has been verified that adhesion between both fibers and the starch matrix is good, addition of jute fibers, which exhibited higher specific surface area and was stiffer than flax fibers did, in a starch batter should result in SCFs having higher strength than those with addition of flax fibers.

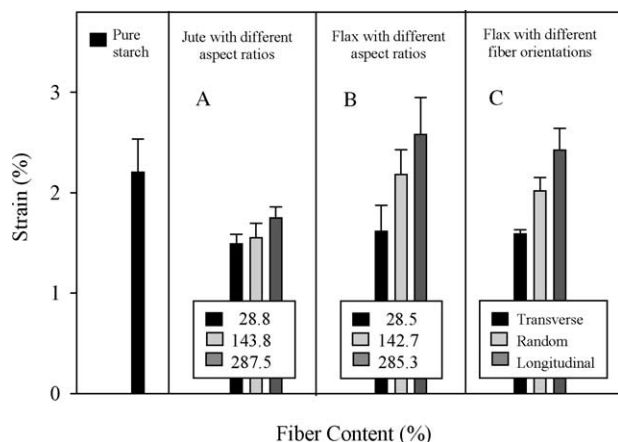


Fig. 9. Effects of fiber aspect ratio and fiber orientation on flexural strain at maximum force for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

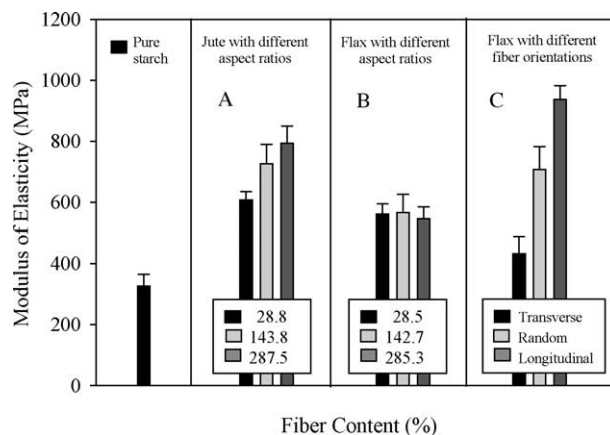


Fig. 10. Effects of fiber aspect ratio and fiber orientation on flexural modulus of elasticity for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

The effects of fiber aspect ratio and fiber orientation on flexural strength, flexural strain at maximum force, and flexural modulus of elasticity for both jute- and flax-reinforced SCFs are illustrated in Figs. 8–10, respectively. As evidently shown in Figs. 8A, B, 10A and B, the flexural strength and the flexural modulus of elasticity for both jute- and flax-reinforced SCFs were found to increase with increasing fiber aspect ratio (reported at a fixed fiber content of 10 wt%). Possible explanation may lie on the fact that fibers of high aspect ratios should provide large surface area that can interact with the starch matrix, resulting in an increased efficiency for stress transfer from the matrix to the fiber, hence higher strength and stiffness (Albano et al., 2001; Averous et al., 2001; Lodha et al., 2002). Furthermore, addition of fibers of high aspect ratios could attribute to an increase in the viscosity of the starch-based batters, as a result of the fibrous network formation (Shogren et al., 2002). This led to increased density, decreased average cell size, and thicker cell wall of the SCFs reinforced with jute or flax fibers of increasing aspect ratio (see Table 4 and Fig. 11).

Table 4
Effect of fiber aspect ratio on densities of jute- and flax-reinforced SCFs (reported with the standard deviations in parentheses)

Foam type	Fiber content (%)	Fiber aspect ratio (L/D)	Average density (g/cm ³)
SF ^a	–	–	0.214 (0.014)
Jute-reinforced SCF ^b	10	28.8	0.322 (0.024)
		143.8	0.341 (0.012)
		287.5	0.360 (0.028)
Flax-reinforced SCF ^b	10	28.5	0.339 (0.031)
		142.7	0.344 (0.007)
		285.3	0.347 (0.012)

^a Denotes starch-based foam.

^b Denotes SCFs.

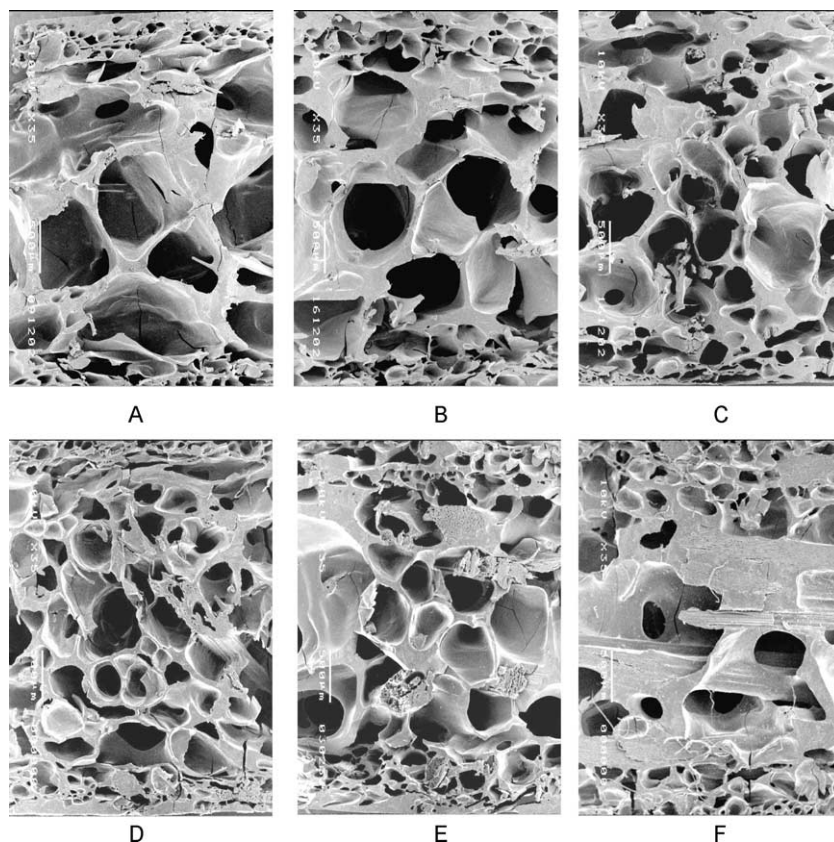


Fig. 11. Scanning electron micrographs for cross-sections of starch-based composite foams reinforced with jute fibers having fiber aspect ratio of (A) 28.75, (B) 143.76, and (C) 287.52, respectively, and with flax fibers having fiber aspect ratio of (D) 28.53, (E) 142.67, and (F) 285.33, respectively.

The flexural strain at maximum force for both jute- and flax-reinforced SCFs was shown to increase with increasing fiber aspect ratio (see Fig. 9), due possibly to the increased strength of the materials (Shogren, Lawton, Tiefenbacher, & Chen, 1998b). Interestingly, SCFs reinforced with flax fibers having the aspect ratios of 142.67 and 285.33 showed significant improvement in the flexibility over that of the SFs. This may be a result of the high percentage of elongation that flax fibers exhibited (see Table 1), which makes flax-reinforced SCFs being able to sustain large

deformation elastically before rupture. On the contrary, all of the jute-reinforced SCFs exhibited lower flexural strain at maximum force than SFs did. This may be a direct result of the low percentage of elongation that jute fibers exhibited (see Table 1), which limits the critical flexural strain that jute-reinforced SCFs could withstand.

The effect of fiber orientation on flexural strength, flexural strain at maximum force, and flexural modulus of elasticity for SCFs was only performed for flax-reinforced SCFs, since flax fibers were long enough to be

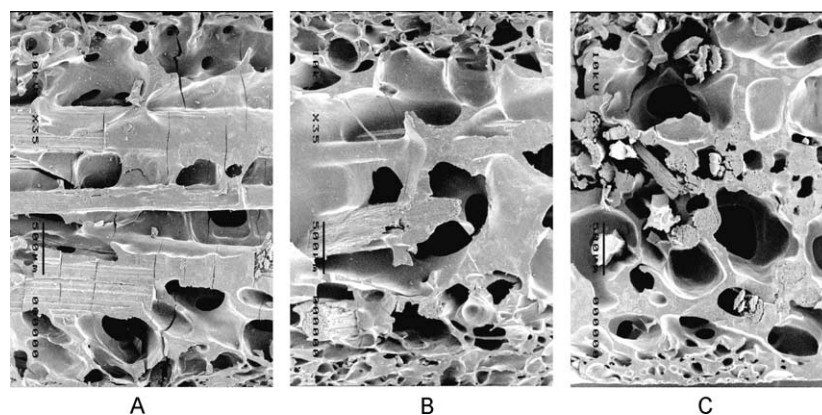


Fig. 12. Scanning electron micrographs for cross-sections of starch-based composite foams reinforced with flax fibers oriented (A) transversely, (B) randomly, and (C) longitudinally.

oriented unidirectionally. As evidently shown in Figs. 8C and 10C, SCFs reinforced with flax fibers being oriented longitudinally showed the most improvement in the flexural strength and the flexural modulus of elasticity over the SFs (i.e. almost three-fold increase in the property values), followed, respectively, by SCFs reinforced with fibers being oriented randomly and transversely. This is because, in SCFs with longitudinal fiber arrangement, the majority of the fibers were oriented perpendicularly to the crack propagation direction, hence crack propagation was retarded by the presence of these fibers (Clemons, Caulfield, & Giacomini, 1999). According to Fig. 9C, only SCFs reinforced with fibers being oriented in the longitudinal direction showed comparable flexural strain at maximum force to that of the SFs, while SCFs reinforced with fibers being oriented in the transverse direction showed the lowest value. Fig. 12 shows scanning electron micrographs for cross-sections of SCFs reinforced with flax fibers being oriented transversely, randomly, and longitudinally.

4. Conclusions

Jute- and flax-reinforced SCFs with varying fiber content, fiber aspect ratio, and fiber orientation were successfully prepared. An increase in the storage relative humidity level resulted in an increase in the moisture content of the pure starch-based foams (SFs). The moisture content of the pure starch foams was constant after three days of conditioning. Addition of jute or flax fibers to the starch-based foams resulted in the much improvement in the flexural strength and the flexural modulus of elasticity, at the expense of the flexural strain at maximum force. The optimal moisture content which resulted in the maximum values of the flexural strength and the flexural strain at maximum force for both jute- and flax-reinforced SCFs was observed between 8 and 10%, depending on the batter formulations. The reinforcing effect of the fibers was found to increase with increasing fiber content and fiber aspect ratio, with jute fibers providing more improvement to the flexural strength of the SCFs than flax fibers did. Lastly, the SCF reinforced with flax fibers being oriented in the longitudinal direction showed a dramatic improvement in the flexural strength and the flexural modulus of elasticity over the SFs.

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