CHAPTER IV
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 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 to 4% (Shogren *et al.*, 1998c).

Because starch is naturally hydrophilic, the derived pure starch-based foams (SFs) are hygroscopic materials (Glenn *et al.*, 1997). Figure 4.1 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, 11.1, and 16.6%, respectively. Lourdin and co-workers (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.

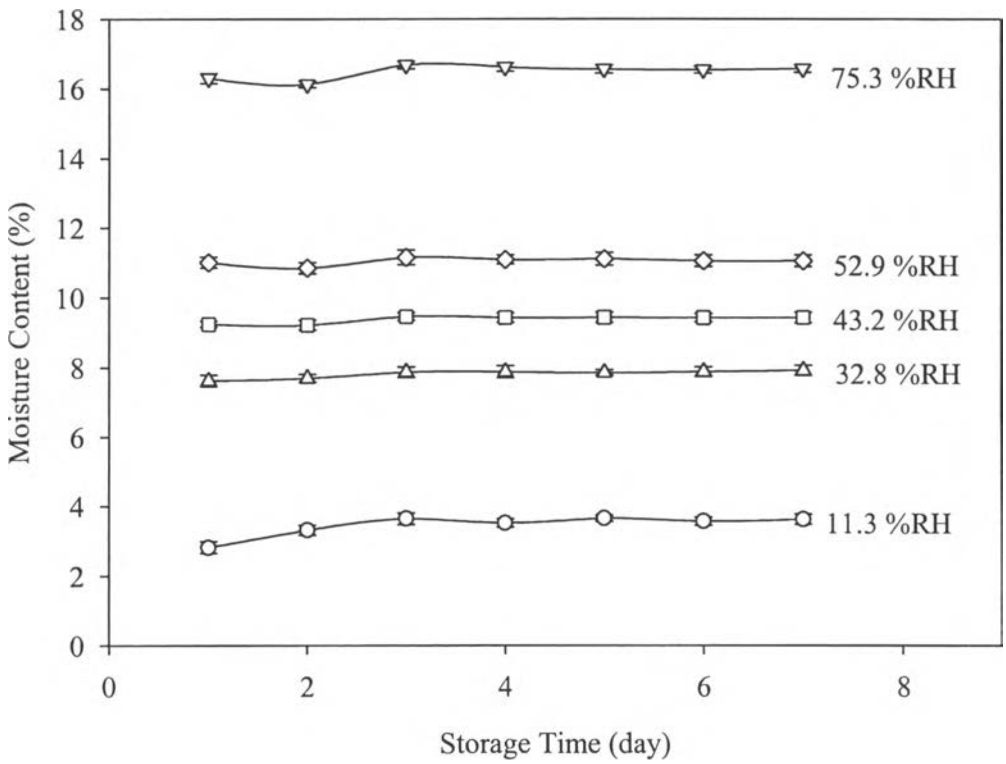
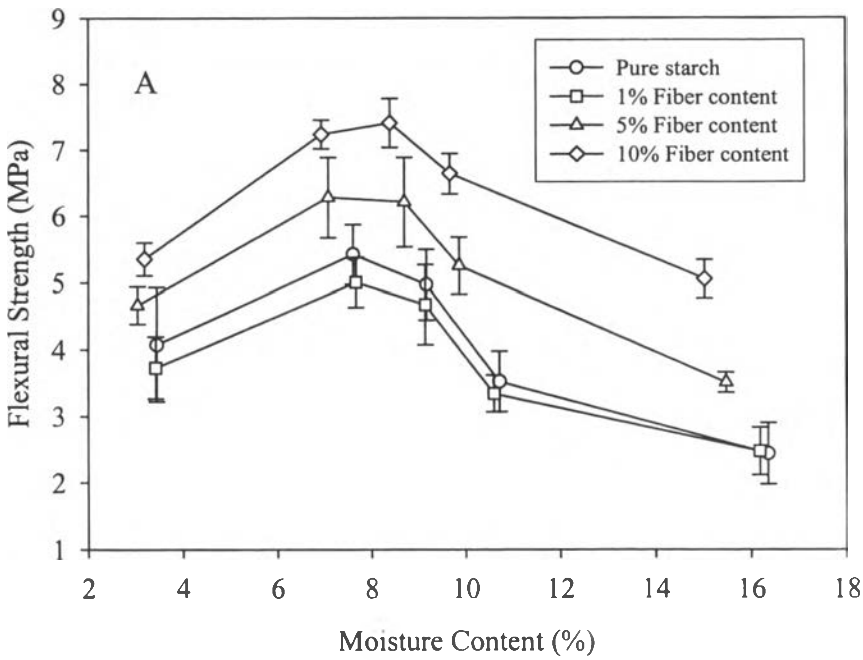


Figure 4.1 Effects of storage relative humidity and storage time on moisture content for pure starch foams, which were conditioned at (○) 11.3, (△) 32.8, (□) 42.3, (◇) 52.9, and (▽) 75.3 %RH.

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 Figures 4.2, 4.3, and 4.4, respectively. In Figure 4.2, 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 to 9% where the flexural strength reached a maximum and then decreased with further increase in the moisture content. For a fixed 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.



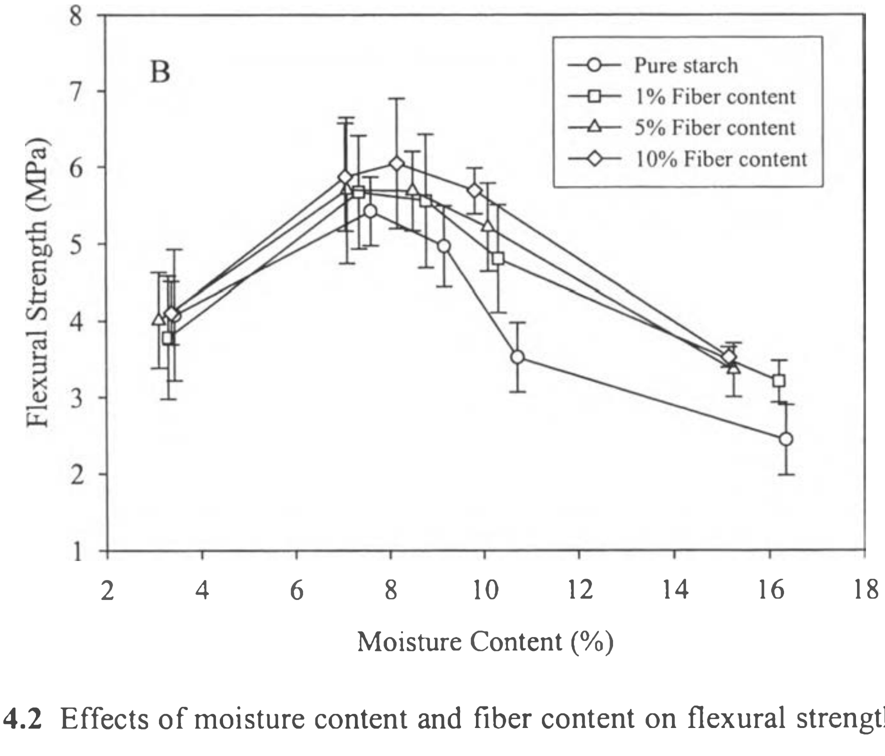


Figure 4.2 Effects of moisture content and fiber content on flexural strength for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

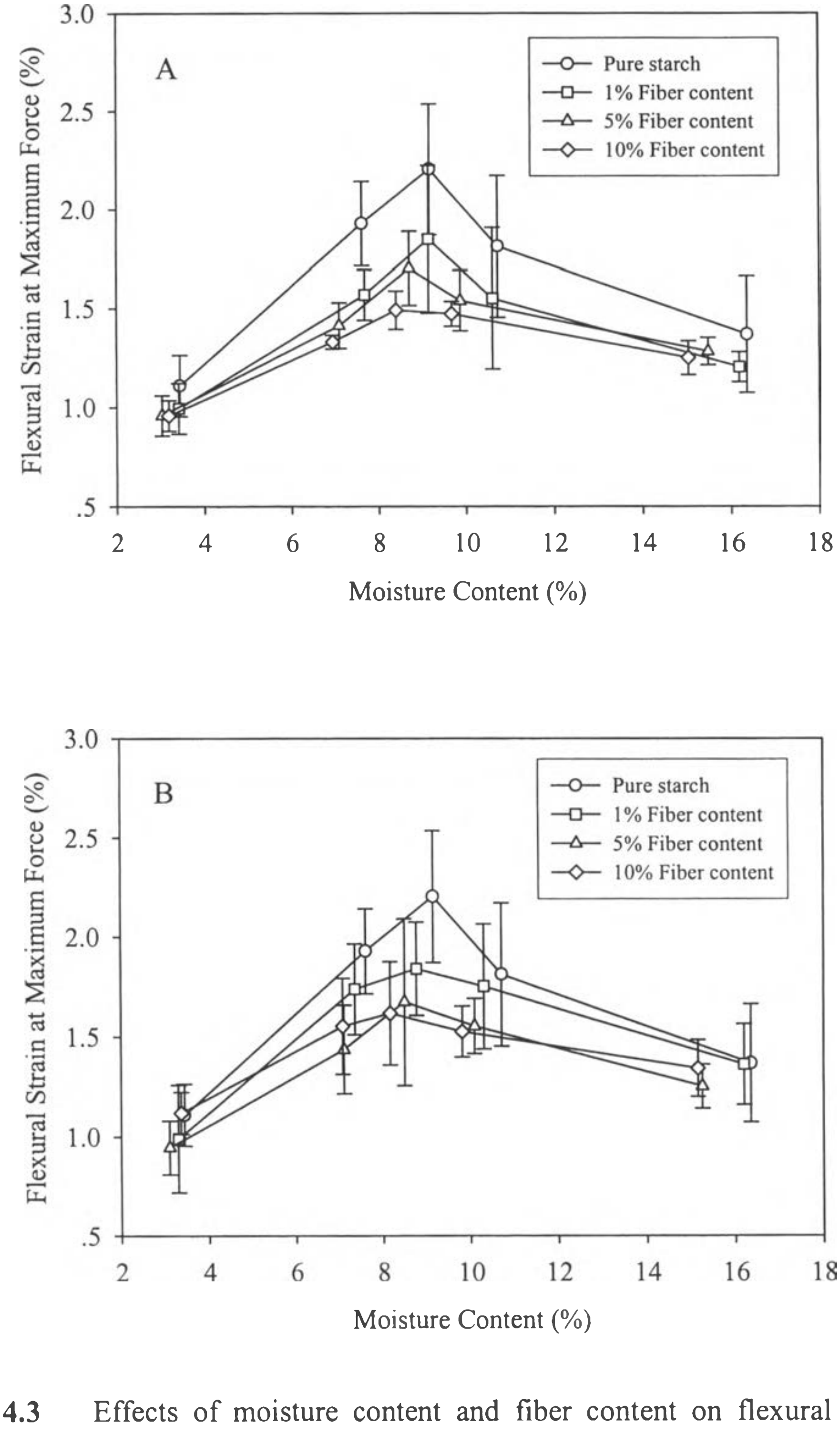
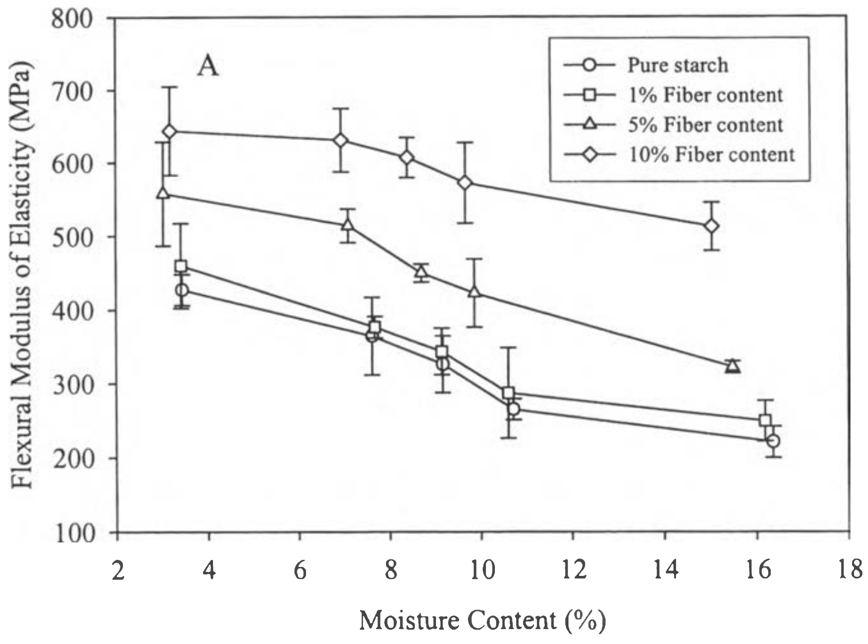


Figure 4.3 Effects of moisture content and fiber content on flexural strain at maximum force for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.



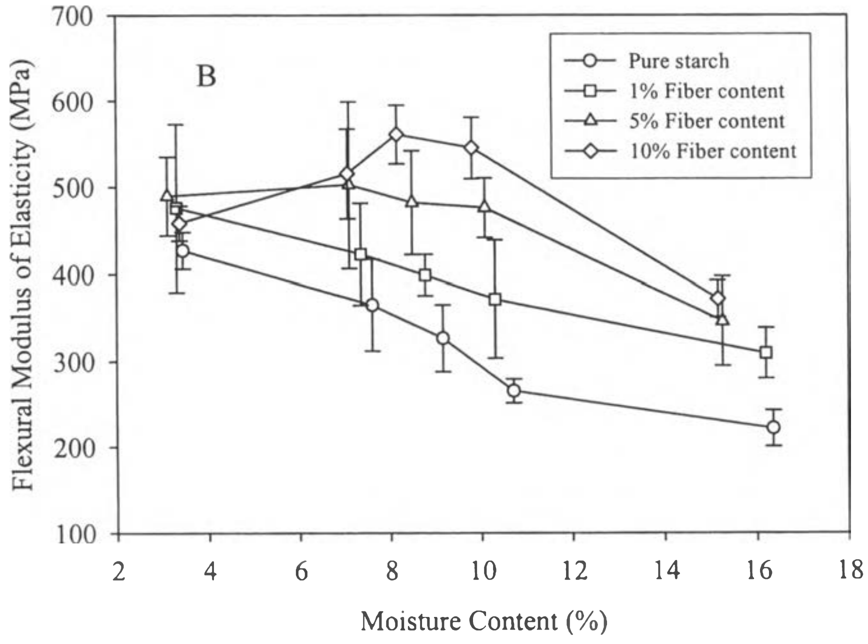


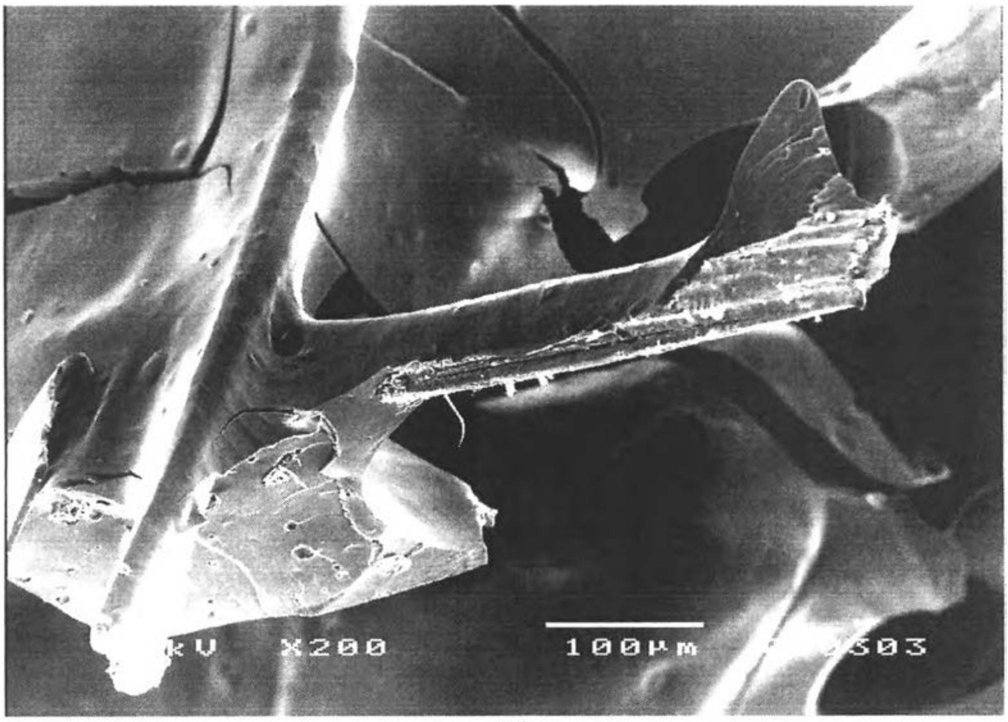
Figure 4.4 Effects of moisture content and fiber content on flexural modulus of elasticity for (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

In Figure 4.3, 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 to 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 Figure 4.4, 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 a maximum value at the moisture content of around 8 to 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.

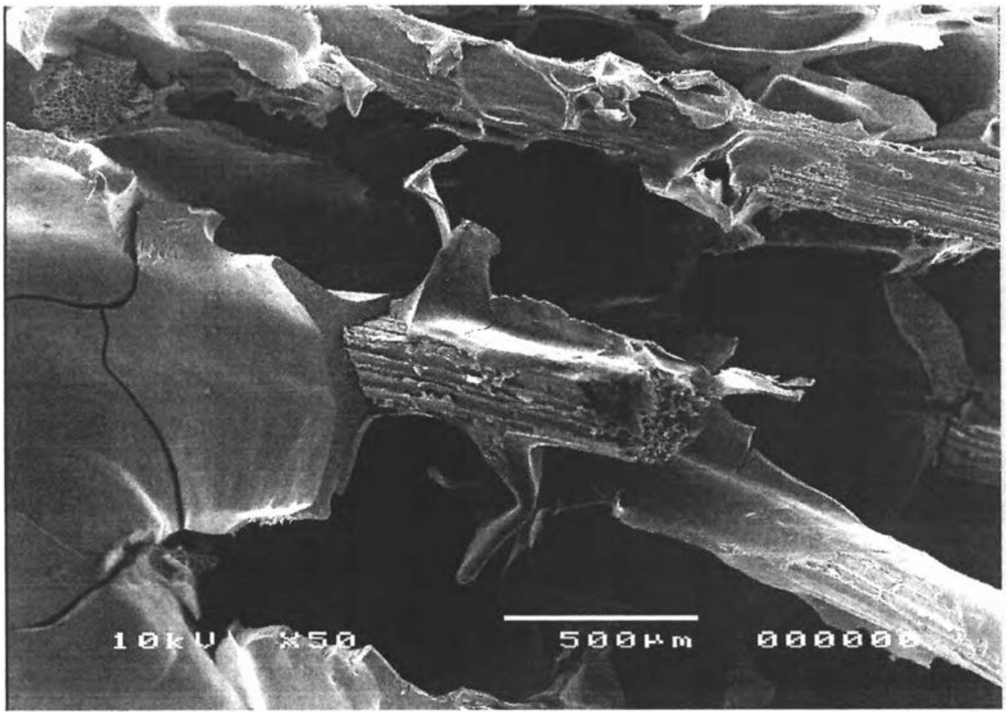
Similar results were also reported in the literature (Glenn *et al.*, 2001b). Glenn *et al.* (2001b) studied the effect of moisture content on mechanical properties of starch-based panels having the moisture contents of 3.4, 7.5, 11.1, and 14.5%. For flexural strength and flexural strain at maximum force, 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 accordance with our results.

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 (Lourdin *et al.*, 1997; Shogren *et al.*, 1998b; Dufresne *et al.*, 1999). 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 *et al.*, 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 *et al.*, 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 Figure 4.5, reveal that interfacial interaction between fibers and starch matrix was very good, most likely a result of both having similar chemical functional groups. This effect might be enhanced by surface roughness of the fibers due to mechanical interlocking. 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. The increase in the viscosity causes the batter to be less expandable, less coalescence of small voids giving rise to smaller average cell size, thicker cell wall, and higher density (Shogren *et al.*, 2002). According to Table 4.1, the density for all of SCFs prepared was greater than that for SFs and the density for SCFs increased with increasing fiber content. Figures 4.6 and 4.7 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 (Shogren *et al.*, 1998c; Anderson *et al.*, 1999).



(A)



(B)

Figure 4.5 Scanning electron micrographs for fracture surfaces of (A) jute-reinforced and (B) flax-reinforced starch-based composite foams.

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Table 4.1 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 (L/D)	Fiber content (%)	Average density (g/cm^3)
SF[1]	-	-	0.214 (0.015)
Jute-reinforced SCF[2]	28.75	1	0.223 (0.020)
		5	0.276 (0.019)
		10	0.323 (0.017)
Flax-reinforced SCF[2]	28.53	1	0.248 (0.019)
		5	0.295 (0.021)
		10	0.336 (0.023)

[1] denotes starch-based foam

[2] denotes starch-based composite foam

Between jute- and flax-reinforced SCFs, jute-reinforced SCFs showed much greater flexural strength than flax-reinforced ones did (see Figure 4.2). 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 3.1 that the average diameter and the average density of flax fibers were greater than those of jute fibers (see Table 3.1). In the same weight 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 (Karnani *et al.*, 1997; Albano *et al.*, 2001). 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.

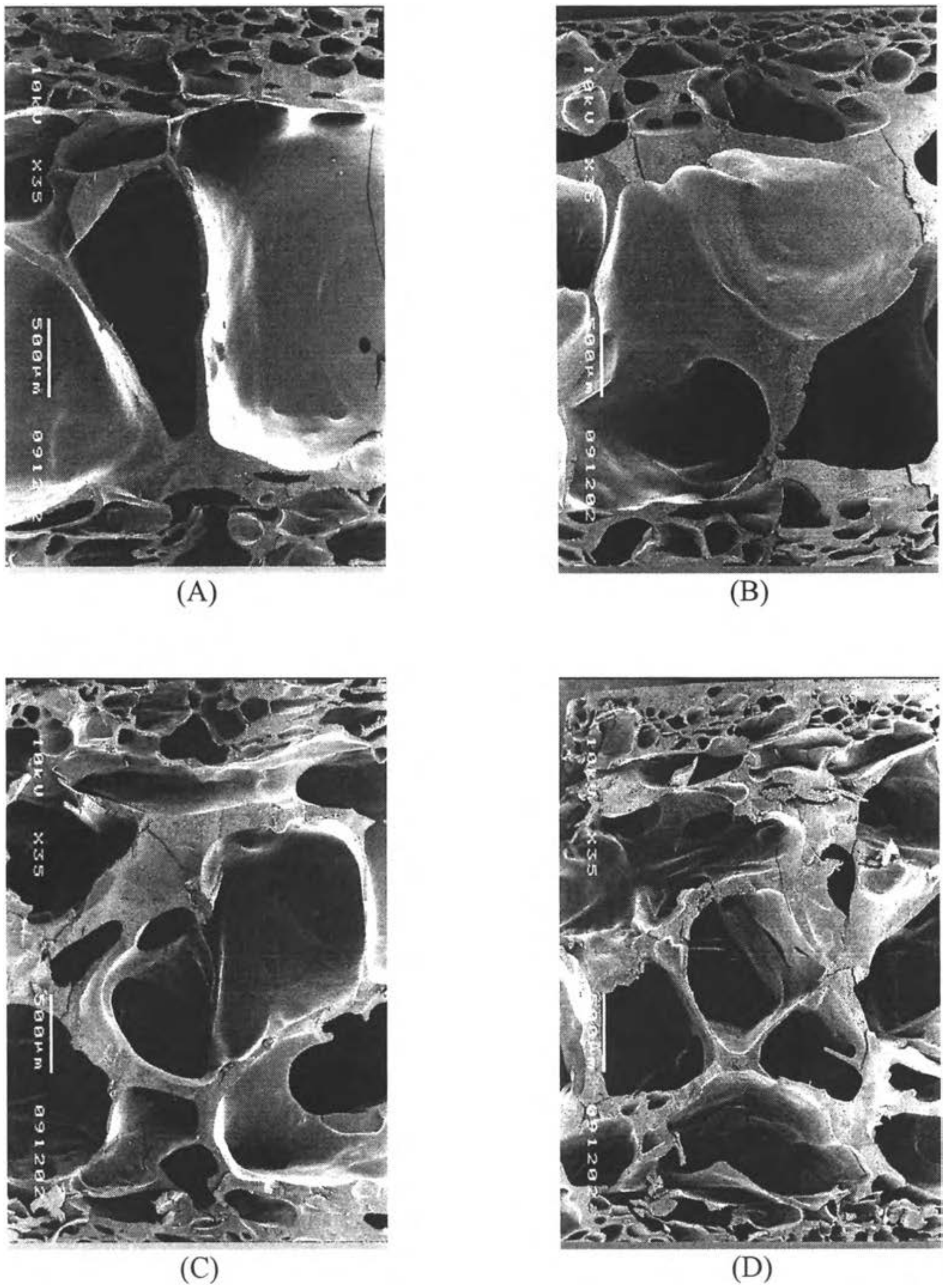


Figure 4.6 Scanning electron micrographs for cross-sections of (A) pure starch-based foam and jute-reinforced starch-based composites foams at (B) 1, (C) 5, and (D) 10% fiber content, respectively.

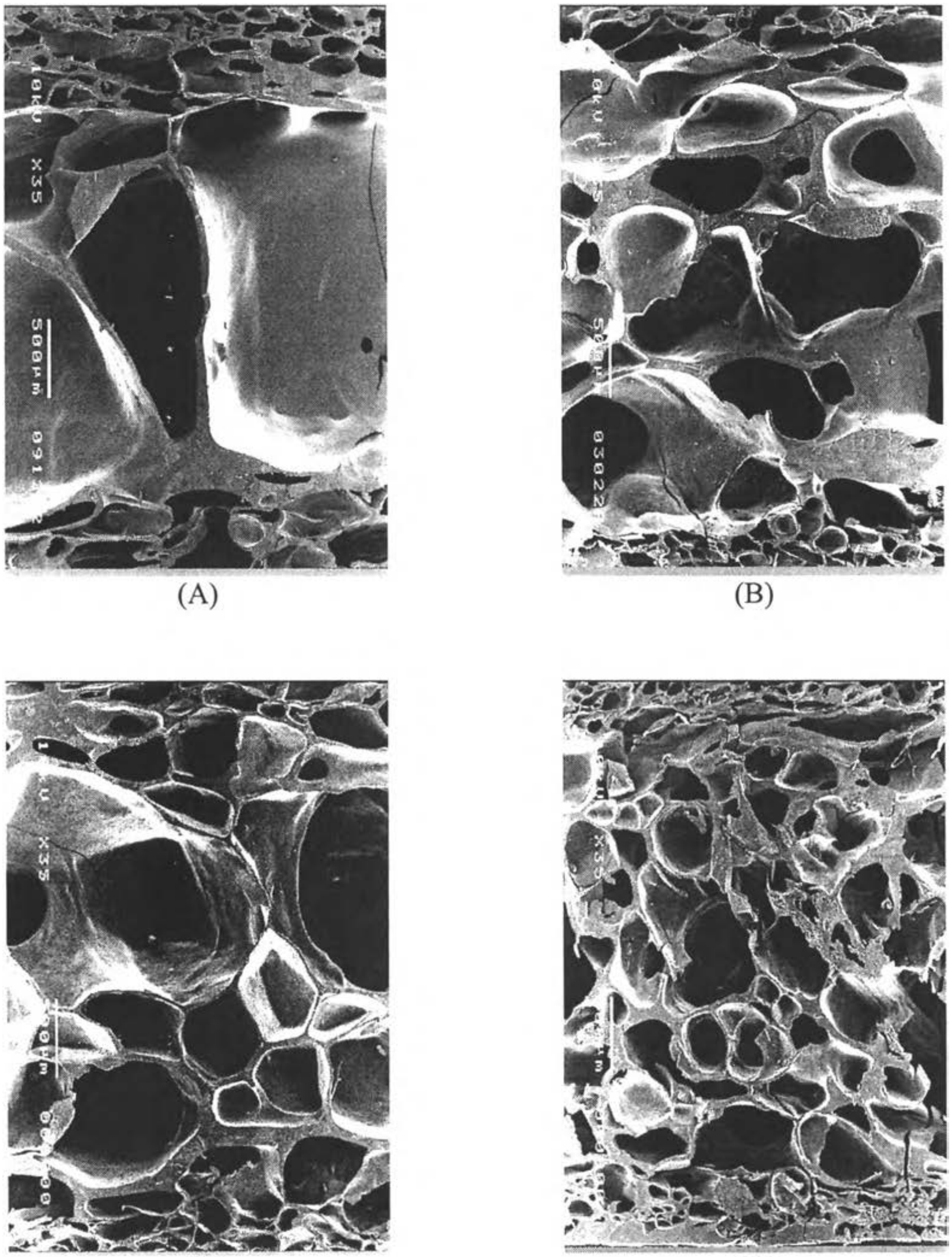


Figure 4.7 Scanning electron micrographs for cross-sections of (A) pure starch-based foam and flax-reinforced starch-based composites foams at (B) 1, (C) 5, and (D) 10% fiber content, respectively.

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 Figures 4.8, 4.9, and 4.10, respectively. As evidently shown in Figures 4.8A, 4.8B, 4.10A and 4.10B, 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 4.2 and Figure 4.11).

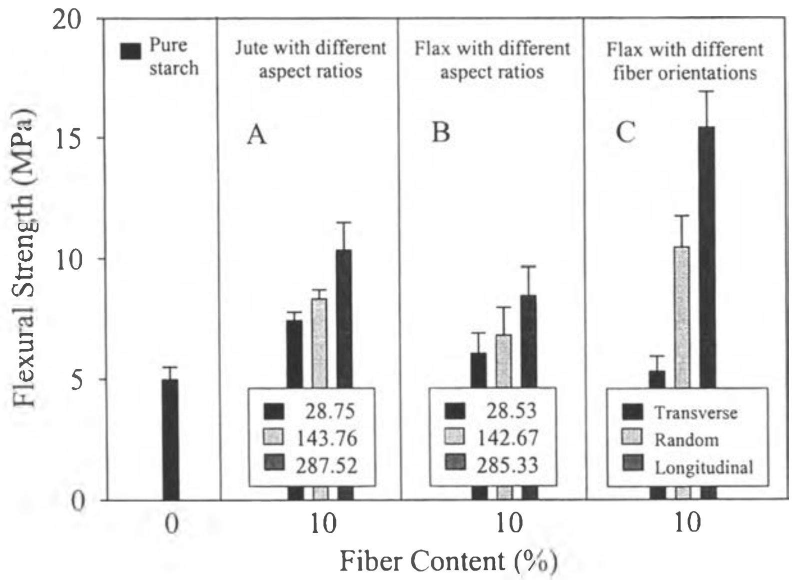


Figure 4.8 Effects of fiber aspect ratio and fiber orientation on flexural strength for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

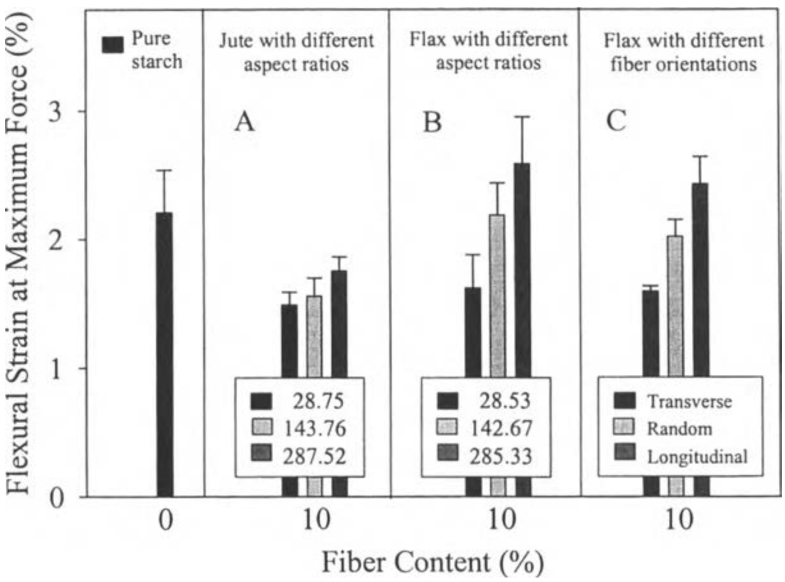


Figure 4.9 Effects of fiber aspect ratio and fiber orientation on flexural strain at maximum force for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

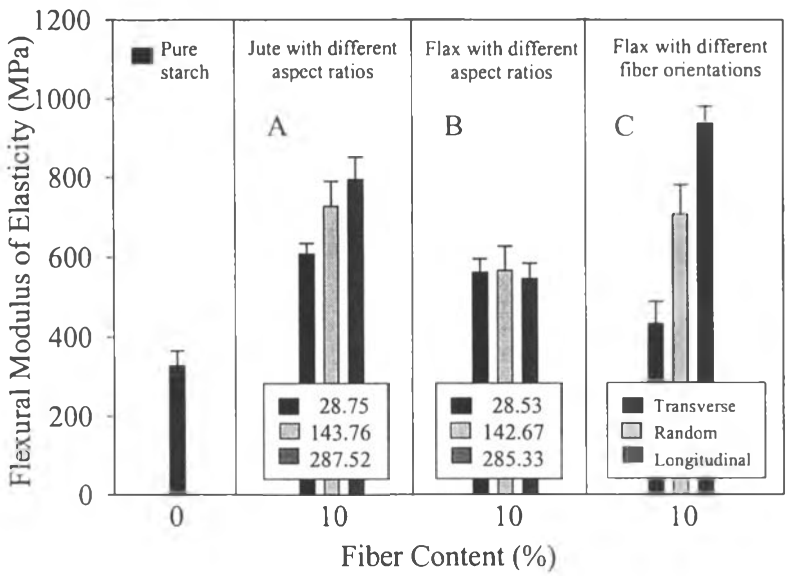


Figure 4.10 Effects of fiber aspect ratio and fiber orientation on flexural modulus of elasticity for starch-based composite foams reinforced with (A) jute and (B) flax fibers of different aspect ratios, and for (C) starch-based composite foams reinforced with flax fibers of different fiber orientations.

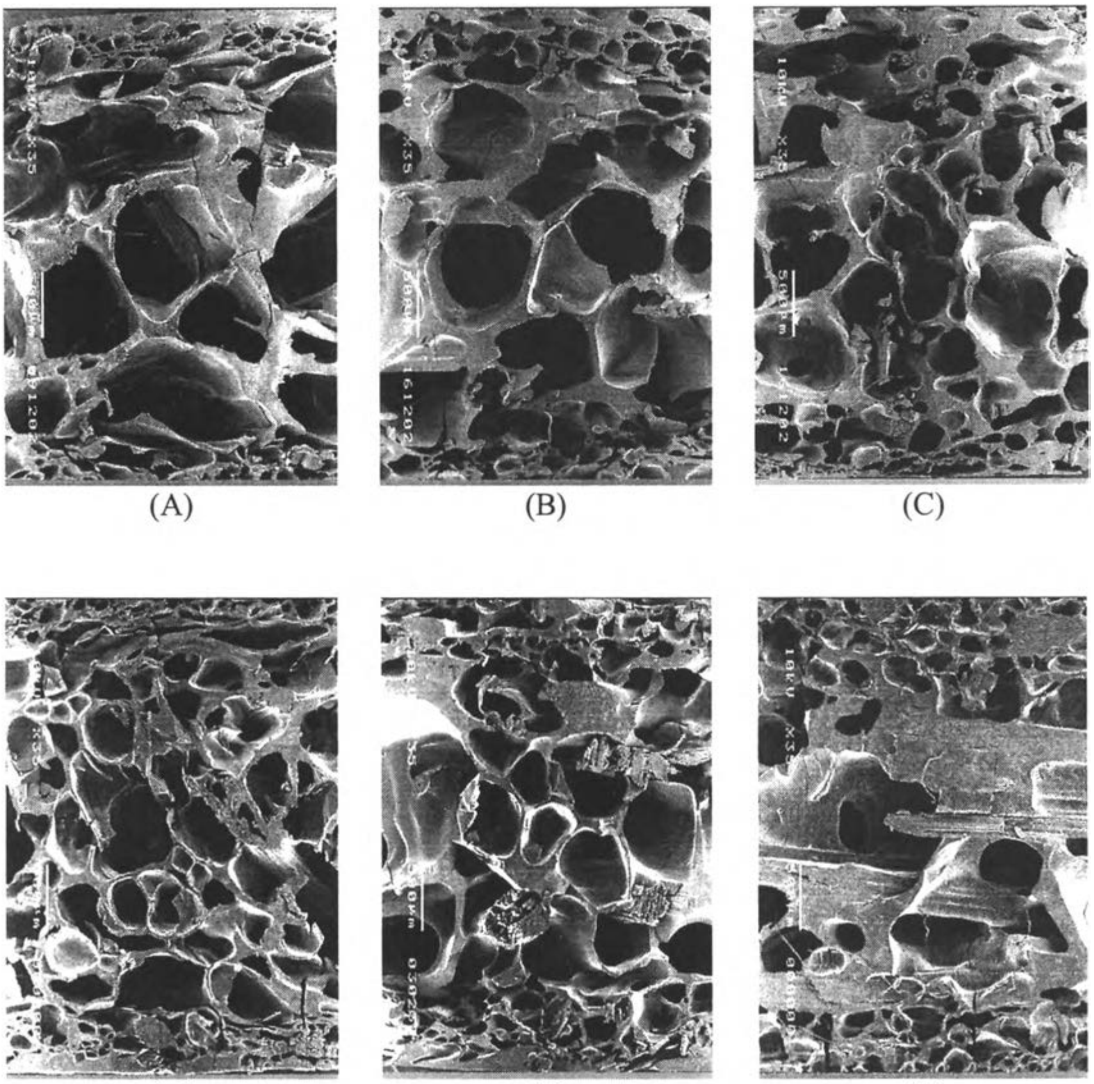
Table 4.2 Effect of fiber aspect ratio on densities of jute- and flax-reinforced starch-based composite foams (reported with the standard deviations in parentheses)

Foam Type	Fiber content (%)	Fiber aspect ratio (L/D)	Average density (g/cm^3)
SF[1]	-	-	0.214 (0.014)
Jute-reinforced SCF[2]	10	28.75	0.322 (0.024)
		143.76	0.341 (0.012)
		287.52	0.360 (0.028)
Flax-reinforced SCF[2]	10	28.53	0.339 (0.030)
		142.67	0.344 (0.007)
		285.33	0.347 (0.012)

[1] denotes starch-based foam

[2] denotes starch-based composite foam

The flexural strain at maximum force for both jute- and flax-reinforced SCFs was shown to increase with increasing fiber aspect ratio (see Figure 4.9), due possibly to the increased strength of the materials (Shogren *et al.*, 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 3.1), 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 3.1), which limits the critical flexural strain that jute-reinforced SCFs could withstand. However, at too low fiber aspect ratio, different percent elongation of the fibers does not affect flexural strain at maximum force of SCFs.

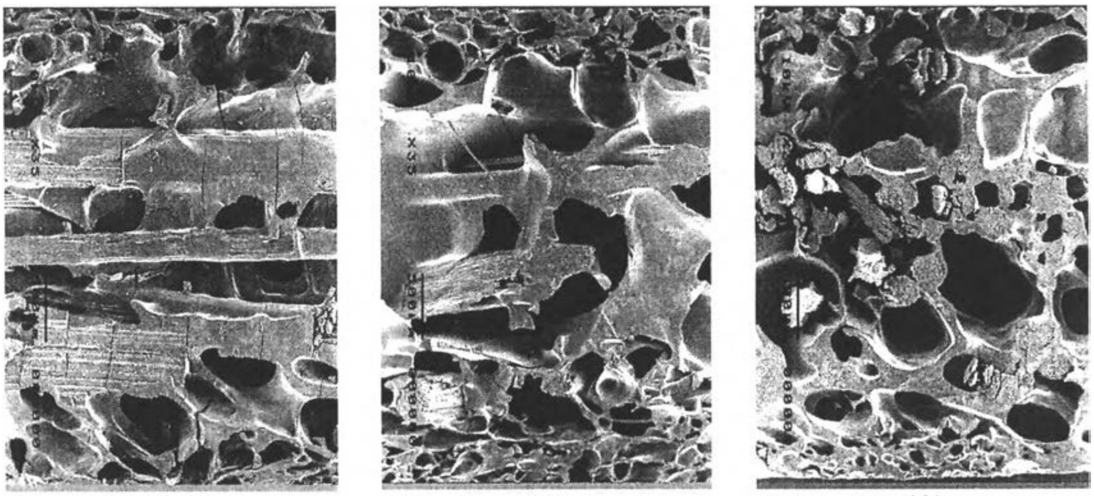


(A) (B) (C)

(D) (E) (F)

Figure 4.11 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 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 oriented unidirectionally. As evidently shown in Figures 4.8C and 4.10C, 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 *et al.*, 1999). According to Figure 4.9C, 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. Figure 4.12 shows scanning electron micrographs for cross-sections of SCFs reinforced with flax fibers being oriented transversely, randomly, and longitudinally.



(A) (B) (C)

Figure 4.12 Scanning electron micrographs for cross-sections of starch-based composite foams reinforced with flax fibers oriented (A) transversely, (B) randomly, and (C) longitudinally.