4.1.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is a powerful imaging tool used to analyze the physical structure of a substance under various magnifications. In this study, SEM analyses was carried out on different categories of treated fibers and composites to determine several features to support the results obtained by other experimental analysis done such as the tensile test and the biodegradable test. The fibers used in this study as part of the composite blends was first subjected to a bleaching process and then treated with NaOH to remove all of its natural and artificial impurities adhering to the fibers.

Pure samples (Different fibers)

Figure 4.37 to 4.44 illustrates the SEM images of the different fibers used that have been bleached and mercerized under various magnifications.

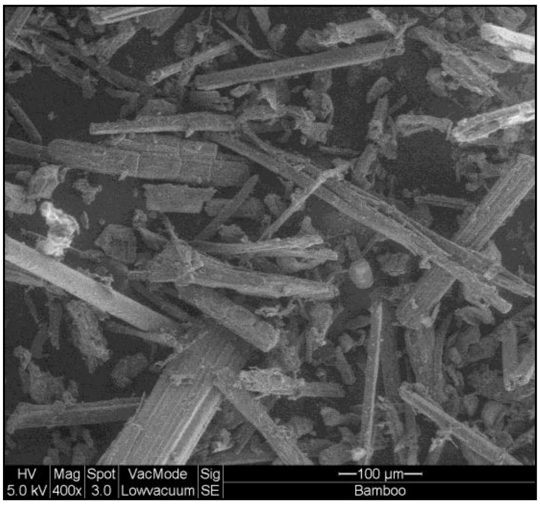

Fig. 4.37 SEM image of treated bamboo fibers at 400x magnification

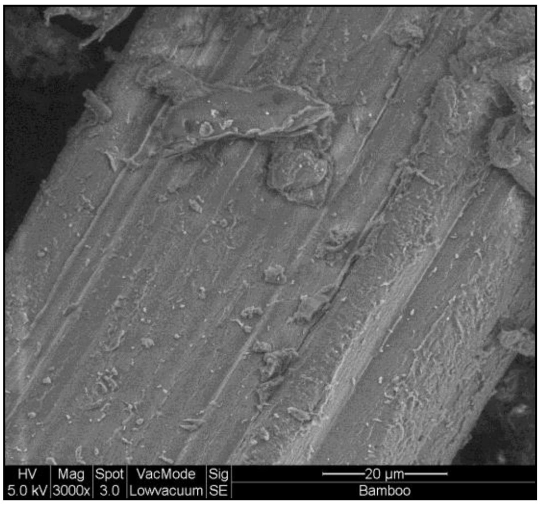

Fig. 4.38 SEM image of treated bamboo fibers at 3000x magnification

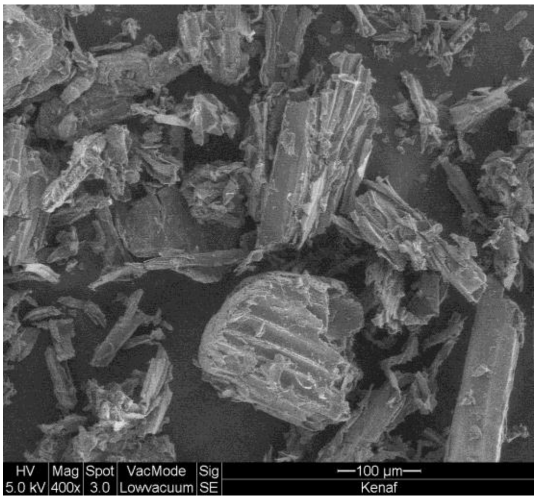

Fig 4.39 SEM image of treated kenaf fibers at 400x magnification

Fig. 4.40 SEM image of treated kenaf fibers at 3000x magnification

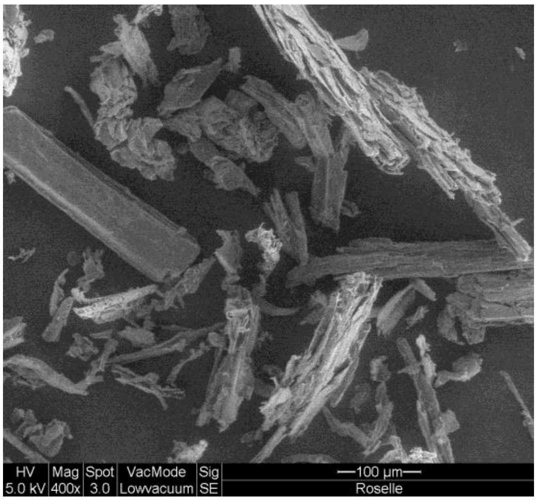

Fig. 4.41 SEM image of treated roselle fibers at 400x magnification

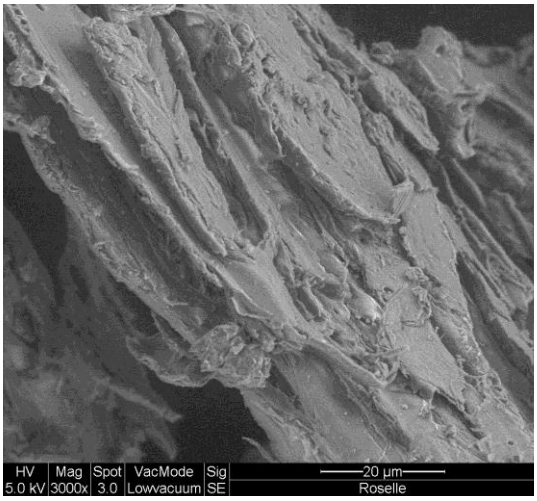

Fig. 4.42 SEM image of treated roselle fibers at 3000x magnification

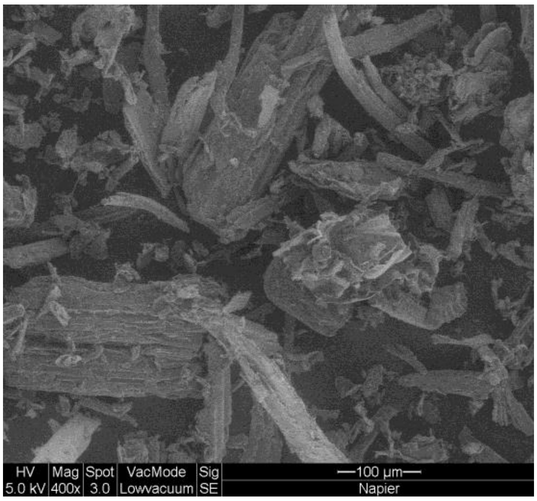

Fig. 4.43 SEM image of treated Napier fibers at 400x magnification

Fig. 4.44 SEM image of treated Napier fibers at 3000x magnification

From the SEM images of the treated fibers, generally it can be observed that the bleaching and mercerization process done on the fibers resulted in the breaking down of the fiber bundles into smaller bundles that measures around 100-250 μm in length and 40-60 μm in width. This fibrillation process increases the effective surface area available for contact with the polymer matrix. The removal of impurities such as wax, oil, hemicellulose, and pectin covering the external surface of the fiber cell wall by the surface treatment process can also be seen from the 3000x magnification images of the different fibers. Rough surface topography was formed upon removal of these impurities. The surface roughness of the treated fibers will result in better mechanical interlocking between fibers and the polymer matrix thus producing stronger interfacial adhesion. The removal of the impurities also produces an increased amount of cellulose exposed on the fiber surface, and this increases the number of possible reaction sites for the hydroxyl groups of fibers and matrix to interact and form linkages (Rosa, et al., 2009). Despite of the positive attributes distinguished from the images that will contribute to better interfacial interlocking and subsequently producing a stronger blend when the fibers are incorporated into the PVA/starch matrix, one feature that can cause the composite to have lower mechanical strength is that the treated fibers illustrated in the images varies in shapes and sizes. Basically, if the fibers are of non-uniform sizes and shapes, then the capability of the fiber to support stress in the composites are lower (Rosa, et al., 2009). The non-uniformity of the treated fibers can contribute to poor interfacial adhesion between fiber and matrix, which results in a less efficient stress transfer between matrix and fiber. Inefficient stress transfer between fibers and matrix will lead to breakage of the fibers during the mechanical testing. This phenomenon can be seen in the SEM images of the fractured samples of the PVA/starch/fibers composites.

The SEM analysis was also carried out to determine on how well the dispersion of the treated fibers in the polymer matrix, the adhesion between fiber and matrix and to detect the presence of any micro defects on the surface of the composites. A comparison was done on the images of pure PVA, PVA/different starches and PVA/different starches/different fibers composites.

PVA/different starches composites

SEM images of the pure PVA, PVA/starch composites are shown in Figures 4.45 and Figures 4.46 to 4.51.

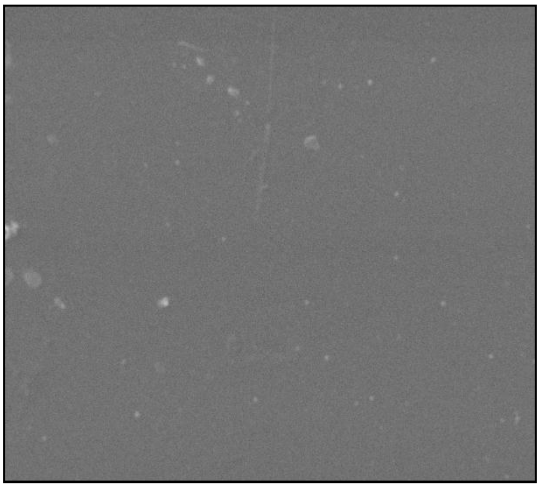

Fig. 4.45 SEM image of pure PVA film at 500x magnification

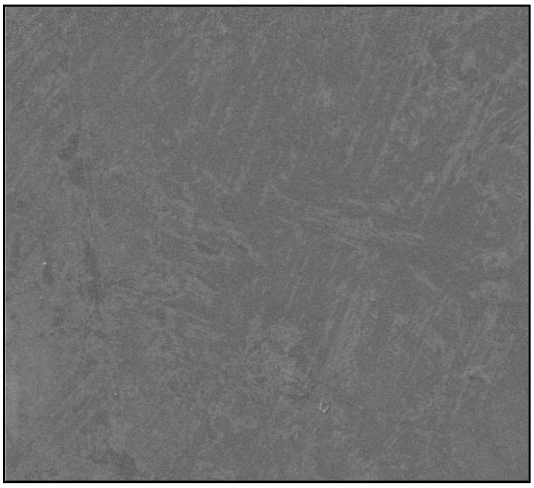

Fig. 4.46 SEM image of PVA/1TS film at 500x magnification

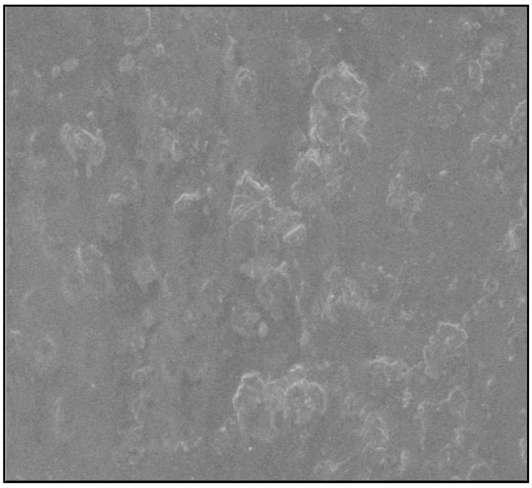

Fig. 4.47 SEM image of PVA/3TS film at 500x magnification

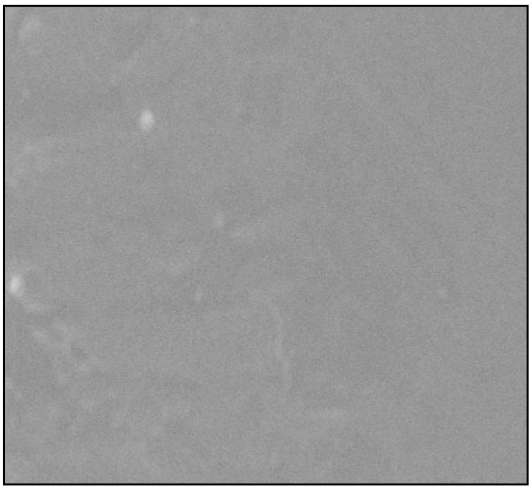

Fig. 4.48 SEM image of PVA/1RS film at 500x magnification

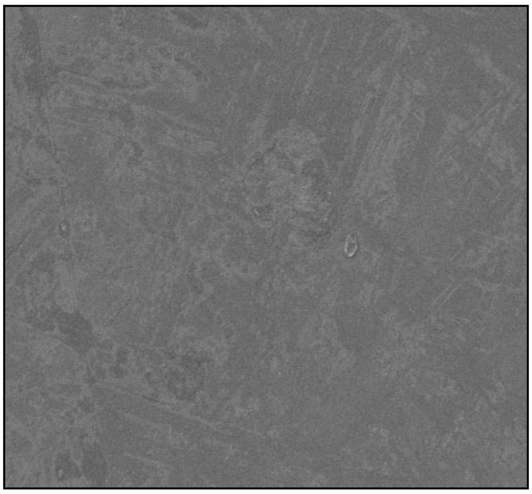

Fig. 4.49 SEM image of PVA/3RS film at 500x magnification

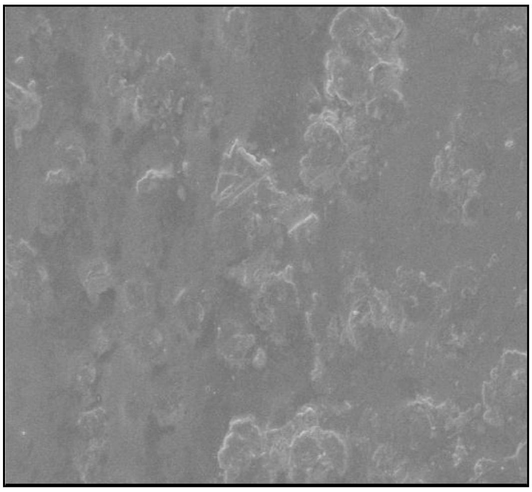

Fig 4.50 SEM image of PVA/1SS film at 500x magnification

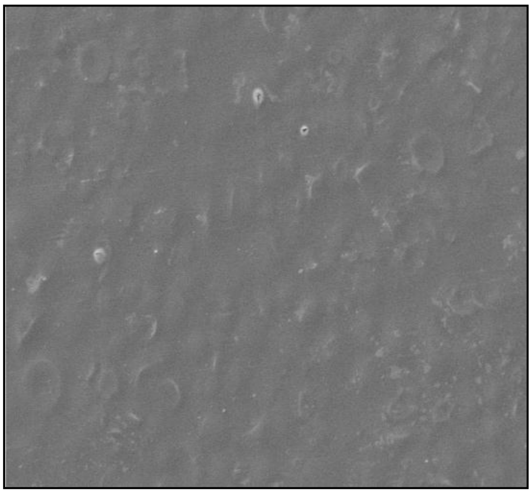

Fig. 4.51 SEM image of PVA/3SS film at 500x magnification

As shown in Figure 4.45, the surface of pure PVA film appeared to be smooth and compact. The photomicrographs of PVA blended with 1g of different starches show a rather smooth surface except for the composite of PVA blended with 1g of sago starch. Images in Figure 4.46 and 4.48 show a rather clean surface except for a few scratches produced when the films were removed from the casting plate. The smooth surface structure may indicate that the miscibility and compatibility in each component of the PVA/starch blends were increased. The image of PVA blended with 1g of sago starch composite revealed in what appears to be groups of bumps that resembles agglomeration of sago starch granules on the film's surface. Sago starch has by far one of the biggest granule sizes of all the commercial starches. The average sago starch granule is around 30 μm. Due to the large granule size and partly because of partial gelatinization of the sago starch; several agglomerations of sago starch were clearly seen on the surface of the film. Agglomeration of starches will lead to a decrease in the tensile properties of the composite formed because these agglomerations may not be fully covered by the PVA phase and eventually will lead to the weakening of the interfacial adhesion between starch and the polymer matrix. These findings conforms the result obtained from the tensile test where the PVA blended with 1g of sago starch shows the lowest tensile strength value. For the composite PVA blended with 3g of different starches, the blend of PVA/3TS and PVA/3SS showed rough surfaces where agglomerations of starch granules can be clearly seen on the surface of the films. This may be due to partial gelatinization of the starches and the blends mentioned have lower tensile properties. Gelatinization of starch is a method to enhance the interfacial affinity between starch and polymer matrix. Starch is gelatinized to disintegrate granules and overcome the strong interaction of starch molecules in the presence of water and polymer matrix, which leads to well dispersion of the starches. If only partial gelatinization occurred then the ungelatinized starches would form clusters and this leads to the lowering of mechanical properties of the composites.

PVA/different starches/different fibers composites

Figure 4.52 to 4.75 show the SEM images of PVA blended with 1g of different starches and blended with different concentration of different fibers.

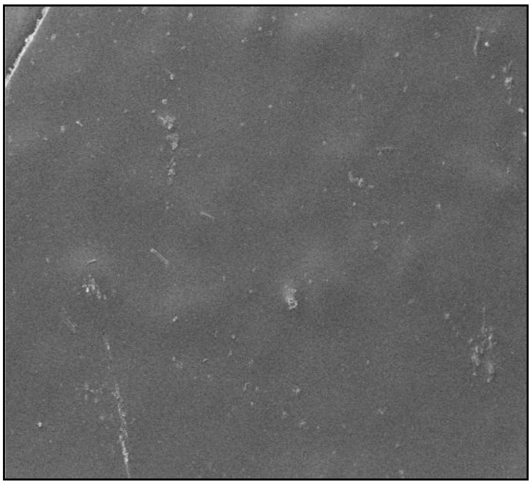

Fig. 4.52 SEM image of PVA/1TS/1BB at 25x magnification

Fig. 4.53 SEM image of PVA/1TS/3BB at 25x magnification

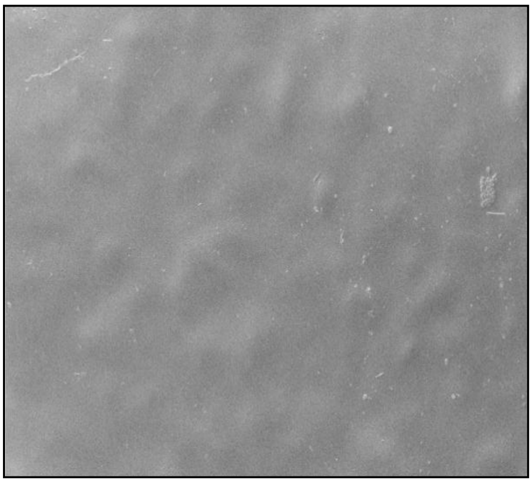

Fig. 4.54 SEM image of PVA/1TS/1KF at 25x magnification

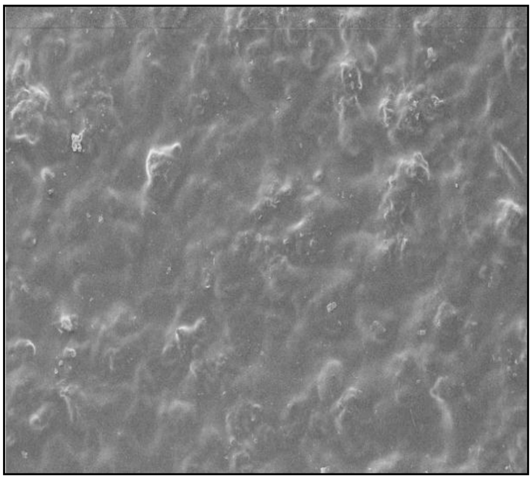

Fig 4.55 SEM image of PVA/1TS/3KF at 25x magnification

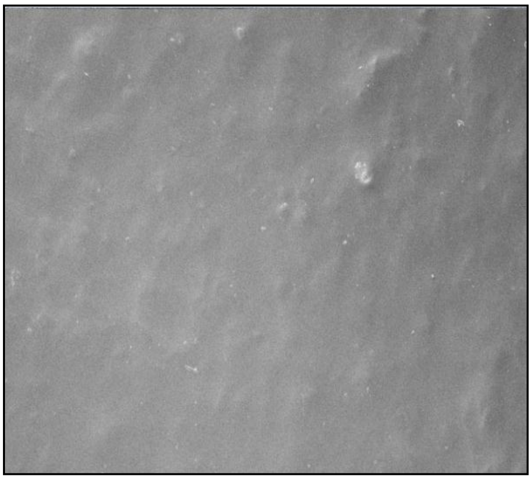

Fig. 4.56 SEM image of PVA/1TS/1ROS at 25x magnification

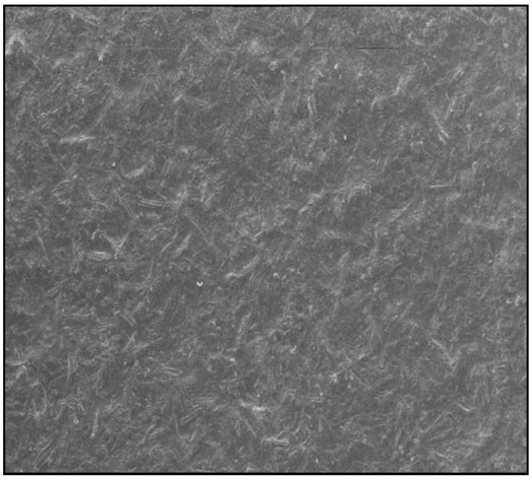

Fig. 4.57 SEM image of PVA/1TS/3ROS at 25x magnification

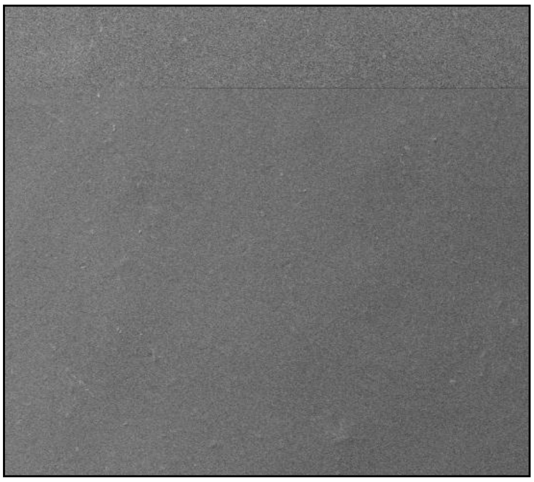

Fig. 4.58 SEM image of PVA/1TS/1NP at 25x magnification

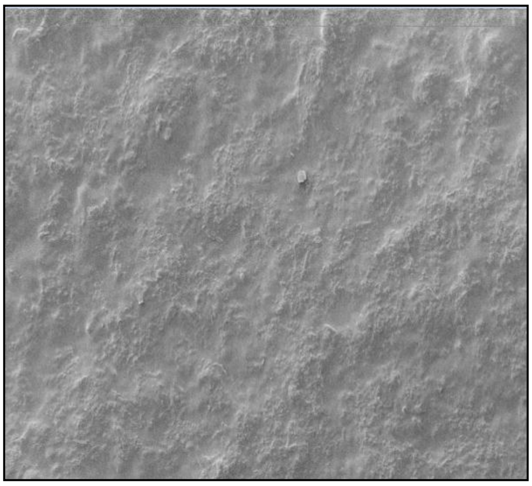

Fig. 4.59 SEM image of PVA/1TS/3NP at 25x magnification

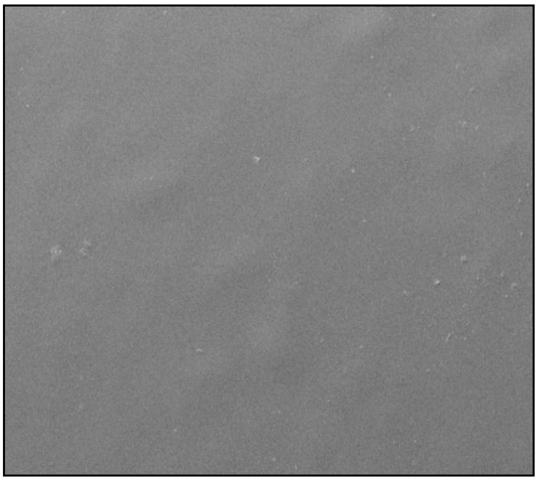

Fig. 4.60 SEM image of PVA/1RS/1BB at 25x magnification

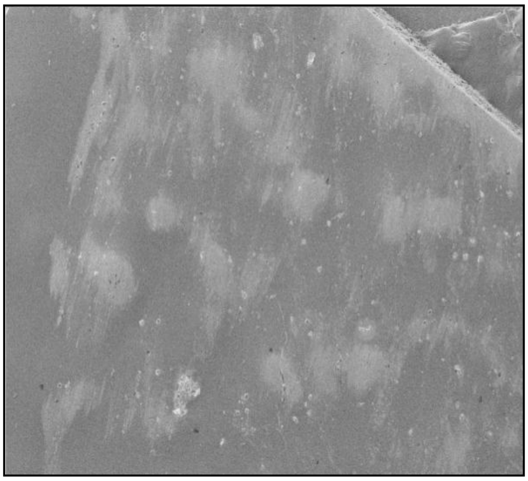

Fig. 4.61 SEM image of PVA/1RS/3BB at 25x magnification

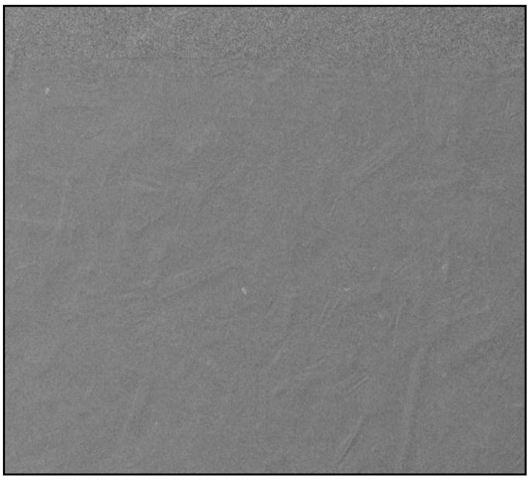

Fig. 4.62 SEM image of PVA/1RS/1KF at 25x magnification

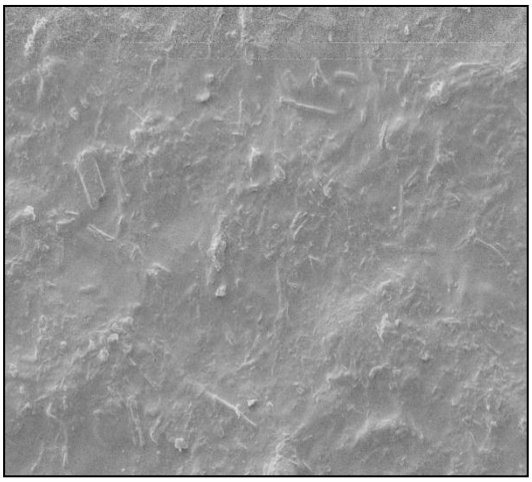

Fig. 4.63 SEM image of PVA/1RS/3KF at 25x magnification

Fig. 4.64 SEM image of PVA/1RS/1ROS at 25x magnification

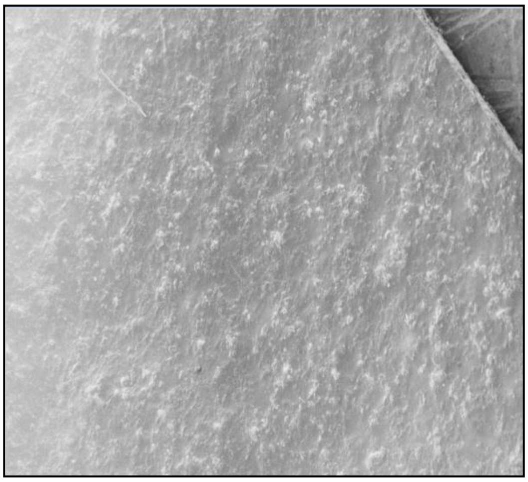

Fig. 4.65 SEM image of PVA/1RS/3ROS at 25x magnification

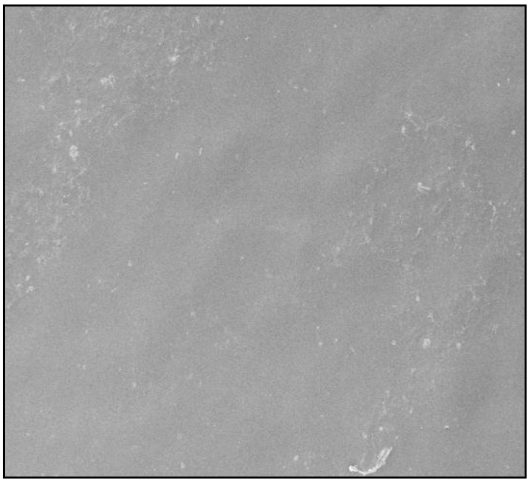

Fig. 4.66 SEM image of PVA/1RS/1NP at 25x magnification

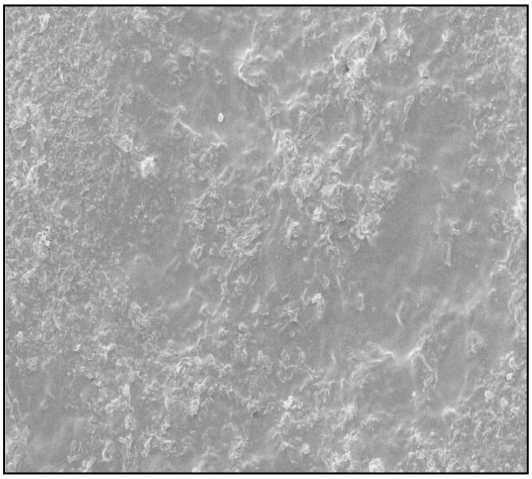

Fig. 4.67 SEM image of PVA/1RS/3NP at 25x magnification

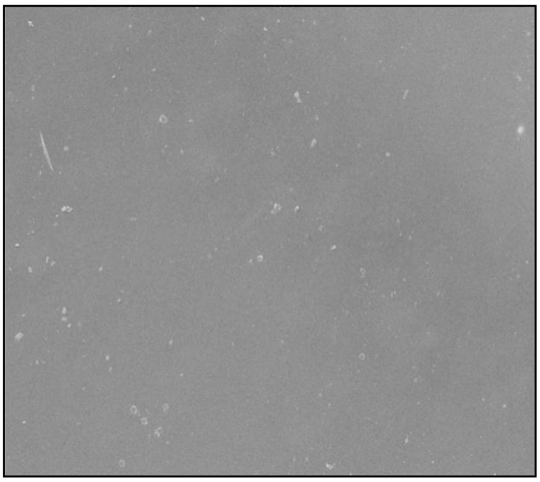

Fig. 4.68 SEM image of PVA/1SS/1BB at 25x magnification

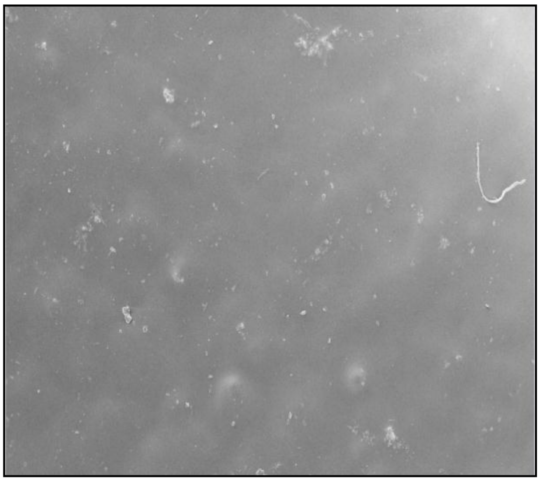

Fig. 4.69 SEM image of PVA/1SS/3BB at 25x magnification

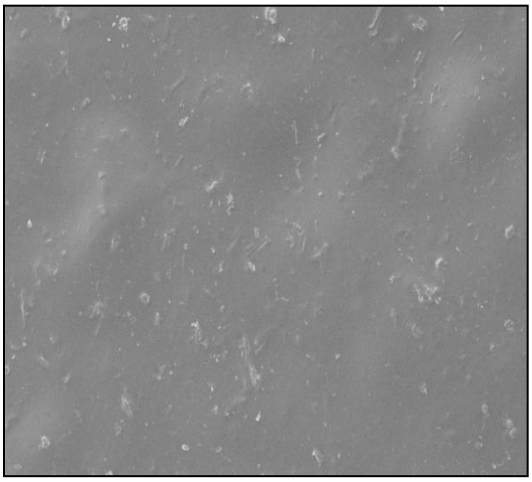

Fig. 4.70 SEM image of PVA/1SS/1KF at 25x magnification

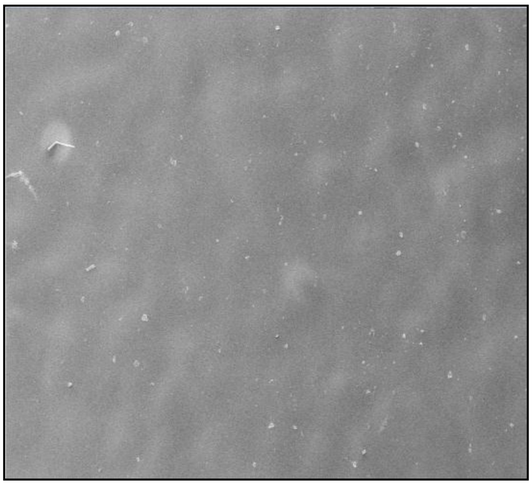

Fig. 4.71 SEM image of PVA/1SS/3KF at 25x magnification

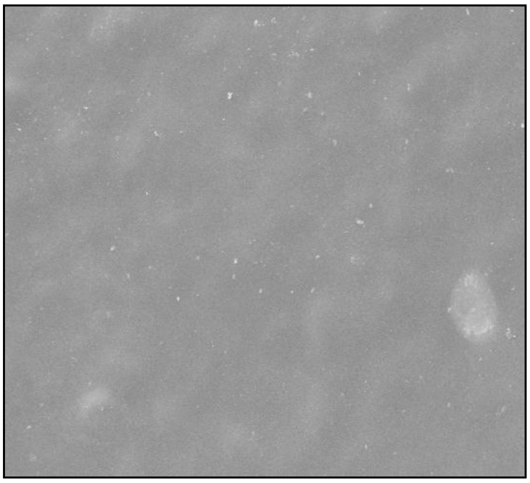

Fig. 4.72 SEM image of PVA/1SS/1ROS at 25x magnification

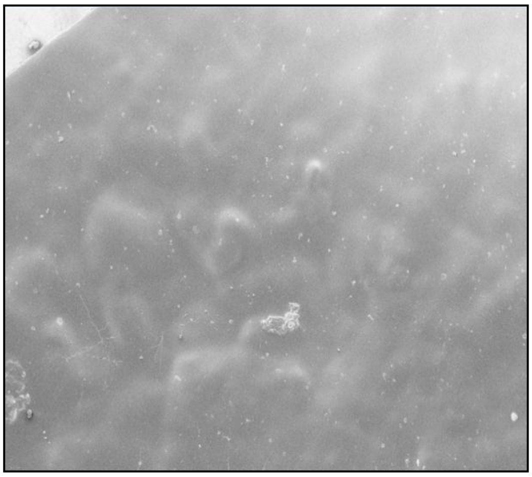

Fig. 4.73 SEM image of PVA/1SS/3ROS at 25x magnification

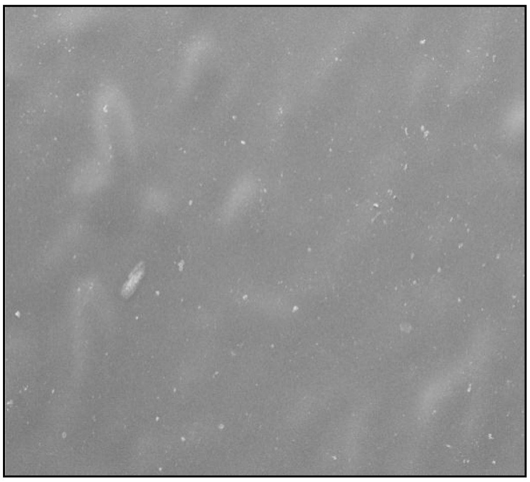

Fig. 4.74 SEM image of PVA/1SS/1NP at 25x magnification

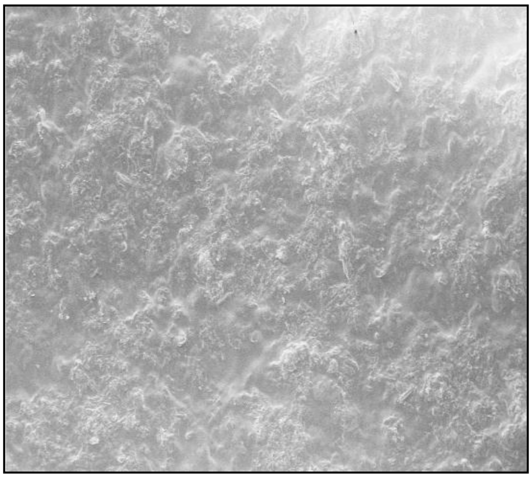

Fig. 4.75 SEM image of PVA/1SS/3NP at 25x magnification

The SEM images of PVA blended with 1g of different starches and different fibers composites show that the films had several bumps resembling fibers spread throughout the surface. These bumps formed by the embedded fibers were difficult to identify as the structure of the fibers were not clearly seen on the surface of the films. The fibers that were incorporated into the composite were seen to be well covered by the polymer matrix. As the concentration of fibers increases in the blends, the number of bumps on the film's surface increases. The increased amounts of fibers incorporated into the blend appeared to be well dispersed on the surface of the films and was also observed to be coated by the polymer matrix. No large agglomerates of fibers were seen and good adhesion between the fibers and matrix was observed, which should play an important role in improving the mechanical performance of the composites formed. For composites of PVA blended with sago starch and 3g of different fibers, no formation visually resembling fibers were clearly seen on the surface of the films but rather several apparent bumps were seen on the surface unlike other composite films like PVA/1TS/different fibers and PVA/1RS/different fibers where the shape of the fibers were clearly seen even though covered by the PVA matrix. The fibers blended in the PVA/sago/different fiber composites were well coated and this provides a graphical evidence for its slightly improved mechanical performance when compared to its tapioca and rice composites counterpart.

Fracture samples (PVA/different starches/different fibers composites)

Figure 4.76 to 4.82 shows the SEM images of the fractured surface of the composite PVA blended with 1g of tapioca starch and different fibers.

Figure 4.83 to 4.87 show the SEM images of the fractured surface of the composite PVA blended with 1g of rice starch and different fibers.

Figure 4.88 to 4.92 show the SEM images of the fractured surface of the composite PVA blended with 1g of sago starch and different fibers.

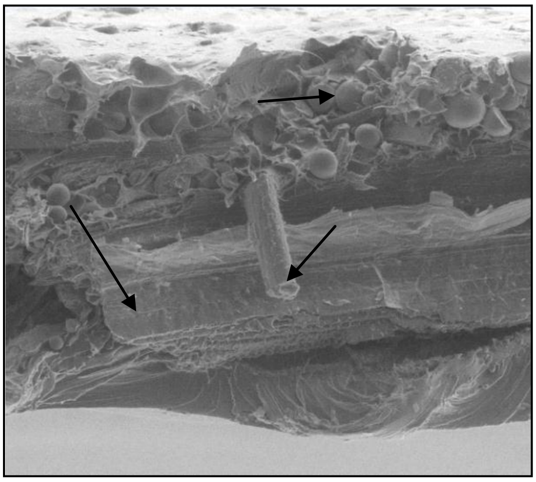

Fig. 4.76 SEM image of fractured surface of PVA/1TS/1BB at 1000x magnification

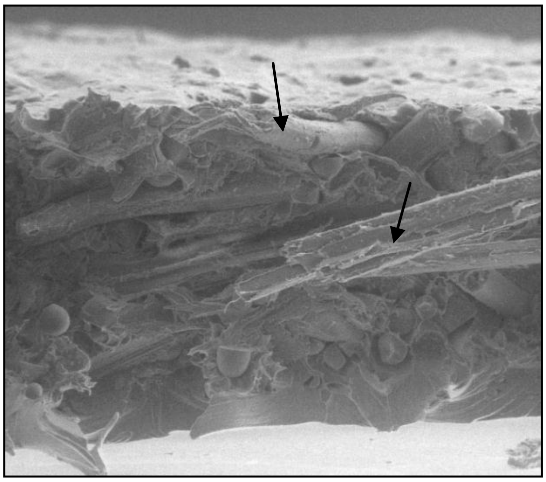

Fig. 4.77 SEM image of fractured surface of PVA/1TS/3BB at 1000x magnification

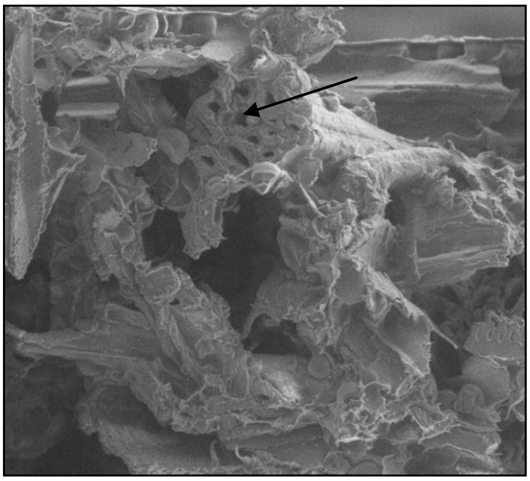

Fig. 4.78 SEM image of fractured surface of PVA/1TS/3KF at 1000x magnification

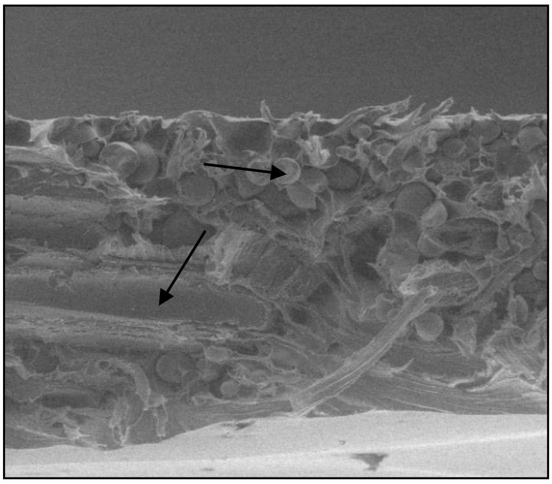

Fig. 4.79 SEM image of fractured surface of PVA/1TS/1ROS at 1000x magnification

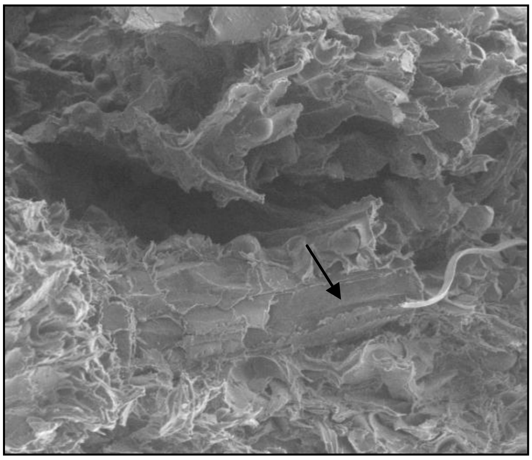

Fig. 4.80 SEM image of fractured surface of PVA/1TS/3ROS at 1000x magnification

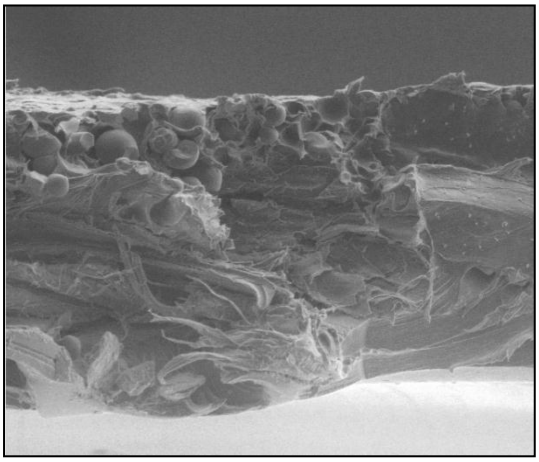

Fig. 4.81 SEM image of fractured surface of PVA/1TS/1NP at 1000x magnification

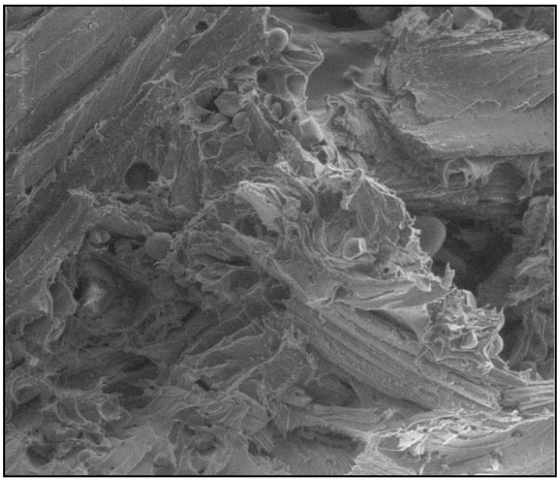

Fig. 4.82 SEM image of fractured surface of PVA/1TS/3NP at 1000x magnification

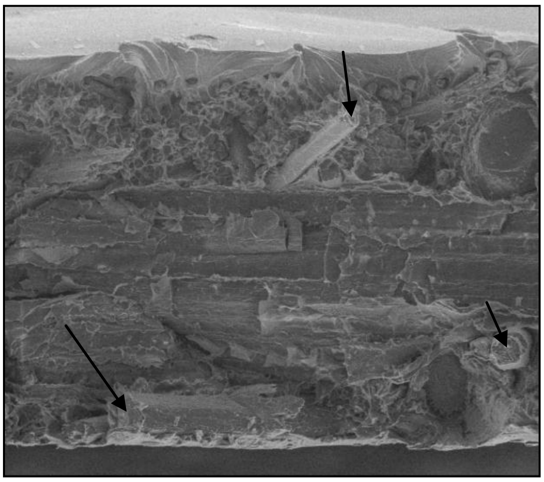

Fig. 4.83 SEM image of fractured surface of PVA/1RS/1BB at 1000x magnification

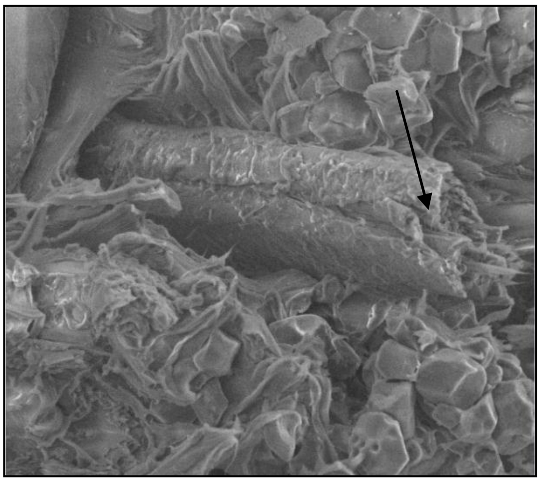

Fig. 4.84 SEM image of fractured surface of PVA/1RS/3BB at 1000x magnification

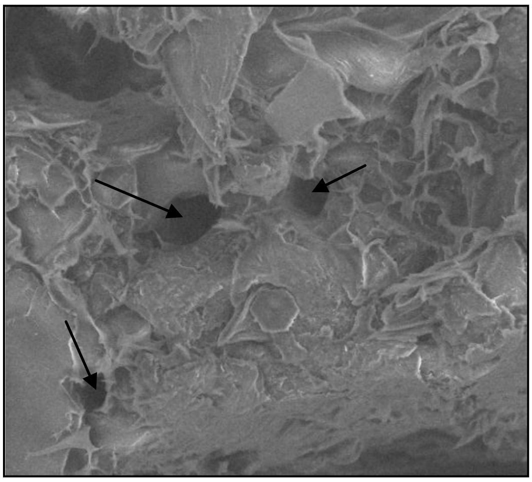

Fig. 4.85 SEM image of fractured surface of PVA/1RS/3KF at 1000x magnification

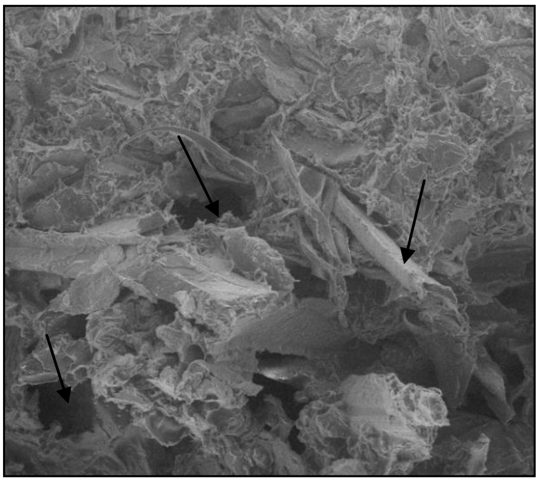

Fig. 4.86 SEM image of fractured surface of PVA/1RS/3ROS at 1000x magnification

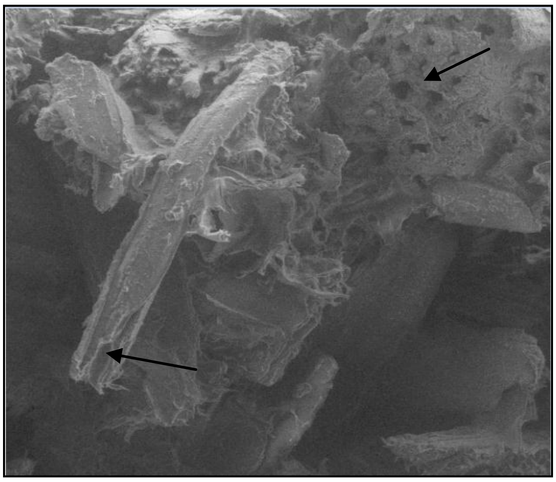

Fig. 4.87 SEM image of fractured surface of PVA/1RS/3NP at 1000x magnification

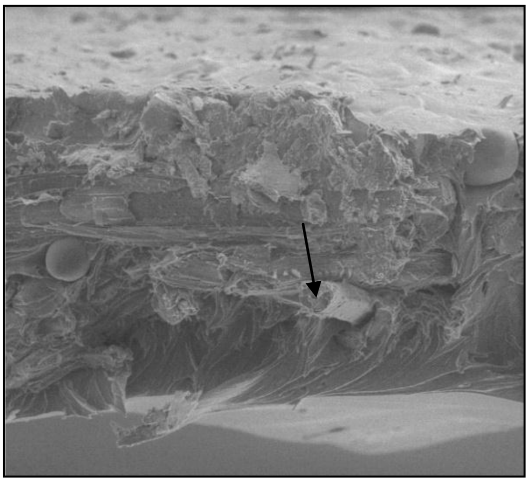

Fig. 4.88 SEM image of fractured surface of PVA/1SS/3BB at 1000x magnification

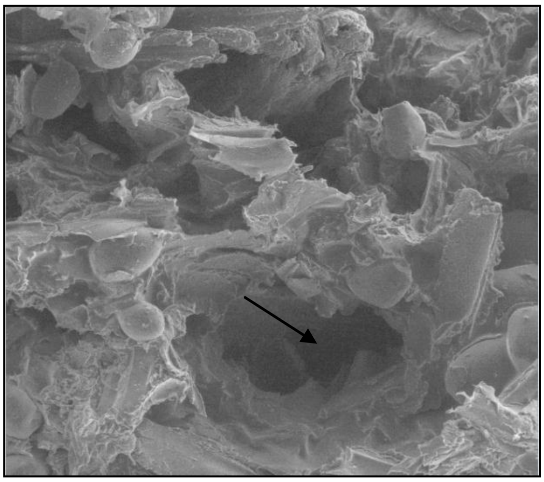

Fig. 4.89 SEM image of fractured surface of PVA/1SS/3KF at 1000x magnification

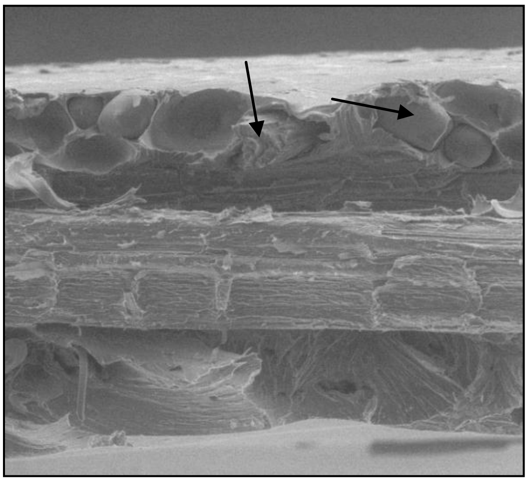

Fig. 4.90 SEM image of fractured surface of PVA/1SS/1ROS at 1000x magnification

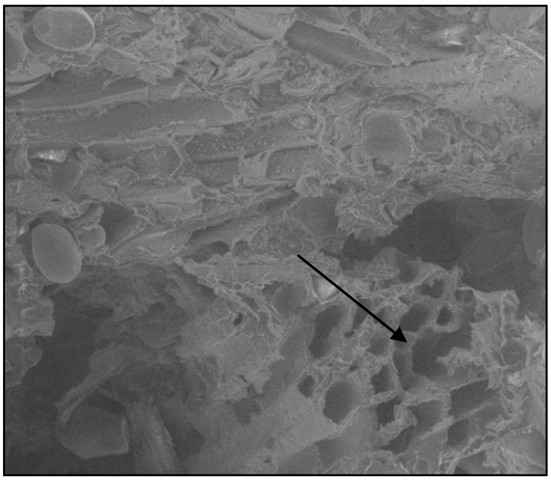

Fig. 4.91 SEM image of fractured surface of PVA/1SS/3ROS at 1000x magnification

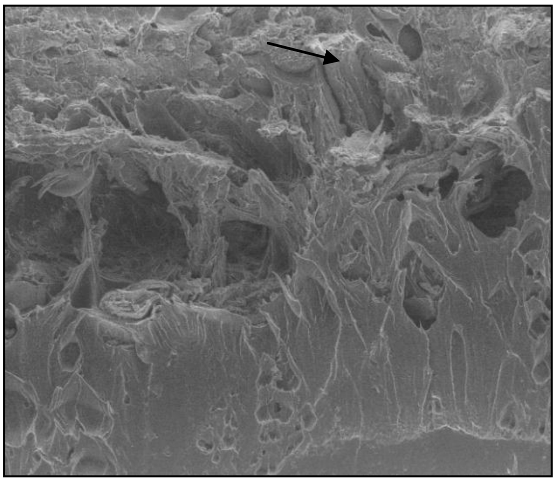

Fig. 4.92 SEM image of fractured surface of PVA/1SS/3NP at 1000x magnification

For the fracture samples, only certain SEM images were shown from all of the blended composites because some of the images did not show the presence of fibers or starches. The different natural fibers that were incorporated into the composites were bleached and mercerized. The effects of the surface modifications done on the fibers were investigated on the micro failure mode using the fractured specimens of the composites that were subjected to tensile stresses in the mechanical test. The SEM images of the fractured surfaces were taken at a cross section angle to discover the effects mentioned. Observations from the SEM images of the fractured specimens, as a whole, revealed that several fibers were pulled out from the matrix in the fracture process, with large voids thereby being created (as indicated by the arrows). Despite the fact that there were many cavities left by the pulled-out fibers, the surface of the fibers pulled-out from the matrix showed many traces of polymer matrix adhering to it indicating a closer contact between fiber and matrix and a better wetting of the fibers. This is also an indication that there were good fiber-matrix adhesions. In the blended composites, it can also be seen that were several breakages of fibers (as indicated by the arrows). This event may be caused by the non-uniformity of the treated fibers used. The non-uniformity of the fibers contributes to poor interfacial adhesion between the fiber and matrix that leads to inefficient stress transfer between fiber and matrix. Inefficient stress transfer subsequently leads to the breakage of fibers during the tensile testing. From the SEM images of the surface of the composites, the starch granules were not clearly seen but under SEM analyses of the fractured specimens it was revealed that there were some exposed starch granules underneath the surface of the films (indicated by arrows in the figures). The ungelatinized starches may partly cause lower mechanical properties for the composites because the starch granules form gaps between the granule and the continuous phase of the PVA matrix.