SEM Analysis
7.1 INTRODUCTION

The usage of polymer composites is increasing day by day because of their outstanding properties. The performance of a polymer composite depends not only on the selection of their components, but also on the interface between them. In order to meet the specific needs, sometimes it is necessary to modify the matrix, and the reinforcement.

The use of natural fibers as reinforcement in composites is a new field of research, which has been growing in the last decades and has been given a great interest by the automotive industry. However, the lack of good interfacial adhesion is a restriction for the use of natural fiber reinforced composites for high performance applications. This problem can be overcome by treating the fibers with suitable chemicals. In the present work different % of NaOH treatments have been applied to sisal fibers in order to improve their adhesion in composites materials. The effect of chemical modifications on the structure and morphology of the fibers has been studied by SEM.

Scanning Electron Microscopy [SEM] is used to reveal microstructural information of fractured surfaces of composites. Malunka et al. [1] studied the Preparation and characterization of EVA-sisal fiber composites. Morphological changes
were observed by SEM analysis. Mohanty Smita et al. [2] studied the interfacial, dynamic mechanical, and thermal fiber reinforced behavior of MAPE treated sisal fiber reinforced HDPE composites. The fiber-matrix morphology in the treated composites was confirmed by SEM analysis of the tensile fractured specimens. FTIR spectra of the treated and untreated composites were also studied, to ascertain the existence of type of interfacial bonds.

Martins et al [3] studied the effect of chemical modifications on the structure and morphology of the fibers by using $^{13}$C VACP-MAS NMR in solid state, and SEM. The results have shown that the best treatment conditions for sisal fibers were mercerization (5% NaOH, 3 h, 50°C) and acetylation prior to the treatment with 20/10 g/L R/H solution for 15 min. Antich et al. [4] studied the mechanical behavior of high impact polystyrene reinforced with short sisal fibres. They also studied the SEM analysis of these composites.

Varada Rajulu et al. [5] studied the mechanical properties of short natural fibre Hildegardia populifolia reinforced styrenated polyester composites. The fractured surfaces of these composites were investigated by SEM technique. Zarate et al. [6] studied the influence of fiber volume fraction and aspect ratio in resol-sisal composites. With the help of SEM analysis, they observed a good fibre-matrix adhesion in these composites. Mohanty et al [7] studied the effect of MAPP as Coupling Agent on the Performance of Sisal-PP Composites. They studied the morphology of the interface region by SEM analysis.
Varada Rajulu et al. [8] determined the Inter laminar shear strength of polycarbonate toughened epoxy composites reinforced with glass rovings. The morphology of the fractured surfaces of these composites was investigated using SEM technique. Varada Rajulu et al. [9] determined the void content, density, weight reduction and mechanical properties of short bamboo fibres/ styrenated polyester composites. The cellulose structure of the bamboo fibre and the morphology of the fractured surface of these composites were probed by SEM technique. Varada Rajulu et al. [10] studied the tensile properties of glass rovings/ hydroxyl terminated polyester toughened epoxy composites.

The morphology (SEM) of the cryogenically cooled and fractured surfaces indicated good bonding between the matrix and the reinforcement when a coupling agent was used. Xun et al [11] studied the self reinforced composites based on sisal. They studied the SEM analysis of benzylated sisal with different weight gains. Bai et al. [12] studied the failure mechanisms of sisal fibres in composites. They studied the micro-failure behaviour of sisal fibres by using Scanning Electron Microscopy (SEM). They observed the interfacial debonding of both sisal fibre bundle/epoxy matrix and tubular micro-fibre/bonding material was also noted in all embedded fibres. The fibre bundle/matrix interface had a moderate high strength: but the adhesive strength between the micro-tubular fibres and the bonding material appeared to be small.
In the present work the author studied the SEM analysis for composites prepared by reinforced with sisal fibres by following treatment with polyester matrix.

1. The composites prepared by reinforcing the sisal fibre boiled in 5% NaOH
2. The composites prepared by reinforcing the sisal fibre boiled in 10% NaOH
3. The composites prepared by reinforcing the sisal fibre boiled in 18% NaOH
4. The composites prepared by reinforcing the sisal fibre treated with 5% NaOH
5. The composites prepared by reinforcing the sisal fibre treated with 10% NaOH
6. The composites prepared by reinforcing the sisal fibre treated with 18% NaOH
7. The composites prepared by reinforced sisal fibre treated with 20% acetic acid
8. The composites prepared by reinforced sisal fibre treated with methanol

The author also studied the SEM analysis of 1:1 untreated and 18% boiled sisal/carbon and sisal/Kevlar hybrid composites. The results are presented in this chapter.

7.2 EXPERIMENTAL

7.2.1. Materials

In the present work unsaturated polyester resin was used as the matrix. Sisal fibre was used as one of the reinforcement in unhybridized composites. Carbon and Kevlar fibres were used as two reinforcements in hybrid composites with sisal fibres. NaOH, acetic acid and methanol were used for surface modification of sisal fibres. Methyl ethyl ketone peroxide and cobalt naphthenate were used as catalyst and as an
accelerator were supplied by M/S Bakelite Hylam, Hyderabad. Styrene monomer was used as cross linker.

7.2.2 Fiber Treatment

The sisal fibers were boiled in 5%, 10% and 18% aqueous NaOH for 30 minutes. The fibres were washed with water to remove the excess of NaOH sticking to the fibres. Finally the fibres were washed with distilled water and dried in hot Oven at 70°C for 3 hours. A 5%, 10%, 18% NaOH aqueous solution, acetic acid and methanol were taken in different trays and the sisal fibers were allowed to soak in the solutions for 1 hour. The fibers were then washed thoroughly with water to remove the excess of NaOH sticking to the fibers; Final washing was carried out with distilled water and the fibers were then dried in hot air oven at 70°C for 3 hours. The fibers were chopped into short fiber length of 2cm for moulding the composites.

7.2.3 Sample Preparation

For making composite, a glass mould with 280mmX150mmX30mm dimensions was used. The mould was coated with a thin layer of aqueous solution of poly vinyl alcohol (10WT %), which acts as a good releasing agent. Unsaturated polyester resin and styrene were mixed in the ratio of 100: 25 parts by weight respectively. Later, 1 Wt% methyl ethyl ketone peroxide and 1 Wt% cobalt naphthanate were added and mixed thoroughly. The fibre is added to matrix mixture, which was poured in the glass mould.
The excess resin was removed from the mould and glass plate was placed on top. In the present work the author used a hand lay-up method for preparing the composites. The castings were allowed to cure for 24hrs at room temperature and then casting is placed at a temperature. To ensure complete curing the composite samples were post cured at 80° C for 1 hour and test specimens of the required size were cut out from the sheet and were fractured after cooling them in liquid nitrogen.

7.3 SEM ANALYSIS

In order to observe the bonding between the reinforcement and matrix, the composite samples were cryogenically fractured. For SEM studies the samples were coated with gold by sputtering technique in order to ensure the conductivity. The SEM study was carried out by using Jeol JSM – 840 A, Japan at Indian Institute of Sciences, Bangalore.
7.4 RESULTS AND DISCUSSIONS

Scanning electron micrograms of the sisal fibre reinforced in polyester composites with 5%, 10 % and 18 % NaOH treatment are shown in figures 7.1 (a), 7.1 (c) and 7.1(e) at 100um magnification. From the microgram it is clearly evident that still some fibres are pulled out of the matrix. However at some places the matrix skin formation is observed on the fibres. These observations indicate that the bonding between the reinforcement and the matrix has partially improved by the alkali treatment.

Scanning electron micrograms of the sisal fibre reinforced in polyester composites with 5%, 10 % and 18 % NaOH boiled are shown in figures 7.1 (b), 7.1 (d) and 7.1(f) at 100um magnification. From these SEM analysis it was observed that fibres are broken and pulled out this observation indicates improvement in the bonding between the fibers of sisal fibre and the matrix when the fibres was boiled in NaOH. The surface of the fibres in the fibre became rough with the removal of hemicellulose and lignin. All these factors may be responsible for the improvement of the bonding between the reinforcement and the matrix.

Scanning electron microgram of the 20% acetic acid treated sisal fibre reinforced in polyester composites are shown in figure 7.1 (g) at 100um magnification. From the SEM analysis, there is more pullout of fibre is seen in the composites, which shows that carboxylic acid present in acetic acid weaken the interface bonding.
Scanning electron microgram of the sisal fibre reinforced in polyester composites with methanol treated are shown in figures 7.1 (h) at 100um magnification. From the SEM analysis, in the case of methanol surface of the fibre more smoother than the acetic acid group and more adhesion is observed when compare to acetic acid.

Scanning electron microgram of the 50:50 weight ratio of untreated sisal/kevlar hybrid composites are shown in figures 7.1 (i) at 100um magnification. From the SEM analysis it is cleared that the fibre contains bundles of individual cells that have been bound together by lignin-rich and the weak inter molecular bonds. Fibre pullout was identified in these composites and some microsized wholes have been observed on the surface of composites.

Scanning electron microgram of the 50:50 weight ratio of 18 % NaOH boiled sisal/kevlar hybrid composites are shown in figures 7.1 (j) at 100um magnification. From the SEM picture, of boiled composite, it is clearly confirm that the interfacial adhesion between the fibre and matrix is greater in the boiled (18% NaOH) sisal/kevlar composites, rough fibre surface would have a relatively strong mechanical interaction with the matrix resulting a higher mechanical strength.

Scanning electron microgram of the 50:50 weight ratio of untreated sisal/carbon hybrid composites are shown in figures 7.1 (k) at 100um magnification. From the SEM analysis, it is cleared that the fibre contain bundles of individual cells that have been bound together by lignin-rich and the weak inter molecular bonds. Fibre pullout was
identified in these composites and some microsized wholes have been observed on the surface of composites.

Scanning electron microgram of the 50:50 weight ratio of 18% NaOH boiled sisal/carbon hybrid composites are shown in figures 7.1 (1) at 100µm magnification. From the SEM picture, of boiled composite, it is clearly confirm that the interfacial adhesion between the fibre and matrix is greater in the boiled (18% NaOH) sisal/kevlar composites. The rough fibre surface would have a relatively strong mechanical interaction with the matrix resulting a higher mechanical strength.

Scanning electron microgram of carbon reinforced polyester composites are shown in figures 7.1 (n) at 100µm magnification. From the SEM picture, it is observed that good bonding between carbon matrix than previous cases. When the mechanical properties of fibre reinforced polyester composites prepared under different weight ratios are compared it can be seen that these are maximum for composites with carbon fibre.

Scanning electron microgram of kevlar reinforced polyester composites are shown in figures 7.1 (m) at 100µm magnification. From the SEM picture, it is observed that good bonding between kevlar matrix than previous cases. When the mechanical properties of fibre reinforced polyester composites prepared under different weight ratios are compared it can be seen that these are maximum for composites with kevlar fibre.
Fig: 7.1 (a) SEM of 5% treated sisal fibre polyester composites
Fig: 7.1 (b) SEM of 5% boiled sisal fibre polyester composites.
Fig: 7.1(c) I SEM of 10\% treated sisal fibre polyester composites.
Fig: 7.1 (d) SEM of 5% boiled sisal fibre polyester composites.
Fig: 7.1 (e) SEM of 18 % treated sisal fibre polyester composites.
Fig: 7.1 (f) SEM of 18 % boiled sisal fibre polyester composites.
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Fig: 7.1 (g) SEM of acetic acid treated sisal fibre polyester composites
Fig:7.1 (h) SEM of methanol treated sisal fibre polyester composites.
Fig: 7.1 (i) SEM of 50 : 50 weight ratios of untreated sisal/kevlar polyester hybrid composites.
Fig: 7.1 (j) SEM of 50 : 50 weight ratios of 18% NaOH boiled sisal/kevlar polyester hybrid composites.
Fig: 7.1 (k) SEM of 50 : 50 weight ratios of untreated sisal/carbon polyester hybrid composites
Fig: 7.1 (1) SEM of 50 : 50 weight ratios of 18% NaOH boiled sisal/carbon polyester hybrid composites.
Fig: 7.1 (m) SEM of kevlar polyester composites.
Fig: 7.1 (n) SEM of carbon polyester composites.
7.5 REFERENCES


