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SYNTHESIS AND CHARACTERISATION OF SISAL FIBRE REINFORCED BIO POLYESTER COMPOSITES FROM MUSTARD OIL

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Abstract

Bio polyesters are emerging and promising biomaterials for tissue scaffolding drug delivery, implant coatings and biomedical. In this work, Bio polyesters were obtained by mustard oil based polyester resin with acrylo nitrile, TEGMA, dimethyl aniline. The prepared hydroxylated mustard oil and polyester resin were characterised by FT-IR. The bio polyester and their composites were characterised by FT-IR, swelling analysis, mechanical properties, soil burial test, SEM analysis and antimicrobial activity. This mustard oil based bio polyesters may serve as a replacement in many applications.

Key words: **Biopolyesters, Mustard oil, Polyester resin, FT-IR and Soil burial test.**

I. INTRODUCTION

Natural oils are oil-based polymers having many advantages compared with the polymers prepared from petroleum-based monomers. They are biodegradable and cheaper than petroleum polymers. The oil constituents are complex multi-component mixtures of different triglycerols [1]. Mustard oil has the least amount of saturated fatty acids. The oil is food grade oil that is reported to have anti-bacterial property [2].

The polyesters obtained from mustard oil are bio polyesters which are generated from the renewable natural sources and they are biodegradable polyesters. These polyesters are non-toxic in nature [3].

The natural fibres are renewable, biodegradable and non-abrasive, possesses a good calorific value, exhibit excellent mechanical properties and can be incinerated for energy recovery have low density and are inexpensive. Sisal fibres are the natural fibres. The sisal fibres are used as a reinforcement and thermoset or thermoplastic materials are used as a matrix [4].

II. EXPERIMENTAL

2.1 MATERIALS

Mustard oil was commercially available in local markets. Formic acid, hydrogen peroxide, maleic anhydride, dimethyl aniline, TEGMA were purchased from Sigma-Aldrich.

2.2 METHODS

2.2.1 Synthesis of bio polyester resin

Mustard oil was carried out using 30% hydrogen peroxide and formic acid, in ice water bath. The reaction was vigorously stirred at 8 hours. Then it was poured into a separator funnel and extracted with ether. The resulting product obtained as hydroxylated mustard oil. The resulting product was reacted with maleic anhydride, sodium acetate, morpholine and the mixture was refluxed for 2 hours at 70-80^{o}C and 160^{o}C for 20 minutes under vacuum condition using rota mandle to yield a yellow transparent liquid bio polyester resin.

2.3 Synthesis of bio polyester and their composites

The bio polyester neat sheet and their composites were synthesised by treating bio polyester resin with triethylene glycol dimethacrylate, benzoyl peroxide, dimethyl aniline and acrylo nitrile. The neat sheet was coded as MFB. The treated sisal fibre with varying compositions (5%, 10%, 15%) added to the above mixture. The mixture was poured into the clean silicon oil spreaded glass mould. The mixture was dried in vacuum air oven at 80^{o}C for 6 hours. The 5%, 10% and 15% sisal fibre reinforced composites were coded as MFBSL5, MFBSL10 and MFBSL15.

III. CHARACTERISATION

3.1 Spectral studies

FT-IR spectral analysis of synthesised resin and composites were analysed by KBr pellet method via Shimadzu FT-IR 8400S spectrometer.

3.2 Determination of swelling coefficient

The bio polyester neat sheet and their composites were subjected to swelling experiments. The density of bio polyester neat sheet and their composites were analysed using ASTM D792 method.

The swelling coefficient 'Q' was evaluated using the formula,

$$\text{Swelling coefficient (Q)} = \frac{\text{Weight of the solvent in swelled polymer}}{\text{Weight of the swelled polymer}} \times \frac{d_r}{d_s}$$

Where,

d_r = Density of polymer

d_s = Density of solvent

3.3 Soil burial test

Biodegradation of the polyester and their composites were studied by soil burial test. For the soil burial test the replicate pieces of the sample (5 x3 cm) were buried in the garden soil at the depth of 30 cm from the ground surface for 3 months, inoculated with the sewage sludge having ability to adhere and degrade the polyester sheets. The test specimen was periodically removed from the soil and the specimen was then gently washed to remove attached soil and dust after being dried in vacuum oven.

Degree of biodegradation,

$$D = \frac{W_O - W_t}{W_O} X\, 100$$

Where,

W_o = Weight of the original film

W_t = Weight of residual film after degradation for different time

3.4 Scanning electron microscope analysis

Scanning electron microscope analysis was conducted (ESEM-Quanta 200, Fei) to study the degradation of biopolyester sheet and their composites before and after soil burial degradation test.

3.5 Evaluation of performance under antimicrobial activity

Antimicrobial activity was evaluated by agar diffusion method. The test was done in triplicates. Amikacin of positive control was used for antimicrobial activity testing. The microbial strains used for bacterial adhesion study were gram positive bacteria such as *Bacillus subtilis* and gram negative bacteria such as *Pseudomonus aeruginosa*. The diameters of zones were measured to the nearest millimeter with vernier calipers or a thin transparent millimeter scale.

IV. RESULTS AND DISCUSSIONS

4.1 Characterisation of bio polyester resin

4.1.1 FT-IR analysis

The FT-IR spectra of hydroxylated mustard oil resin and bio polyester resin as shown in Figure 4.1 and Figure 4.2. The hydoxylated mustard oil resin has been showed the peak at 3473.72 cm^{-1} due to the presence of free -OH groups in the molecule. The bio polyester resin was showed the carbonyl band of mustard oil combined with fumarate group at 1733.07 cm^{-1}.

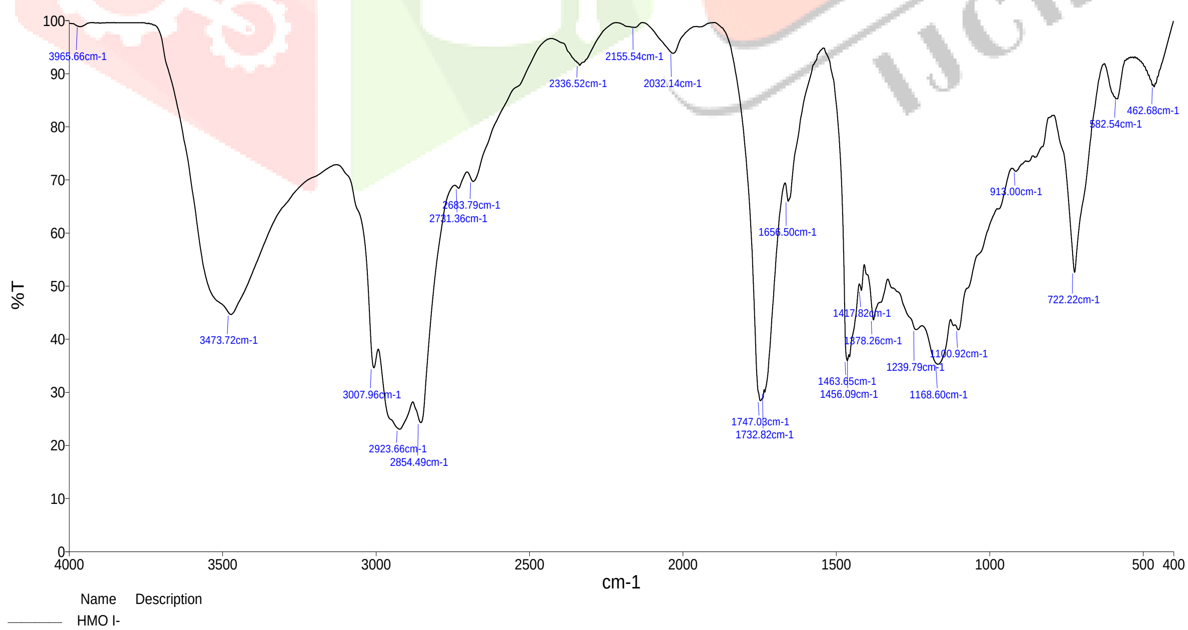


Figure 4.1 FT-IR spectrum of hydroxylated mustard oil

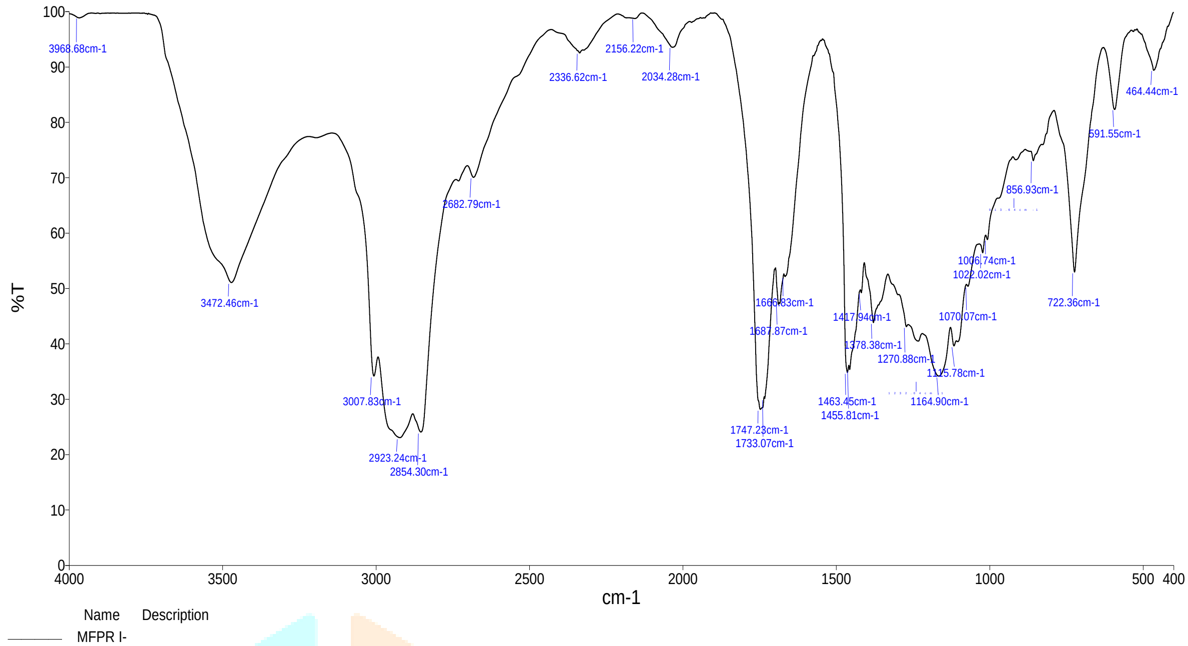


Figure 4.2 FT-IR spectrum of polyester resin

4.2 Characterisation of bio polyester sheet and their composites

4.2.1 FT-IR analysis

The FT-IR spectra of bio polyester neat films and their composites (Fig. 4.3-Fig. 4.5) almost same as bio polyester resin but the absence of peak at 1666.83 cm^{-1} indicated to the absence of double bonds.

The FT-IR spectra of sisal fibre reinforced bio polyester composites have irregular pattern of absorption bands formed and no new bands were obtained due to the mixture of fibres and bio polyester resin.

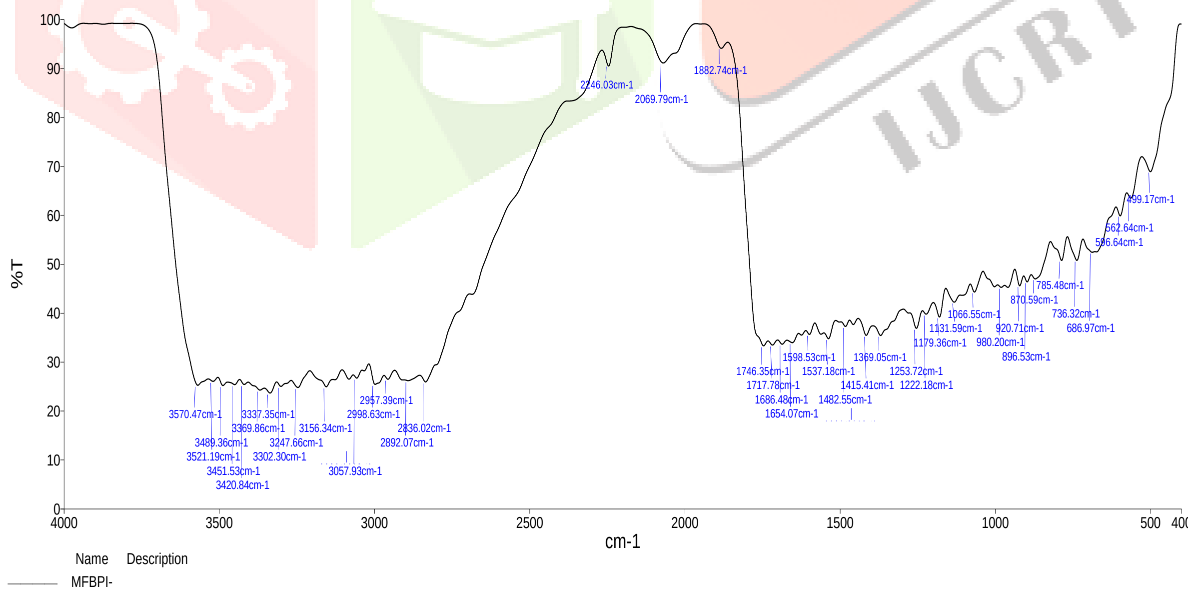


Figure 4.3 FT-IR spectrum of MFB

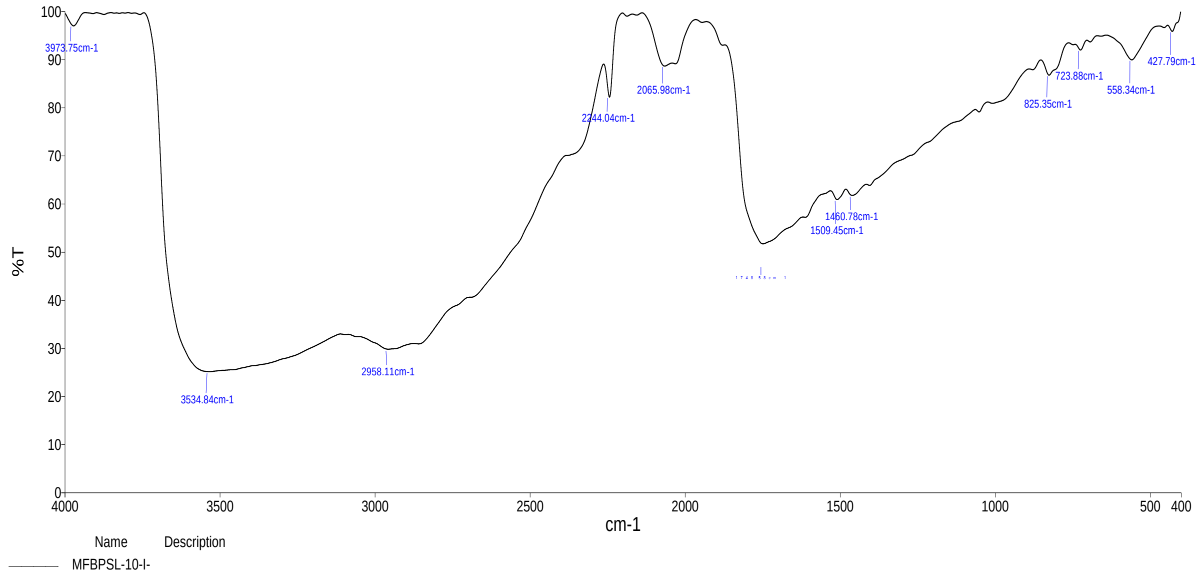


Figure 4.4 FT-IR spectrum of MFBSL10

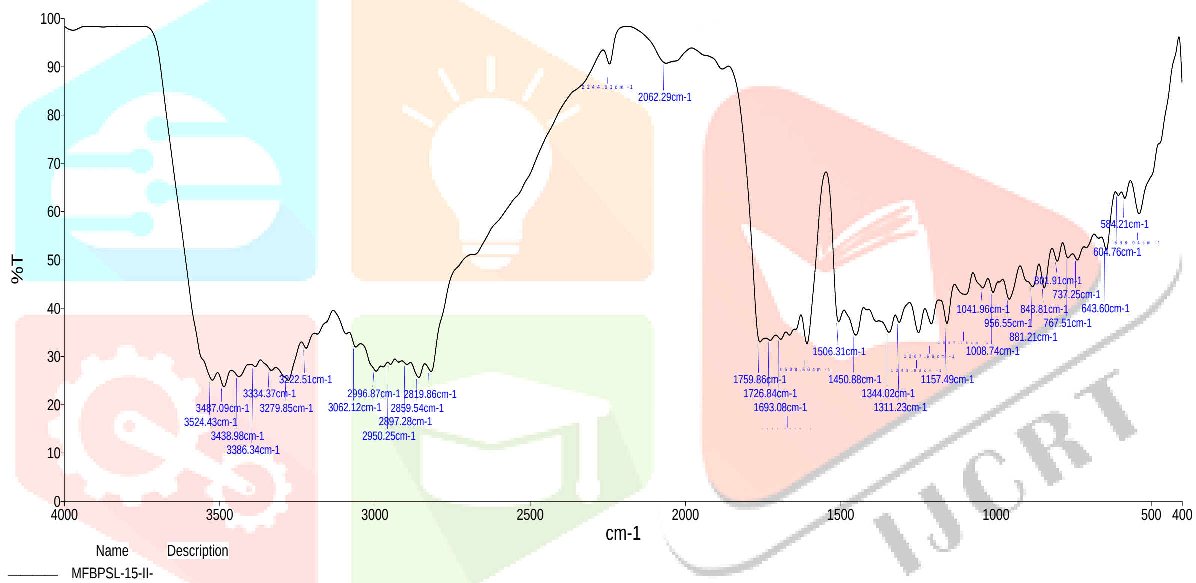


Figure 4.5 FT-IR spectrum of MFBSL15

4.2.2 Swelling coefficient

The bio polyester and their composites have been maximum in dimethyl acetamide. They revealed that the higher swelling showed that the bio polyester and their composites were crosslinked. The higher percentage of fibre reinforced bio polyester composites has higher crosslink density. The swelling coefficient of bio polyester and their composites are given in Table 1.

Table 1. Swelling coefficient of bio polyester neat sheet and sisal fibre reinforced composites

Biopolyester and their composites	EMK	DMA	Toluene	Chloroform
MFB	0.23	1.03	0.24	0.27
MFBSL5	0.25	1.06	0.24	0.30
MFBSL10	0.25	1.09	0.25	0.32
MFBSL15	0.26	1.12	0.27	0.32

4.2.3 Soil burial test

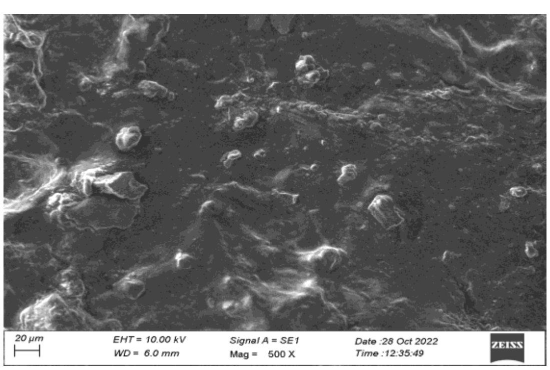
The weight loss percentage of bio polyester films and their composites are given in Table 2. After period of two months of soil burial the prepared bio polyester and their composites were losses due to microorganism attack, thus confirmed that the bio polyester and their sisal fibre reinforced composites were biodegradable. The percentage of sisal fibre content increases, the degradation rate also increases. They revealed that the sisal fibre reinforced bio polyester composites has higher degradation than bio polyester sheet.

Table 2. Weight loss percentage of bio polyester sheet and their composites

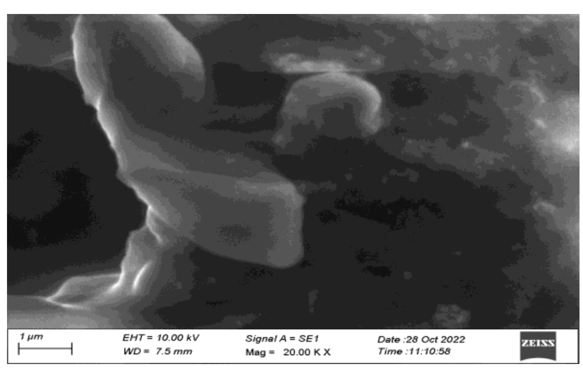
Biopolyesters and their composites	Weight loss (%)
MFB	26.175
MFBSL5	33.619
MFBSL10	42.01
MFBSL15	51.87

4.2.4 Scanning electron microscope analysis

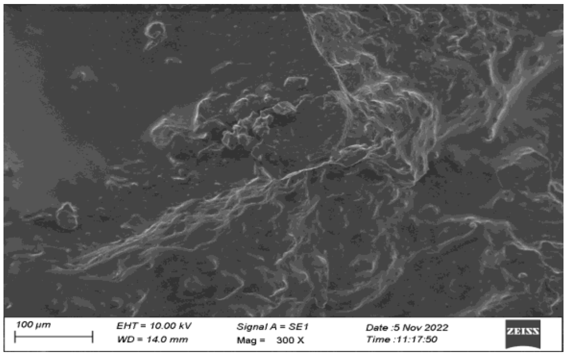
Surface damage and microbial growth on the bio polyester composites were studied from SEM analysis. SEM is used to study the morphological behavior of bio polyester composites. SEM micrographs of the bio polyester composites before and after soil burial were analysed which shows the degradation of the bio polyester composites by microbial action. Figure 4.6 shows the SEM micrographs of the bio polyester composites MFBSL10 and MFBSL15 before and after degradation.



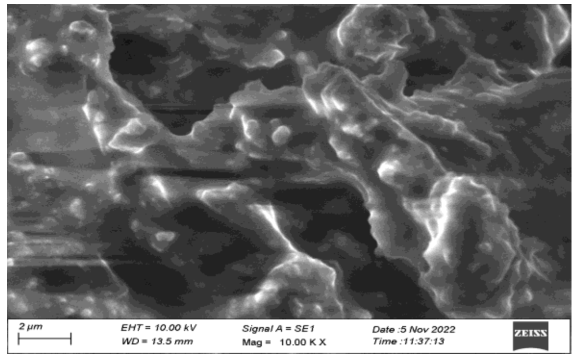
MFPSL10 (before)



MFBPSL15 (after)



MFBSL15 (before)



MFBSL15 (after)

Figure 4.6 SEM micrographs of the bio polyester composites MFBSL10 and MFBSL15 before and after degradation.

4.2.5 Antimicrobial studies

The antimicrobial activities of bio polyester composites were investigated using gram negative bacteria *Pseudomonas aeruginosa* and gram positive bacteria *Bacillus subtilis*. The microbial activity images of bio polyester composites are shown in Figure 4.7 and the results are given in Table 3. Among prepared bio polyester composites, 10% and 15% sisal fibre reinforced composites showed maximum zone of inhibition against gram negative bacteria *Pseudomonas aeruginosa* and gram positive bacteria *Bacillus* subtilis when compared to 5% sisal fibre reinforced composites.

Table 3 Inhibition zone (mm) of biopolyester composites

Biopolyester composites	**Microorganisms**	
	Pseudomonas aeruginosa	***Bacillus subtilis***
MFBSL5	12	14
MFBSL10	14	16
MFBSL15	14	15

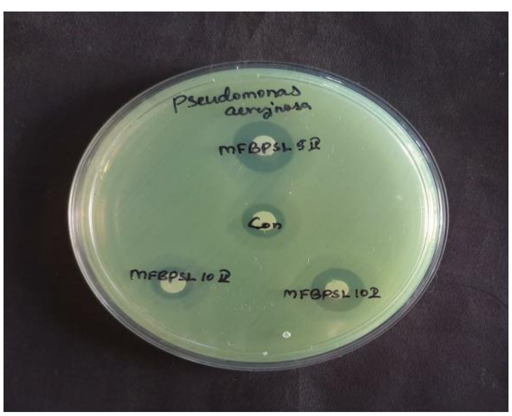


Figure 4.7. Culture plates of bio polyester composites using *Pseudomonas aeruginosa* and *Bacillus subtilis*

V. CONCLUSIONS

The synthesis of hydroxylated mustard oil resin and polyester resin were confirmed by FT-IR. The synthesis of bio polyester and their composites were confirmed by FT-IR analysis. The swelling coefficient of the bio polyester and their composites were maximum in dimethyl acetamide. The sisal fibre content of bio polyester composites increases, solvent absorptivity can also increases. Soil burial degradation study showed that the bio polyester sheet, 5%, 10% and 15% of sisal fibre reinforced composites were biodegradable. The biodegradation of bio polyester and their composites have been confirmed by SEM analysis. The anti-microbial activity of 10% and 15% sisal fibre reinforced composites showed maximum zone of inhibition when compared to 5% sisal fibre reinforced composites.

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