



ISSN: 0976-3031

Available Online at <http://www.recentscientific.com>

CODEN: IJRSFP (USA)

International Journal of Recent Scientific Research
Vol. 13, Issue, 11(A), pp. 2510-2513, November, 2022

**International Journal of
Recent Scientific
Research**

DOI: 10.24327/IJRSR

Research Article

STUDIES ON AGEING CHARACTERISTICS OF NOVEL BIOPOLYESTER COMPOSITES BASED ON MUSTARD OIL AND SISAL FIBRE

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DOI: <http://dx.doi.org/10.24327/ijrsr.2022.1311.0513>

ARTICLE INFO

Article History:

Received 10th October, 2022

Received in revised form 25th October, 2022

Accepted 18th November, 2022

Published online 28th November, 2022

ABSTRACT

Poly (mustard oil fumarate) biopolyester and their composites were synthesised from poly (mustard oil fumarate) biopolyester resin with acrylo nitrile, TEGMA, benzoyl peroxide and sisal fibre. The ageing characteristics of biopolyester and their composites have been studied. These revealed that the 15% and 10% sisal fibre reinforced biopolyester composites were good hydrolytic stability and thermally stable. The antimicrobial activities were studied.

Keywords:

Biopolyester, Sisal fibre, Benzoyl peroxide, Composites and Antimicrobial activity

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INTRODUCTION

Polymers are complex and giant molecules composed of many repeated smaller molecules. Polymers are having physical properties, mechanical behaviors and chemical structures etc. They are used in chemical industry as lubricants, coatings and stabilizers^[1]. Natural oils consist of molecules which can be categorized as comprising of a glycerol molecule attached to three fatty acid chains. The oils are the most important sources for biopolymers^[2]. Mustard oil is a triglyceride molecule. It contains 81% of unsaturated acids and 19% of saturated acids. The oil has high levels of alpha-linoleic acid and erucic acid. Erucic acid can also cause liver enzymes to malfunction and additionally reduces fertility^[3].

Polyesters are vital class of high performance and engineering polymers, which find use in a number of chemical applications. Unsaturated polyester resins are creating fibre reinforced plastics because of the ease of use fabrication and the low cost^[4]. The natural fibres are obtained from nature. The fibres are included into a matrix material such as thermosetting plastics and rubber^[5]. The fibres are used for particle boards, medicine and chemicals^[6]. Ageing is the most important effects that limits the lifetime of plastics. It is the slow and irreversible change in the properties of polymers under the action of heat, light, chemicals such as acid, alkali and some salts. Each environment contains different microorganisms and has different degradation^[7].

MATERIALS AND METHODS

Materials

Mustard oil was commercially available in local markets. Formic acid, hydrogen peroxide, sodium acetate, morpholine, acrylo nitrile were obtained from Sigma-Aldrich. Sisal fibre was collected locally.

Synthesis of poly (mustard oil fumarate) biopolyester resin

Mustard oil was carried out using 30% hydrogen peroxide and formic acid, in ice water bath. The reaction was vigorously stirred at 6 hours. Then it was poured into a separator funnel and extracted with ether. The resulting product was obtained as hydroxylated mustard oil resin. The resin was reacted with maleic anhydride, sodium acetate, morpholine and the mixture was refluxed for 3 hours at 70-80°C and 160°C for 30 minutes under vacuum condition using rotamandle to yield a yellow transparent liquid poly (mustard oil fumarate) biopolyester resin.

Synthesis of poly (mustard oil fumarate) biopolyester and their composites

The poly (mustard oil fumarate) biopolyester neat sheet and their composites were synthesised by treating poly (mustard oil fumarate) biopolyester resin with triethylene glycol dimethacrylate, benzoyl peroxide, dimethyl anile and acrylo nitrile. The neat sheet was coded as MFB. The treated sisal fibre with varying compositions (5%, 10%, 15%) added to the

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above mixture. The mixture was poured into the clean silicon oil spreaded glass mould. The mixture was dried in vacuum air oven at 80°C for 7hours. The 5%, 10% and 15% sisal fibre reinforced composites were coded as MFBSL5, MFBSL10 and MFBSL15.

Ageing characteristics of biopolyester sheet and their composites

The studies on the stability of biopolyester and their composites under various ageing conditions were carried out using ASTM standards. The conditioning as well as specimen preparation was followed using proper procedure discussed in the following sections. The determination of stability was carried out by weight loss estimation.

Determination of weight loss in hydrolytic conditions

The weight loss of biopolyester sheet and their composites (3 x 1 x 0.1 cm) were immersed separately in the media of water, alcohol and salt solution (1N NaCl) for a period of 60 days under ambient conditions. The medium was changed and fresh medium was added at an interval of one week. The biopolyester sheets were removed at the end of the exposure, allowed dried in vacuum oven and then weighed.

Determination of weight loss in hostile chemical environment

The degradation of biopolyester sheet and their composites in hostile acidic (1N HCl), basic (1N NaOH) and oxidant (H₂O₂) medium were studied. The medium was changed and the fresh medium was added at the interval of one week. The biopolyester sheets were removed at the end of exposure, dried in vacuum oven and then weighed.

Determination of weight loss in organic solvents

The solubility of biopolyester sheet and their composites were determined in organic solvents such as ethyl methyl ketone, toluene, dimethyl acetamide and chloroform. The prepared biopolyester sheet and their composites were conditioned and weighed before exposing to solvent. The samples were immersed in solvent an airtight container at two months. The solvent was changed and fresh medium was added at the interval of one week. The biopolyester sheet and their composites were removed after two months dried in vacuum oven and weighed.

Determination of stability under thermal ageing

The stability of biopolyester sheet and their composites under prolonged exposure in thermal environment was evaluated. The newly prepared biopolyester sheet and their composites were kept in an oven. Then the biopolyester sheet and their composites were removed from the oven, cooled to ambient temperature and weighed.

Soil burial test

The biopolyester sheet and their sisal fibre reinforced composites (5 x 3 cm) were buried in the soil at a depth of 30 cm from the ground surface for 60 days, inoculated with compost having the capacity to hold and degrade the polymer. At fixed time, the samples were detached, washed with distilled water in order to ensure the stop of the degradation, dried out at room temperature to a constant weight and stored in dusk.

Degree of biodegradation,

$$D = \frac{W_0 - W_t}{W_0} \times 100$$

Where,

W₀= Weight of the original film

W_t= Weight of residual film after degradation for different time

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 *Pseudomonas aeruginosa*. The diameters of zones were measured to the nearest millimeter with vernier calipers or a thin transparent millimeter scale.

RESULTS AND DISCUSSION

Hydrolytic degradation of biopolyester and their composites

The weight loss of the poly (mustard oil fumarate) biopolyester and their composites in media like water, ethanol and sodium chloride solutions were given in Table 1. The poly (mustard oil fumarate) biopolyester sheet and their composites have no weight loss in ethanol medium. But a small weight loss has been observed in water and sodium chloride solution.

The sodium chloride solution influences the hydrolytic stability of the biopolyesters. Since the ionic permeation in the biopolyester matrix was considerable, the effect of Na⁺Cl⁻ ions on the degradation was also notable.

Table 1 Weight loss of poly (mustard oil fumarate) biopolyester and their composites under hydrolytic conditions

Biopolyesters and their composites	Weight loss (%)		
	Water	Ethanol	Salt solution (NaCl)
MFB	1.753	0	0.03
MFBSL5	2.468	0	0.05
MFBSL10	2.921	0	0.057
MFBSL15	3.273	0	0.09

Oxidative degradation of the biopolyester and their hostile chemical environment

The hostile reactive chemicals such as acid (1N HCl), base (1N NaOH) and oxidant (30 % H₂O₂) also induced the degradation of polymers. The weight loss of the poly (mustard oil fumarate) biopolyester and their composites aged in the hostile reactive chemicals such as acid (1N HCl), base (1N NaOH) and oxidant (30 % H₂O₂) were given in Table 2. The degradation of poly (mustard oil fumarate) biopolyester and their composites were very high in NaOH. The biopolyesters with ester linkages can be easily hydrolysed in the presence of alkalis.

The 15% and 10% sisal fibre reinforced biopolyester composites have higher weight loss in alkaline medium than 5% sisal fibre reinforced biopolyester composite and biopolyester neat sheet due to its high crosslink density.

Table 2 Weight loss of poly (mustard oil fumarate) biopolyester and their composites in hostile chemical environment

Biopolyesters and their composites	Weight loss (%)		
	Acid (1N HCl)	Base (1N NaOH)	Oxidant (30% H ₂ O ₂)
MFB	1.87	4.351	0.87
MFBSL5	1.42	4.963	0.21
MFBSL10	1.36	5.475	0.35
MFBSL15	1.55	5.793	0.54

Stability of biopolyester and their composites in organic solvents

The solvents like ethyl methyl ketone, toluene, dimethyl acetamide and chloroform were selected for the stability of newly synthesised biopolyester and their composites. The weight loss percentage of samples has been presented in Table 3. The poly (mustard oil fumarate) biopolyester and their composites have been no weight loss in ethyl methyl ketone, toluene and dimethyl acetamide. But very small weight loss was observed in chloroform solution. This weight loss was due to the hydrophobic nature.

Table 3 Weight loss of poly (mustard oil fumarate) biopolyester and their composites in organic solvents

Biopolyesters and their composites	EMK	Toluene	Chloroform	DMA
MFB	0	0	0.704	0
MFBSL5	0	0	0.692	0
MFBSL10	0.029	0	0.713	0
MFBSL15	0.043	0	0.625	0

Stability of biopolyester and their composites under thermal ageing

Thermal ageing of biopolyester and their composites leads to vary in weight. The weight loss percentage of biopolyester and their composites were shown in Table 4. The poly (mustard oil fumarate) biopolyester and their composites were almost same under thermal ageing.

Table 4 Weight loss of poly (mustard oil fumarate) biopolyester and their composites under thermal ageing

Biopolyesters and their composites	Weight loss (%)
MFB	1.172
MFBSL5	0.726
MFBSL10	0.761
MFBSL15	0.973

Soil burial test

After two months of soil burial of biopolyester and their composites weights were losses due to microorganism attack, thus confirmed that the biopolyester and their composites have been biodegradable. Weight loss of biopolyester and their composites were given in Table 5.

Table 5 Weight loss of poly (mustard oil fumarate) biopolyester and their composites under soil burial test

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

Antimicrobial studies

The antimicrobial activities of biopolyester composites were investigated using gram negative bacteria *Pseudomonas aeruginosa* and gram positive bacteria *Bacillus subtilis*. The microbial activity images of biopolyester composites were shown in Figure 1 and the results have been given in Table 6. Among prepared biopolyester 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 6 Inhibition zone (mm) of biopolyester composites

Biopolyester composites	Microorganisms	
	<i>Pseudomonas aeruginosa</i>	<i>Bacillus subtilis</i>
MFBSL5	12	14
MFBSL10	14	16
MFBSL15	14	15



Figure 1 Culture plates of biopolyester composites using *Pseudomonas aeruginosa* and *Bacillus subtilis*

CONCLUSIONS

The ageing characteristic showed that the poly (mustard oil fumarate) biopolyester and their composites were small weight loss was observed in water and sodium chloride solution. The biopolyester and their composites were stable towards acid and oxidant. The high weight loss percentage was observed in alkaline medium. The composites have no weight loss in toluene, ethyl methyl ketone and dimethyl acetamide but very small weight was observed in chloroform solution. 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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