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Research Article

FTIR, SEM AND THERMAL ANALYSIS OF HIBISCUS MUCILAGE BLENDED LOW DENSITY POLYETHYLENE

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ABSTRACT

The trends of applications of plastics material have got very wide in our daily life as well as in industries. In this research work the evaluations of biodegradation has been done by FTIR and SEM analysis. The thermal properties of the LDPE and hibiscus mucilage compounds were characterized by differential scanning calorimeter (DSC). The samples of different level of combinations of hibiscus mucilage were processed. The melting behavior, crystallization temperature and degree of crystallinity were analyzed and comparative study of different concentrations of hibiscus mucilage with LDPE was done. The comparative study of before and after soil burial also done.

Key Words:

LDPE, Mucilage, SEM, FTIR, DSC,
Crystallinity, Thermal Characterization.

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INTRODUCTION

Now a day plastics material have found wide applications in every aspect of life and industries and manufactured in very large quantities. Non-biodegradable plastics have become technologically important since 1940s in many industrial, domestic, commercial and environmental applications due to their stability and durability¹. Development of innovative biodegradable plastics can be considered as the best way to solve solid waste problem. Blending synthetic polymers with natural polymers, such as starch, mucilage, cellulose, lignin, chitin and chitosan, is applied as an important way to accelerate polymer degradation². It is generally recognized that the polyethylenes are bioinert and hydrophobic in nature³. By the use of green polymers (mucilage/polysaccharides/starch) the polyethylenes will become bioactive and hydrophilic due to their hydrophilic nature. Conversion of non-biodegradable plastics into biodegradable plastics is an alternative, eco-friendly way to solve disposal problem. The important advantages of substituting conventional plastics with bioplastics are that they are compostable with organic wastes and returned to enhance the soil fertility⁴. The peak point of melting curve is the regularly examined property of semi crystalline polymeric materials⁵.

Mucilage is a physiological product of polysaccharide mixture⁶. The mucilage powder was taken out from leaves of *Hibiscus rosa-sinensis* (China rose) of Malvaceae family which was arranged from Bandel area of Hooghly district (W.Bengal, India).

In the current project, the compounds of low density polyethylene and hibiscus mucilage of different level concentrations were made. An attempt of effect of mucilage concentrations on morphology and chemical structure of polymer studied by SEM and FTIR respectively. Apart from these melting characteristics and crystallinity of the compounds were characterized by DSC (differential scanning calorimeter).

MATERIALS AND METHODS

Materials

The grade 16MA400 Low Density Polyethylene (LDPE) polymerized by high pressure polymerization process was obtained from Reliance Polymers, India. The mucilage was prepared from the leaves of *Hibiscus rosa-sinensis* commonly known as China rose. About ninety eight percent (98%) purified glycerol of Merk Co., Germany was utilized as a plasticizer. Hydrated crystal purified Fe₂SO₄ of Merk Co., Germany was utilized as pro-oxidant.

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Methods

The prepared mucilage powder was added in same amount of glycerol and Fe_2SO_4 (1 mass % of LDPE) traditionally. The formulated polymers were processed with different levels of variations of hibiscus mucilage in two roll mill at 150°C - 160°C for 10 – 12 minutes of rolling. Sheets of 03 mm thickness were processed by compression moulding at 150°C under pressure. One sheet of 100 % virgin LDPE was also processed to be used as controlled reference. The sample sheets of all concentrations were shaped into strips and buried into the soil for six months to evaluate biodegradation.

Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted on a Shimadzu, Japan spectrometer to understand the chemical changes in the polymeric materials before and after soil burial. The range of wave number of $400 - 4000 \text{ cm}^{-1}$ was taken for measurement.

The scanning electron microscopic (SEM) analysis of the samples was carried out using ZEISS scanning electron microscope at 10 KV to understand the morphology of the samples. The surfaces of the samples were coated with metal such as gold.

The thermal characteristics in terms of melting point, crystallization temperature and degree of crystallinity of the mucilage based compounds and virgin LDPE were characterized, using Mettler Toledo (Switzerland) differential scanning calorimeter (DSC). The weighed samples of 5-10 milligrams were heated in the temperature range of 25° to 160°C at a heating rate of 10°C per minute in nitrogen atmosphere to determine the melting temperature of the compounds before and after soil burial. Then the samples were cooled from 160° to 25°C at 10°C per minute cooling rate in nitrogen atmosphere to check the percentage crystallinity and crystallization temperature. As a reference an empty aluminium crucible was used. Indium and Zinc were used to calibrate the instruments.

RESULTS AND DISCUSSION

FTIR Analysis

The FTIR spectra of compound of LDPE and hibiscus mucilage before and after soil biodegradation have shown in Figure – 1. It has been observed that the peaks of compound after six months of soil burial retracted more. The change of peaks after soil burial indicates the degradation of the blend⁷, which means the material, has been consumed by microorganism present in soil.

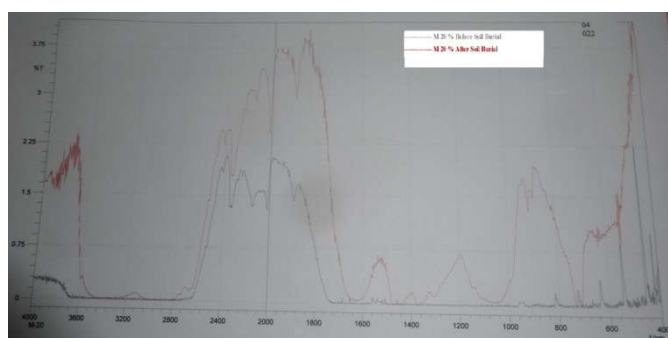


Figure 1 FTIR Spectra of samples (LDPE+Mucilage) before and after Soil Burial.

SEM Analysis

Scanning electron micrograph (SEM) was conducted to evaluate the surface morphology of the samples and comparative compilation was done.

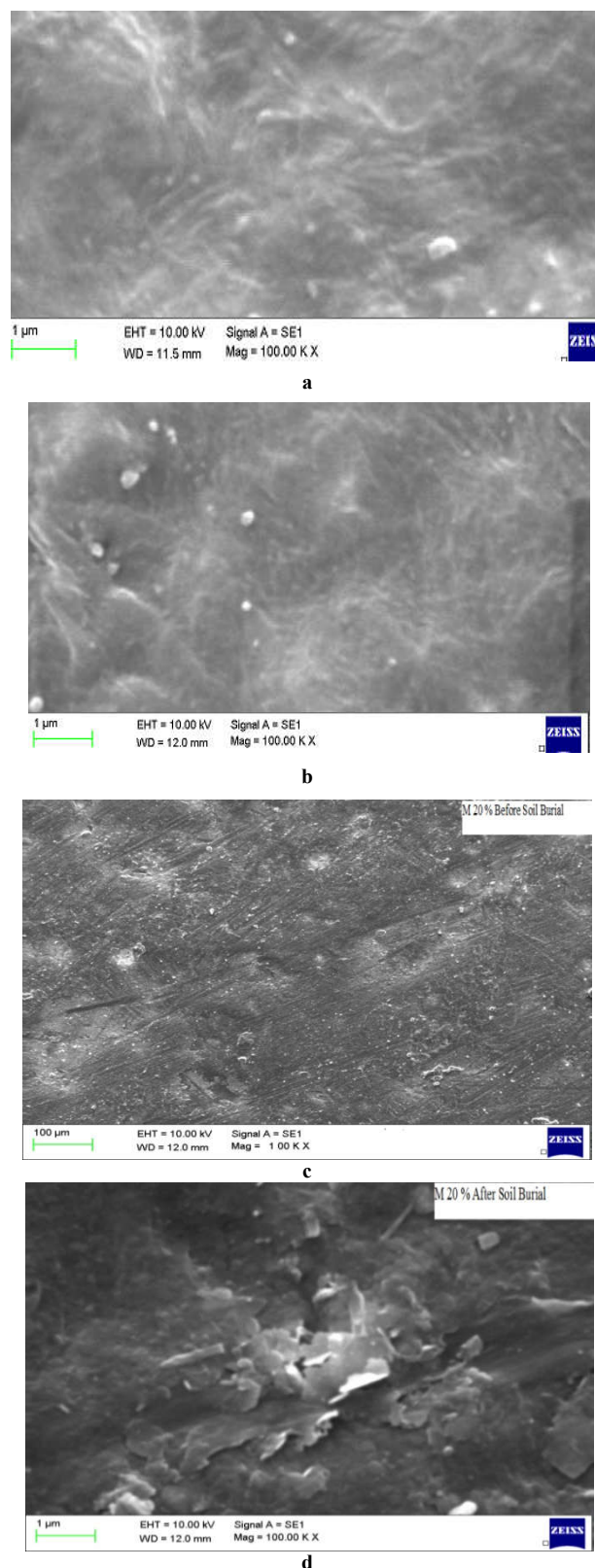


Figure 2 (a) SEM Image of sample (Virgin LDPE 100 %) before soil burial, (b) SEM Image of sample (Virgin LDPE 100 %) after six months of soil burial, (c) SEM Image of sample (LDPE+Mucilage) before soil burial, (d) SEM Image of sample (LDPE+Mucilage) after six months of soil burial.

The scanning electron micrograph of virgin LDPE and compounds of LDPE and hibiscus mucilage before and after soil burial displayed in Figures- 2 (a), (b), (c) and (d). The surfaces of virgin LDPE were observed smooth in the Figures- 2 (a) and (b). Almost unchanged morphology of surfaces of virgin LDPE before and after soil burial indicates the biodegradation happened in soil are negligible, where as in Figures -2 (c) and (d), the surfaces of the compounds were distinct. As compare to before soil burial, in the image of compound after six months of soil burial the surfaces were found rough with some holes and bores. These roughness, holes and bores indicate the rate of biodegradation with the removal of mucilage from the compound.

DSC Analysis

The differential scanning calorimetric results in terms of melting point (Tm), crystallization temperature (Tc) and extent of crystallinity of hibiscus mucilage and low density polyethylene (LDPE) mixed polymers before and after soil burial has been expressed in the Table -1.

In the cooling phase of differential scanning calorimetric analysis the crystallization curves and peaks were found. The crystallization temperatures (Tc) of the virgin low density polyethylene (100 % LDPE) before and after soil burial were found 90.41^oC and 90.71^oC respectively. On blending of mucilage along with glycerol and Fe₂SO₄ with low density polyethylene (LDPE) the crystallization temperature of 20 % mucilage compound before and after soil burial were found 93.29 and 93.81 respectively, where as of 25 % mucilage compound before and after soil burial were obtained 93.28^oC and 93.82^oC respectively. The comparative cooling curves with crystallization peaks of LDPE and hibiscus mucilage compounds before and after soil burial plotted in Figure – 5 and Figure – 6 respectively.

The percentage of crystallinity was found in cooling phase of differential scanning calorimetric analysis. The crystallinity of virgin low density polyethylene (100 % LDPE) before and after soil burial were obtained 40.69 % and 40.96 % respectively.

Table 1 Melting Temperature, Crystallization Temperature and Crystallinity of Mucilage based LDPE.

S.No.	Hibiscus Mucilage Content	Melting Temperature, Tm in (°C)		Crystallization Temperature, Tc in (°C)		Crystallinity in %	
		Before Soil Burial	After Soil Burial	Before Soil Burial	After Soil Burial	Before Soil Burial	After Soil Burial
1.	0 %	108.86	108.09	90.49	90.71	40.69	40.96
2.	20 %	107.62	107.20	93.29	93.81	36.26	32.41
3.	25 %	107.24	107.06	93.28	93.82	33.69	26.69

Note: The degree of crystallinity is determined by comparing the measured heat of fusion with the theoretical heat of fusion of 100% crystalline low density polyethylene of 140 J/g⁸.

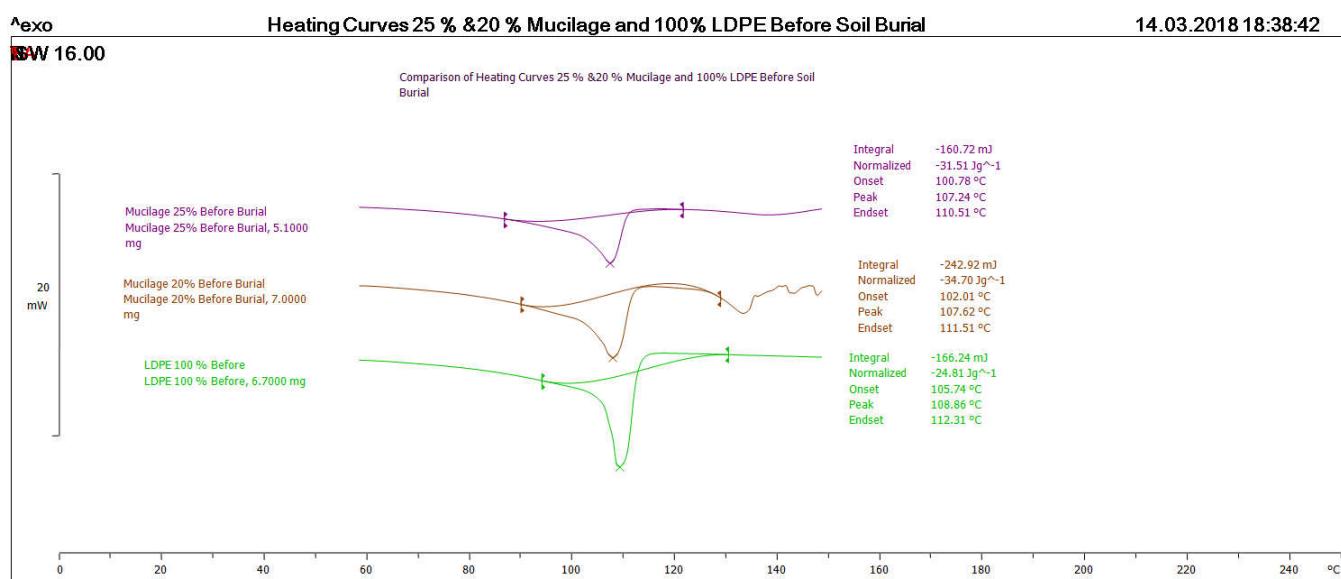
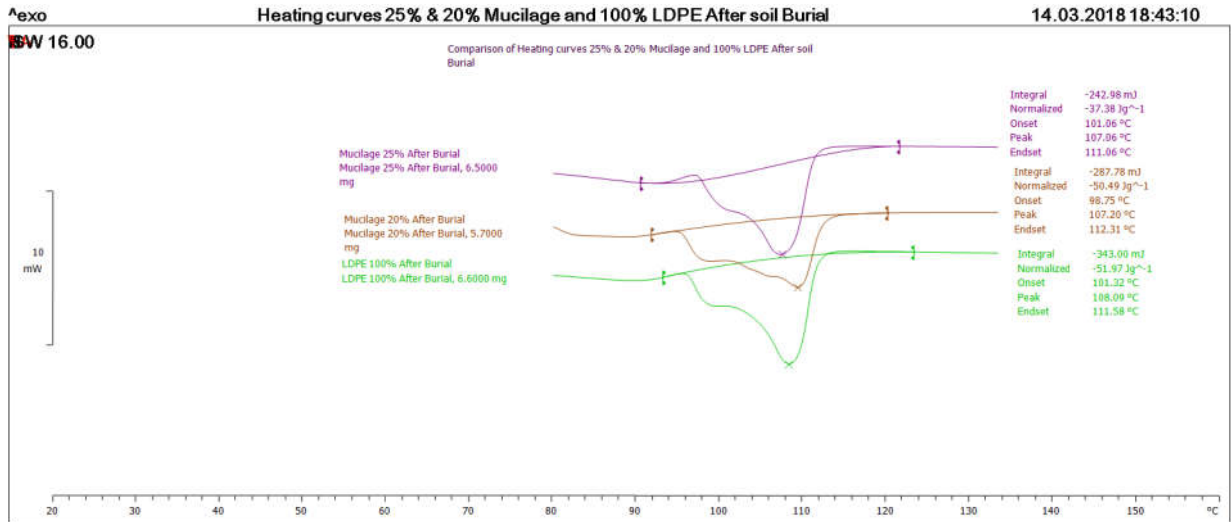


Figure 3 DSC Heating Curves of LDPE and mucilage Compounds before Soil Burial.

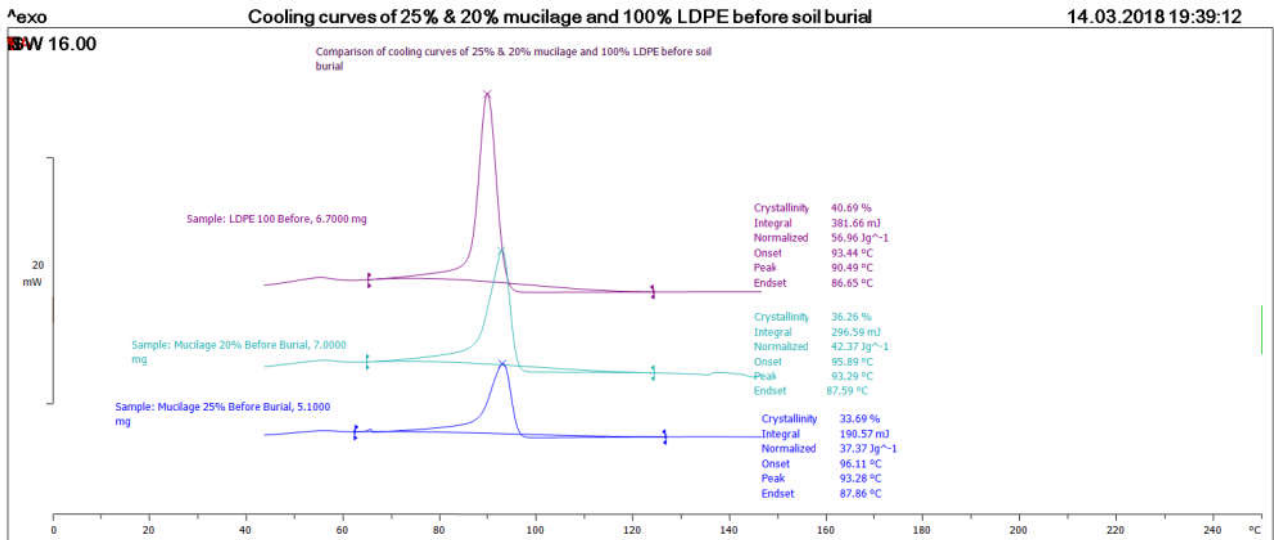
The melting points of virgin low density polyethylene (100 % LDPE) before and after soil burial were obtained 108.86^oC and 108.09^oC respectively. On blending of hibiscus mucilage along with glycerol and Fe₂SO₄ with low density polyethylene (LDPE) melting point decreased by around 1^oC due to presence of additives in the compounds. The heating curves with melting peaks of LDPE and mucilage compounds before and after soil burial plotted in Figure – 3 and Figure – 4 respectively.

On blending of mucilage along with glycerol and Fe₂SO₄ with low density polyethylene (LDPE) the crystallinity of 20 % mucilage compound before and after soil burial were observed 36.26 % and 32.41 % respectively, where as of 25 % mucilage compound before and after soil burial were obtained 33.69 % and 26.69 % respectively. The comparative cooling curves with percentage of crystallinity of LDPE and hibiscus mucilage compounds before and after soil burial plotted in Figure – 5 and Figure – 6 respectively.



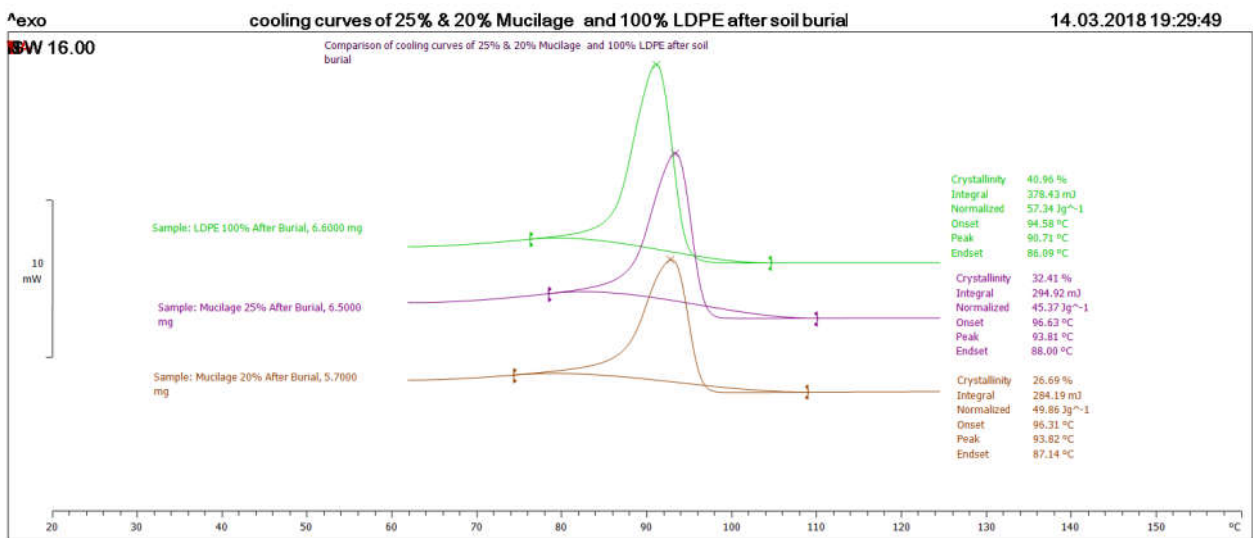
Lab: METTLER

Figure 4 DSC Heating Curves of LDPE and mucilage Compounds after Soil Burial.



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Figure 5 DSC cooling Curves of LDPE and mucilage Compounds before Soil Burial.



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Figure 6 DSC Cooling Curves of LDPE and mucilage Compounds after Soil Burial.

The degree of crystallinity was calculated by comparing the measured heat of fusion with the literature value of heat of fusion of 100 % crystalline low density polyethylene (LDPE) of 140 J/g⁸.

CONCLUSION

In the present research work blend of LDPE and hibiscus mucilage was prepared. Compounding of hibiscus mucilage with traditional plastics is a possible way to make it degradable. On blending of hibiscus mucilage the melting points decreased by about 1⁰C where as crystallization temperature increased by about 3⁰C of compounded polymers. The crystallinity decreased with the increase in concentrations of hibiscus mucilage. After six months of soil burial negligible change in crystallinity of virgin LDPE observed, where as distinguishable decrease in degree of crystallinity of hibiscus mucilage compound was observed. As hardness and strength decrease with decreasing crystallinity, the DSC results can be correlated with the mechanical properties of the polymeric materials⁵. In fact the course of material taken as natural polymer for compounding to accelerate the degradation under natural environment, is entirely different from earlier used natural polymers.

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