

Synthesis Characterization and Biodegradation of Low Density Polyethylene [LDPE] and Partially Carboxymethylated Starch [PCMS] Blend

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Sodium salts of partially carboxymethylated starch (Na-PCMS) with degree of substitution (DS) 0.21, 0.314, 0.58 and 1.10 were synthesized from starch. These starch ethers were blended with low density polyethylene (LDPE) in various proportions using a blender mixer. Effect of DS on compatibility of the blend was studied by monitoring the change in mechanical properties. The blend having optimum properties was studied for the growth of *Bacillus* species [BS] and degradation by means of weight loss and change in tensile strength as well as percent elongation. The morphological change in blend was studied by scanning electron microscopy.

Keywords: Biodegradation; Polyethylene; Carboxymethylated starch; Blends

INTRODUCTION

Polyethylene and its articles are widely used for various purposes both domestically and industrially because of its excellent performance properties. Most of the polyethylene materials do not decompose in the environment and the effect of the discarded plastics on the

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environment can no longer be ignored. Several approaches have been proposed for producing biodegradable polymers by fermentation and chemical method and by utilization of natural polymers [1]. Synthesis of biodegradable plastics by fermentation- and chemical methods may increase the production cost. So, in an attempt to prepare cost effective plastics having biodegradable characteristics as well as optimum mechanical properties, starch was modified to starch ethers and blended with LDPE. Which was decomposed by micro-organisms present in soil and water.

Biodegradable plastics should have needed performance characteristics in their intended use. Therefore, Na-PCMS of varying DS were prepared and blended with LDPE in different proportions to achieve better performance characteristics. Modification of starch was required because starch and PE are non compatible at micro level which often leads to poor performance [2-5].

Changes in mechanical properties as a result of blending have been observed. Also the growth of BS in the blend and total cellular protein was monitored. Weight loss, change in tensile strength and per cent elongation were examined.

EXPERIMENTAL

Materials

LDPE from Indian petrochemicals ltd. [IPCL], Baroda, Gujarat, India. Starch, monochloroacetic acid from National chemicals, Baroda. Solvents of laboratory grade and other laboratory chemicals were used after routine purification.

SYNTHESIS OF Na-PCMS

Na-PCMS of different DS was synthesized by method as reported elsewhere [6, 7].

Blending

Blending of LDPE and Na-PCMS was carried out as reported [8].

Preparation of Polymer Sheet

Polymer sheets of various thickness were prepared as reported [9].

Isolation of Blend Degrading Culture

Organisms capable of degrading blends were isolated by enrichment culture technique using CMS (1% w/v) as sole source of carbon. Medium employed consisted of

Ingredient	gms/lit
Magnesium Sulphate	0.2
Calcium Chloride	0.02
Monopotassium Phosphate	1.0
Dipotassium Phosphate	1.0
Ammonium-Nitrate	1.0
Ferric Chloride	0.05

Suspend 3.27 gms of medium in 1000 ml distilled water. Sterilize it by autoclaving at 15 lbs pressure and 121°C for 15 minutes. 50 ml of above medium was taken in a 250 ml erlenmeyer flask which was inoculated with garden soil suspension. Upon seven days of incubation under shaking condition (150 rpm) at 30°C. Cultures were isolated on starch agar medium. Those colonies showing amylase activity were isolated and purified. Of five cultures isolated, BS₃ was selected for further studies as it showed maximum amylase production.

Degradation of LDPE/Na-PCMS Blend

50 gm of the above blend were taken in each 1000 ml flask containing sterile mineral medium (pH 7). To it 10% v/v of culture suspension of isolated BS₃ were added. All the flasks were kept on shaker (150 rpm) and every alternate day one flask was removed and analysed for dry weight of blend, total cellular protein, tensile strength and percent elongation.

Abiotic controls included blend in medium without culture. Above sets were in duplicates and results are the average of reading obtained.

Weight Loss

Blends were filtered through whatman filterpaper no. 1. The residue was washed with ethanol and dried at 40°C in vacuum oven till constant weight.

Total Cellular Protein

1 ml suspension was centrifuged at 7000 rpm for 10 minutes. The cell pellets were washed with normal saline. The cells were then suspended in 1N NaOH and boiled for 10 minutes to disrupt the cells. It was then centrifuged to remove cell debris. The supernatant was assayed for protein by lowry's method [10].

RESULTS AND DISCUSSION

Mechanical Properties

In the earlier paper we have discussed the synthesis and characterization of LDPE/Na-PCMS blends of two different DS 0.21 and 0.58 [8]. Evaluation of mechanical properties and effect of DS were also discussed. Here we report another two types of Na-PCMS blends of DS 0.314 and 1.10 with LDPE. The comparison of mechanical properties of above four blends are tabulated in Table I. From the comparison of the properties we conclude the following results.

All the blends had melt flow index (MFI) values greater than LDPE. As the DS increases MFI gradually increases. This indicates the lower melt viscosity of the blends which is advantageous from the processing point of view.

Tensile strength [TS] showed different trend. There was a rise in the TS up to 20% addition of Na-PCMS for all DS. Further addition of Na-PCMS lowered the TS for DS 0.21, 0.314, 0.58 while nearly no change were observed for DS 1.10 up to 30% addition of Na-PCMS. The higher values of per cent elongation is attributed to addition of Na-PCMS to all the blends.

From the above results it is clear that optimum properties are obtained in DS 1.10. So it has been selected for biodegradation study.

TABLE I Mechanical properties of the blend

Composition LDPE/	Melt flow index Gms/10 min. D.S.				Tensile strength Kg/Cm ² D.S.				% elongation % D.S.			
	0.21	0.314	0.58	1.10	0.21	0.314	0.58	1.10	0.21	0.314	0.58	1.10
<i>Na-PCMS</i>												
100/0	0.5	0.5	0.5	0.5	31	31	31	31	100	100	100	100
90/10	0.55	0.55	0.55	0.58	31	31	32	33	110	105	110	120
85/15	0.61	0.62	0.76	0.81	32	33	34	36	118	120	125	135
80/20	0.78	0.77	0.82	0.99	32	33	34	36	128	128	133	155
75/25	0.85	0.90	0.91	1.00	30	31	35	36	131	140	150	162
70/30	0.99	1.01	1.04	1.16	28	29	30	36	138	145	160	163

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Biodegradation

Five different bacteria were isolated viz. BS₁, BS₂, BS₃, BS₄, BS₅. Among them BS₃ was found to be maximum amylase producer (data not shown) and hence was used for further studies. BS₃ is gram positive motile, nonspore forming bacilli. Results are shown in Table II. With BS₃, 32% loss in dry weight of blend was obtained within 18 days of incubation. Blends consisted of 30% of Na-PCMS with 70% of LDPE. Thus it seems that Na-CMS from the blend is completely utilized by the isolated BS₃.

Furthermore, total cellular protein was found to increase up to 8 days which reflects increase in biomass after which growth was found to cease. This indicates that extra-cellular enzymes are involved in the degradation of blends.

Tensile Strength and Percent Elongation

TS and percent elongation of blend were measured after every six days degradation. They are tabulated in Table III. TS and percent elongation decreased with increase in degradation time. After 18 days of degradation TS was reported lower than that of the pure LDPE.

Scanning Electron Microscopy

The test samples of LDPE/Na-PCMS blends before and after degradation were examined with ESEM TMP, phillips model scanning electron microscope.

TABLE II % weight loss and total cellular protein

<i>Time (days)</i>	<i>% weight loss %</i>	<i>Total cellular protein ug/ml</i>
0	0.0	136.8
2	10.0	273.6
4	17.6	296.4
6	20.0	296.4
8	24.0	570.0
10	26.0	303.0
12	28.0	303.0
14	29.0	240.0
16	31.0	238.4
18	32.0	237.0

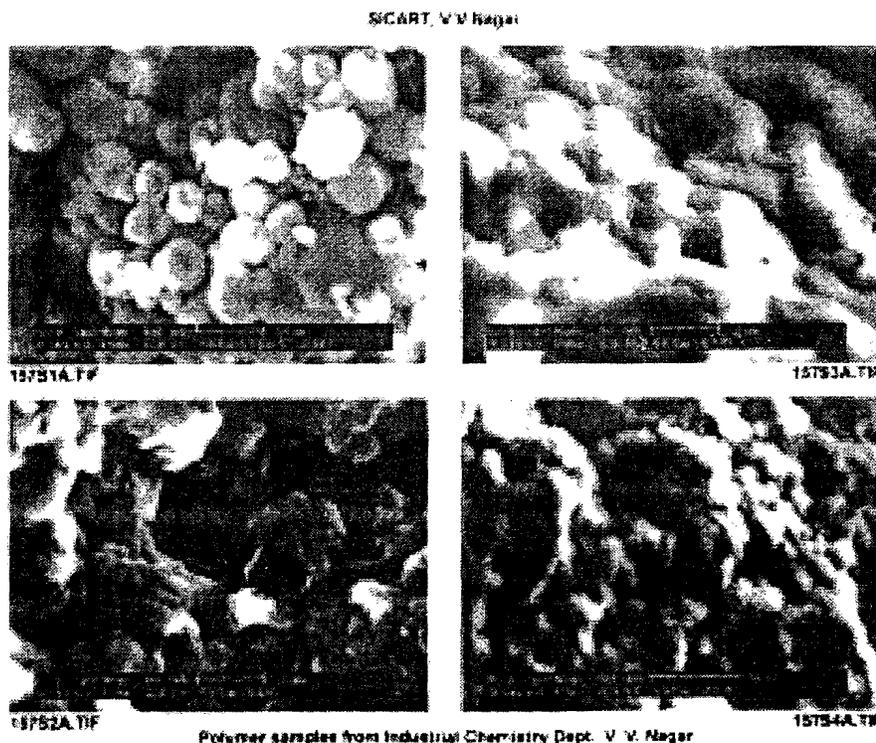
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TABLE III Tensile strength and % elongation after degradation

<i>Degradation time (days)</i>	<i>Tensile strength Kg Cm²</i>	<i>% elongation %</i>
0	36	163
6	34	150
12	32	141
18	28	127

The microphotograph of the LDPE/Na-PCMS blend containing 30 wt% Na-PCMS (DS 1.10) for different degradation time are shown in figure

- 1(a) → Before degradation
- 2(a) → After 6 days degradation
- 3(a) → After 12 days degradation
- 4(a) → After 18 days degradation



FIGURE

Before the attack of BS_3 , the overall surface of the blend is clear and homogeneous. After 6 days, some holes appeared due to degradation of Na-PCMS within the blends and overall surface of the blend was ruptured. The consumption of Na-PCMS within the blend by BS_3 continued during 12 days and also after 18 days. Relatively large holes appeared suggesting more and more consumption of Na-PCMS by BS_3 . Na-PCMS is the main carbon source for the BS_3 by the SEM.

CONCLUSION

From the above study we may conclude that LDPE/Na-PCMS blends with 70% LDPE and 30% Na-PCMS (1.10) have better mechanical properties and compatibility. So we have selected this blend for biodegradation by bacillus species BS_3 . After 18 days of degradation it seems that all the Na-PCMS and little amount of LDPE were degraded because around 32% weight loss was observed and the tensile strength was less than that of pure LDPE. The surface rupture and porosity were clearly shown in SEM of the degraded blends.

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