Characterization of Linear Low-Density Polyethylene/ Poly(vinyl alcohol) Blends and Their Biodegradability by *Vibrio sp*. Isolated from Marine Benthic Environment

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ABSTRACT: Increasing amounts of plastic waste in the environment have become a problem of gigantic proportions. The case of linear low-density polyethylene (LLDPE) is especially significant as it is widely used for packaging and other applications. This synthetic polymer is normally not biodegradable until it is degraded into low molecular mass fragments that can be assimilated by microorganisms. Blends of nonbiodegradable polymers and biodegradable commercial polymers such as poly (vinyl alcohol) (PVA) can facilitate a reduction in the volume of plastic waste when they undergo partial degradation. Further, the remaining fragments stand a greater chance of undergoing biodegradation in a much shorter span of time. In this investigation, LLDPE was blended with different proportions of PVA (5–30%) in a torque rheometer. Mechanical, thermal, and biodegradation studies were carried out on the blends. The biodegradability of LLDPE/PVA blends has been studied in two environments: (1) in a culture medium containing *Vibrio sp*. and (2) soil environment, both over a period of 15 weeks. Blends exposed to culture medium degraded more than that exposed to soil environment. Changes in various properties of LLDPE/PVA blends before and after degradation were monitored using Fourier transform infrared spectroscopy, a differential scanning calorimeter (DSC) for crystallinity, and scanning electron microscope (SEM) for surface morphology among other things. Percentage crystallinity decreased as the PVA content increased and biodegradation resulted in an increase of crystallinity in LLDPE/PVA blends. The results prove that partial biodegradation of the blends has occurred holding promise for an eventual biodegradable product. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 257–265, 2012

Key words: biodegradable; *vibrio sp*; poly(vinyl alcohol); morphology; differential scanning calorimetry

INTRODUCTION

Conventional polymers such as polyethylene (PE) and polypropylene (PP) persist for many years in the environment after disposal. It has been estimated that polyethylene biodegrades less than 0.5% in 100 years and about 1% if pre-exposed to sunlight for 2 years.[1] Since polyolefines such as PE constitute the vast majority of plastic materials used, especially for packaging applications (Fig. 1), it is important to devise ways to improve their biodegradability.

Attempts to produce environmentally degradable, low cost, plastic materials from polyolefines date back to the second half of the 20th century.[2] Synthetic polymers like polyolefins are not degraded by microorganisms in the environment which property is responsible for their long life and an ever increasing volume of plastic waste.[3] These polymers are characterized by high molecular weight, chemical inertness, hydrophobicity, reduced surface area, relative impermeability to oxygen etc. which make them resistant to microbial attack and at the same time impose enormous restrictions on the design and development of biodegradable polymers.[4,5] Therefore, recycling and degradation of plastics is an important issue for environmental and economic reasons.[6,7]

Griffin[8,9] introduced the idea of increasing the biodegradability by adding a biodegradable ingredient to the polymer material. When a biodegradable component is present, microorganisms can readily attack it thereby increasing the porosity of the material and resulting in a mechanically weakened film. The surface area will increase making it more susceptible than the original material to all degradation processes including biodegradation.[10] Partially biodegradable polymers obtained by blending biodegradable and nonbiodegradable polymers undergo considerable loss of volume over a period of time thus effectively reducing the environmental hazards.

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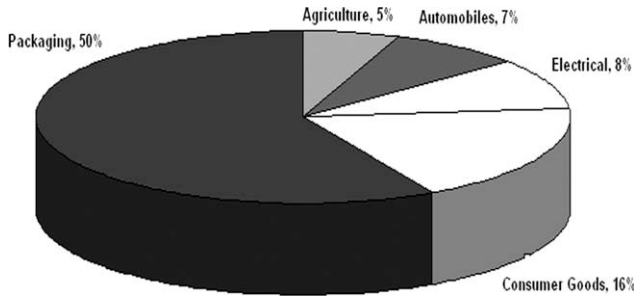

Figure 1 The percentage of polyolefines used for various applications.

They are more useful than completely biodegradable ones due to economic advantages and the superior properties of the nonbiodegradable component.

In general, the degradation of polymers and polymer composites in the environment may proceed in four ways: microbial action, degradation by macroorganisms, photo degradation, and chemical degradation.[11] Biologically initiated degradation is facilitated by prior chemical degradation. Microorganisms produce a great variety of enzymes which are capable of reacting with natural and synthetic polymers. The enzymatic attack on the polymer is a chemical process induced by the organisms in an attempt to secure nourishment (carbon source). The microbial attack of polymers occurs over a rather wide range of temperatures, 60–70°C not being uncommon.[12]

Poly(vinyl alcohol) (PVA) is the only carbon–carbon backbone polymer that is biodegradable under both aerobic and anaerobic conditions.[13] PVA is widely used in the industrial field and recently it has attracted increasing attention as a water-soluble biodegradable polymer. Poly(vinyl alcohol) (PVA) was initially developed for fiber application.[14] However, it is now used for high-value added packaging films due to the antistatic properties together with the excellent barrier properties.[15] PVA is also used for fabric sizing, paper coating, adhesives, and suspension agent for suspension polymerization. It is used as a moisture barrier film for food supplement tablets and for foods that contain inclusions or dry food with inclusions that need to be protected from moisture uptake. Most recently, PVA has been used in pharmaceutical and biomedical applications for controlled drug release tests due to its degradable and nontoxic nature.[16]

The global production of PVA is estimated to be 650,000 tons/year, and the large amount of discharged PVA from industrial effluents has become a significant pollution problem.[17] The degradation of PVA by microbes was reported more than 60 years ago,[18] and previous studies could detect PVA-degrading bacteria from only 4 of 100 soil samples screened.[19] PVA is completely degraded and utilized by a bacterial strain, Pseudomonas O-3, as a soul source of carbon and energy. However, PVA-degrading microorganisms are not ubiquitous within the environment. Almost all the degrading strains belong to the genus *Pseudomonas* although some do belong to other genera.[20] Generally, the carbon–carbon linkage of PVA is degraded either by the enzymes dehydrogenase or oxidase, which is further degraded by the action of hydrolase or aldolase to form simple compounds. In this study, PVA degrading *Vibrio sp*. were isolated from marine benthic environment and biodegradation of the samples were carried out using a consortium consisting of four PVA degrading *Vibrio sp*.

Linear low-density polyethylene (LLDPE) is the common name for copolymers of ethylene with α-olefins: butene, hexene, octene, and 4-methyl-1-pentene.[21] LLDPE has higher tensile strength and higher impact and puncture resistance than LDPE. It is very flexible and elongates under stress. It can be used to make thinner films, with better environmental stress cracking resistance. It has good resistance to chemicals and ultraviolet radiation. It has also good electrical properties. However, it is not as easy to process as LDPE, has lower gloss, and narrower range for heat sealing. It has been shown,[22–25] that such LLDPE often exhibit high heterogeneity in the intermolecular distribution of comonomer units along the polymer chains.

In this study, we have attempted to improve the biodegradability of LLDPE by blending it with a biodegradable component. LLDPE was blended with poly (vinyl alcohol) (5–30%) in a torque rheometer (Thermo Haake) and mechanical, thermal, and biodegradation properties were investigated. The biodegradability of LLDPE/PVA blends has been studied in two environments, viz. (1) a culture medium containing *Vibrio sp*. and (2) a soil environment over a period of 15 weeks.

EXPERIMENTAL

Materials

Film grade LLDPE (LL20FS010) used in this study was supplied by Reliance Industries, Mumbai, India. It has a melt-flow index of 1 g/10 min at 190°C and a 2.16 kg load. The density of the LLDPE sample is 0.920 g/cm^3. Hot water soluble polyvinyl alcohol used in this study was industrial grade obtained from Rolex Chemical Industries, Mumbai. Molecular formula is $(C_4H_{10}O)_n$, viscosity at 4% concentration in water at 20°C is 3 mPa s.

Blend preparation

Blending was carried out in a Thermo HAAKE Polylab System equipped with a pair of roller rotors at

195°C. The rotor rpm was maintained at 50 and LLDPE was added first followed by PVA. LLDPE films have been designated as L0, L5, L10, L15, L20, L25, and L30 for pure LLDPE and that containing 5%, 10%, 15%, 20%, 25%, and 30% of PVA, respectively.

Molding

The LLDPE/PVA blends were compression molded into sheets with the help of a flash mold and an electrically heated hydraulic press. The samples were placed in the hydraulic press and initially heated for 1 min without applying any pressure to ensure uniform heat flow through the material and the molding was subsequently completed by applying pressure for 2 more minutes. The molding temperature and pressure were 195°C and 200 MPa, respectively. The sheet thus obtained was removed from the press after cooling to room temperature.

Mechanical properties

Tensile properties were studied according to ASTM D 882-97,[26] in a Universal Testing Machine (Shimadzu Autograph AG-I Series). Six samples of each specimen were strained at a rate of 50 mm/min at room temperature and average values of tensile strength, elastic modulus, and elongation at break were determined.

Degradation studies

The degradation studies on the samples were studied in two environments: garden soil and culture medium containing *Vibrio sp*. isolated from marine benthic environment.

Biodegradation in culture

Biodegradation of the samples were carried out using a consortium consisting of four PVA degrading *Vibrio sp*. isolated from benthic marine environment, according to ASTM D 5247-92.[27] The consortium comprised of the strains, BTTV4, BTTC10, BTTC27, and BTTN18. The inoculum was prepared as follows: the individual isolates of the consortium were grown overnight at 37°C, at 120 rpm in nutrient broth, pH 7.0 ± 0.3 with 1% NaCl. The pooled isolates were made up to 1 OD at 660 nm and used to inoculate PVA medium composed of 0.5% NH_4NO_3, 0.1% KH_2PO_4, 0.05% $MgSO_4{\cdot}7H_2O$, 0.05% NaCl, and 0.02% yeast extract (pH-7).[28] Incubation was done in an Orbitek environmental shaker (Scigenics, Chennai, India) at 37°C and 120 rpm.

The LLDPE/PVA blends were wiped with 70% alcohol to make them bacteria free. They were subjected to microbial degradation using the consortium of PVA-degraders in the PVA minimal medium,[28] with LLDPE/PVA blends as sole carbon source for a total period of 15 weeks with regular sampling for analyzing the level of degradation. Tensile strength, Fourier transform infrared (FTIR), and scanning electron micrographs (SEM) of these LLDPE/PVA blends were compared with those of the control specimen to indirectly estimate the extent of microbial degradation. The experiments were performed in triplicate with control strips in the same medium without inoculums.

Biodegradation in soil environment

Biodegradability in soil was also tested by burying the samples in garden soil for 15 weeks according to ASTM D5338-98.[29] Before burying, inoculums of PVA degrading microorganisms were applied to the soil. These inoculums were the same as that used in the culture medium studies.

FTIR spectroscopy

FTIR spectra of virgin LLDPE and LLDPE/PVA blends (films) were obtained in FTIR-Avatar 370 (Thermo Nicolet) in the spectral region between 4000 and 400 cm^{-1}.

Thermogravimetric analysis

The thermogravimetric (TG) and derivative thermogravimetric (DTG) analysis of the samples were carried out on a TGA Q-50 (TA Instruments) thermal analyzer under nitrogen atmosphere. Sample weights varied from 5 to 15 mg. The thermograms were recorded for the range from room temperature to 800°C at a heating rate of 20°C/min. The nitrogen gas flow was 50–70 cm^3/min. The maximum degradation temperature (T_{max}), the onset degradation temperature (T_{onset}), and the percentage residual weight were found out.

Differential scanning calorimetry

Crystallinity studies were conducted using a Differential Scanning Calorimeter TA Q-100(TA Instruments) under nitrogen atmosphere. Samples of 5–10 mg were taken and heated in nitrogen atmosphere from −50 to 150°C at a heating rate of 30°C/min and kept at 150°C for 1 min to erase thermal history. Cooling was then performed at a rate of 10°C/min from 150°C to −50°C followed by a second heating from −50 to 150°C at the same rate, resulting in endothermic curves. The heat of fusion was calculated by integrating the areas under the endothermic curve. The degree of crystallinity was obtained from

the ratio between the melting enthalpy of the samples (ΔH_f) and that of 100% crystalline LLDPE (ΔH_f^0).[30]

$$\%\text{Crystallinity} = \frac{\Delta H_f}{\Delta H_f^0} \times 100$$

Scanning electron microscopy

SEM is used widely to study surface morphologies and biological specimens. SEM permits higher magnification and understanding of surfaces without loss of detail. In addition, due to the depth of field of SEM, the micrographs retain a three-dimensional view of the sample. A SEM (JEOL Model JSM-6390LV) was used in this case to study the surface morphological changes after the biodegradation of the samples. Moreover, SEM was employed to study the fracture surfaces of the biodegraded samples after tensile testing. The fracture ends of the specimen were mounted on a metallic stub and were sputter coated with a thin layer of gold to make it conducting.

RESULTS AND DISCUSSION

Mechanical properties

Figures 2–4 show the variation of tensile strength, elastic modulus, and elongation at break of LLDPE/PVA blends. The tensile strength and elongation at break of these blends generally tend to decrease on addition of PVA. This fall in properties is more noticeable beyond a PVA concentration of 15%. Polyvinyl alcohol is highly hydrophilic as it contains hydroxyl groups on its surface, whereas LLDPE is basically nonpolar. Therefore, in such a system, the formation of strong interfacial bonds like hydrogen bonds is not feasible.[30] The drop in the tensile strength of the blends become more drastic as the PVA content is increased. This is because at higher PVA contents, PVA–PVA interaction becomes more pronounced than PVA–PE interaction. Modulus increased due to the stiffening effect of PVA, as the PVA is stiffer than LLDPE. The hydrogen bond in PVA gives it much higher modulus than semicrystalline polymers such as LLDPE, which has no hydrogen bonding. Therefore, there is a direct relation between the amount of PVA in the blends and the increase of the modulus value.

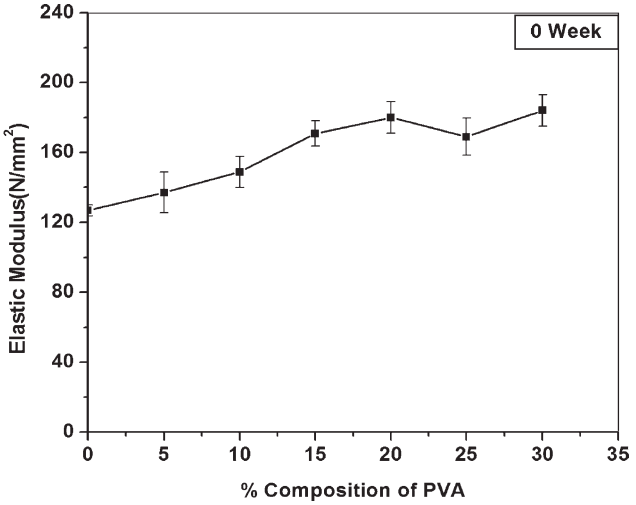

Figure 3 Variation of elastic modulus with the concentration of PVA in LLDPE/PVA blends.

Biodegradation studies

Culture medium

All the *Vibrio sp*. isolates were screened for PVA degradation employing plate assay method (Fig. 5).

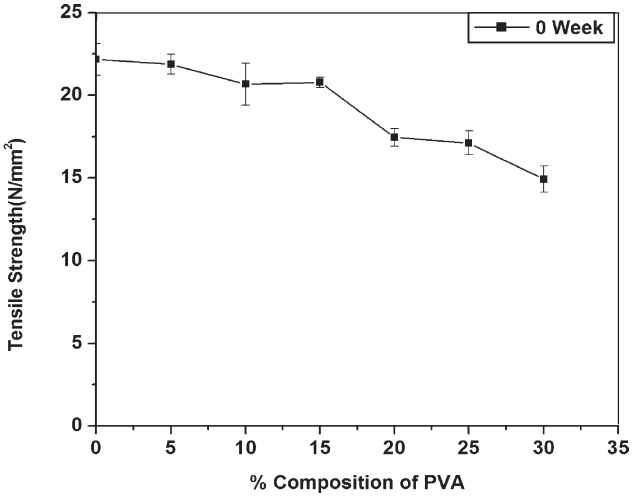

Figure 2 Variation of tensile strength with the concentration of PVA in LLDPE/PVA blends.

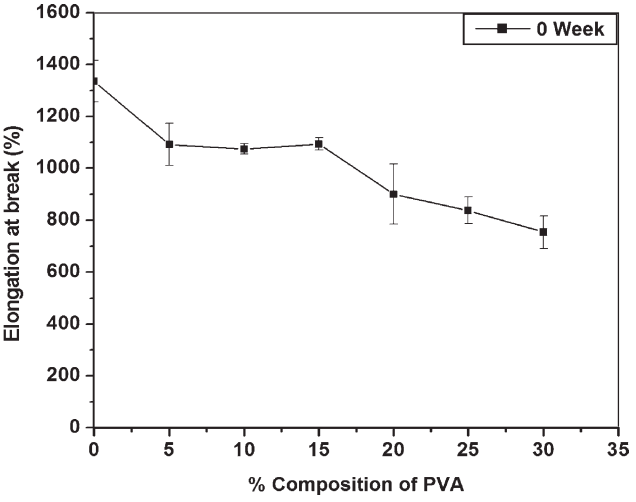

Figure 4 Variation of elongation at break with the concentration of PVA in LLDPE/PVA blends.

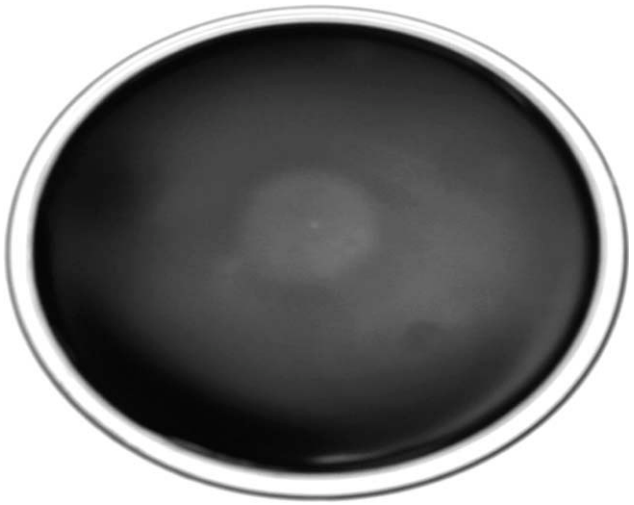

Figure 5 PVA degrading isolate (*V. neries*, strain BTKB4); Zone of clearance surrounding the colony in PVA agar plates after flooding with iodine -boric acid solution indicates PVA degradation.

PVA minimal agar plates were prepared and *Vibrio sp*. was spotted on this plate to detect their PVA degrading ability. The plates were incubated at 37°C for 2 days. The presence of a zone of clearance surrounding the colony after flooding with iodine-boric acid solution,[31] indicates PVA degrading ability.[28] From the results of primary screening, those isolates with large zones of clearance were selected and subjected to further studies.

Polyvinyl alcohol, being a biodegradable polymer, can be used as a carbon source by *Vibrio sp*. As the PVA content is increased, the PVA becomes a continuous phase in the blend and can be easily accessed by the organisms. At low PVA contents, the PVA may remain encapsulated in the synthetic polymer matrix, thereby making it difficult for the organisms to access and use it as a source of carbon. In such a case, the bacterial growth will be slow or negligible. The bacterial growth was notably faster in LLDPE/PVA blends than in virgin LLDPE.

The percentage decrease in tensile strength of blends after biodegradation and soil degradation is shown in Table I. The extent of degradation is more in the case of the culture. A maximum percentage loss of tensile strength of 20.16 is noticed in the case of biodegradation in culture. As against this, only a 16.34% loss of is observed in the samples degraded in the soil. Both sets of samples show a steady loss of tensile strength with passage of time indicating the biodegradability. The decrease in tensile strength of LLDPE/PVA blends after biodegradation in culture is shown in Figure 6.

TABLE I
The Percentage Decrease in Tensile Strength of Blends After Biodegradation and Soil Degradation

Sample designation	Biodegradation (% loss in tensile strength)	Soil degradation (% loss in tensile strength)
L0	2.39	1.98
L5	13.16	10.60
L10	14.27	10.64
L15	14.68	11.83
L20	17.19	14.84
L25	20.16	15.42
L30	20.09	16.34

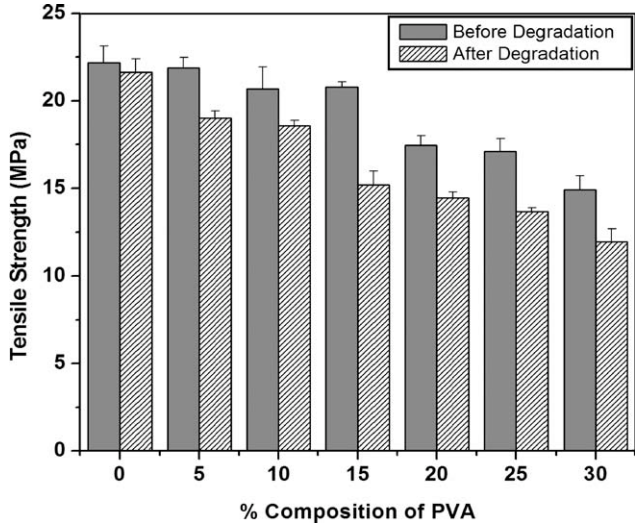

Figure 6 Biodegradability of LLDPE–PVA Blends with respect to tensile strength.

Soil environment

The decrease in tensile strength of LLDPE/PVA blends after soil degradation is shown in Figure 7. Here also, there is decrease in tensile strength indicating degradation by the soil microorganisms. In

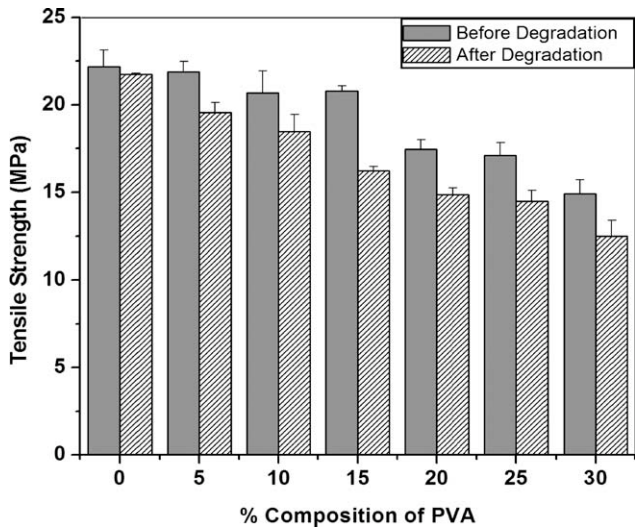

Figure 7 Variation in tensile strength after soil degradation.

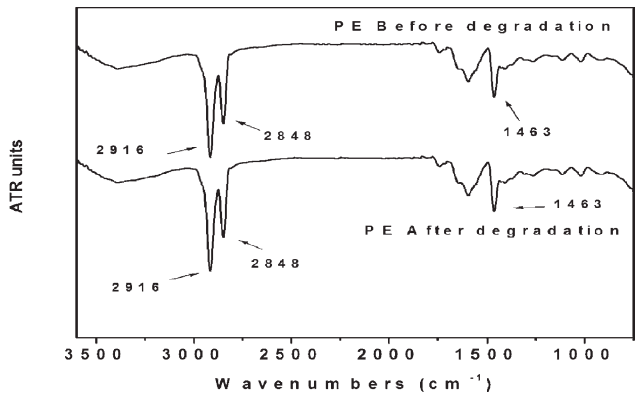

Figure 8 FTIR spectra of neat LLDPE before and after degradation.

the soil, water diffuses into the polymer sample, causing swelling and enhancing biodegradation.

FTIR analysis

The FTIR spectra of virgin LLDPE and LLDPE/PVA blends before and after biodegradation are shown in Figures 8 and 9, respectively. As shown in Figure 9, peak intensities of films at 2916–2848 cm^{-1}, 1463–1473 cm^{-1} after degradation in culture medium have improved considerably. The peaks at 2916–2848 cm^{-1} and 1463–1473 cm^{-1} were due to symmetrical stretching vibration of C—H bonds and bending vibration of middle intensity C—H bond. The increase in the intensity of these peaks was owing to the fracture of polyethylene chains in degradable environments which resulted in the increase in terminal group numbers.[32] The peaks at 1020–1100 cm^{-1} correspond to C—O symmetrical stretching and C—O asymmetrical stretching vibrations in PVA. The peak at 1643 cm^{-1} also corresponds to PVA. Degradation of PVA led to the increase in intensity of these peaks.

Thermogravimetric analysis

The TG and DTG traces for neat LLDPE and LLDPE/PVA blends in nitrogen atmosphere are

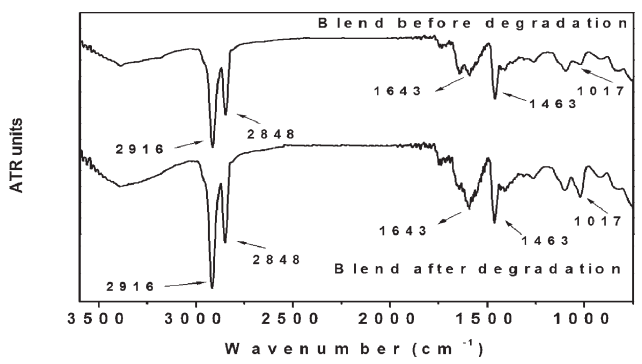

Figure 9 FTIR spectra of LLDPE/PVA blends before and after degradation.

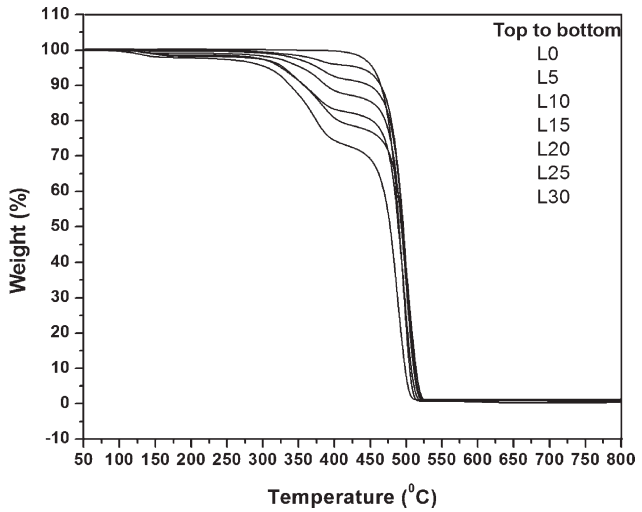

Figure 10 Thermogravimetric analysis curves of neat LLDPE and LLDPE/PVA blends.

shown in Figures 10 and 11, respectively. From the figures, it is evident that all the samples exhibit a two-stage degradation process, except for neat LLDPE. In the temperature range 220–420°C, there is a decrease in the weight of the LLDPE/PVA blend which is not noticed in the case of neat LLDPE. This first degradation stage is due to water evaporation from the decomposition of hydroxyl groups. The second degradation stage between 420 and 520°C is due to chain scission of the carbon–carbon bonds in the main chain. Thus, it is clear that there is a very different trend in the degradation of LLDPE/PVA blends from that of virgin LLDPE. This may indicate that PVA does not affect the degradation process of the blends due to weak interactions between the two components. The two separated DTG maxima in the blend indicate that a separation phase has occurred between the LLDPE and PVA components. The

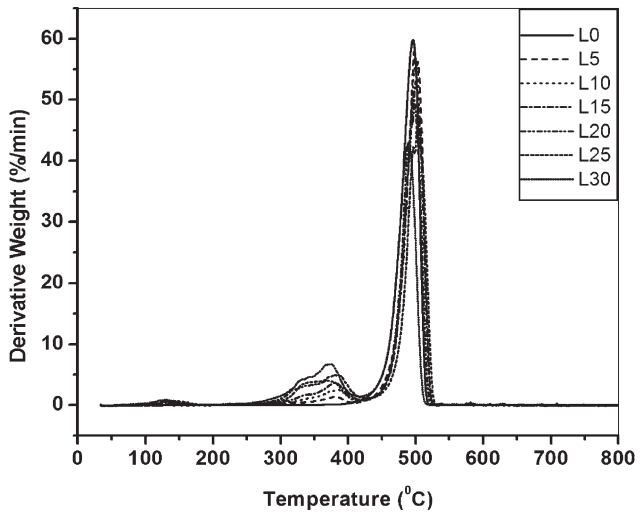

Figure 11 Derivative thermogravimetric curves of neat LLDPE and LLDPE/PVA blends.

TABLE II
TGA Data of Virgin LLDPE and LLDPE/PVA Blends. The Maximum Degradation Temperature (T_{max}), the Onset Degradation Temperature (T_{onset}), and the Percentage Residual Weight Are Shown

Sample designation	Maximum degradation temperature (T_{max})	Onset degradation temperature (T_{onset})	% Residual weight
L0	495.99	448.75	1.781
L5	490.44	435.19	1.168
L10	495.88	442.96	1.263
L15	496.29	446.90	1.058
L20	495.73	447.77	.9153
L25	496.64	448.70	.5718
L30	491.67	451.78	.4336

maximum degradation temperature (T_{max}), the onset degradation temperature (T_{onset}), and the percentage residual weight are shown in Table II. The maximum degradation temperature of the blend is almost the same as that of pure LLDPE. This means that addition of PVA to LLDPE does not adversely affect the thermal stability of LLDPE. The residual weight decreases as the PVA content is increased.

Differential scanning calorimetry studies

Crystallinity is a state of greater order implying a long range periodic geometric pattern of atomic spacings. In semicrystalline polymers such as polyethylene, the degree of crystallinity influences the degree of stiffness, hardness, and heat resistance.[33] Crystallinity can be calculated from a differential calorimetry curve by dividing the measured heat of fusion by the heat of fusion of 100% crystalline material.

LLDPE is a semicrystalline thermoplastic polymer which, on application of heat, undergoes a process of fusion or melting wherein the crystalline character of the polymer is destroyed. Although polymers melt over a temperature range due to differences in the size and regularity of the individual crystallites, the melting point of a polymer is generally reported as a single temperature at which the melting of the polymer is complete. An additional parameter for the characterization of LLDPE is its percent crystallinity.

DSC scans of LLDPE and LLDPE/PVA blends were recorded and enthalpy of fusion (ΔH_f), enthalpy of crystallization (ΔH_c), melting temperature (T_m), and % crystallinity of LLDPE and LLDPE/PVA blends were evaluated The percent crystallinity was calculated on the assumption that the heat of fusion of 100% crystalline LLDPE is 289 J/g.[34] The blend shows a fall in heat of fusion compared with virgin LLDPE. On this basis, a lower degree of crystallinity is observed for the LLDPE/PVA blend. The lowering of crystallinity in LLDPE/PVA blends is understandable in the light of the intrusion of the bulky PVA groups into the crystallite formation,[35,36] and the hydrophilic nature of PVA. Heat of crystallization is also seen to be lower for the blends compared with LLDPE. The melting and crystallization temperatures of LLDPE (Table III) have not been affected to any serious extent by the presence of PVA in the concentration range studied. Hence, it can be safely assumed that the interaction between LLDPE and PVA is minimal.[37]

TABLE III
DSC Analysis Data of Neat LLDPE and LLDPE/PVA Blends. The Melting Temperature (T_m) and Crystallization Temperature (T_c) of Neat LLDPE and LLDPE/PVA Blends Are Shown

Sample designation	L0	L5	L10	L15	L20	L25	L30
T_m (°C)	123.43	123.65	123.51	123.36	123.38	123.41	123.40
T_c (°C)	108.62	111.76	111.41	111.56	111.91	111.97	112.28

TABLE IV
DSC Comparison Data of Blends Before and After Degradation Obtained from the Second Heating Thermograms: A-Before Degradation, B-After Degradation

Sample designation	Heat of fusion (J/g)		% Crystallinity	
	A	B	A	B
L0	96.35	96.93	33.34	33.54
L5	91.88	92.69	31.79	32.07
L10	86.71	89.75	30.00	31.06
L15	83.20	87.03	28.79	30.11
L20	78.74	84.21	27.25	29.14
L25	73.68	78.11	25.29	27.03
L30	68.16	73.93	23.58	25.58

After 15 weeks of incubation in the culture medium containing *Vibrio sp*., the biodegraded samples were again analyzed for crystallinity. The results obtained for all compositions of LLDPE/PVA blends are tabulated in Table IV. From the Table, it is clear that as the PVA content increases the heat of fusion and crystallinity decrease for LLDPE/PVA blends. In all the composition of blends, the heat of fusion and crystallinity increased after biodegradation. Albertsson[38] and Kestelman et al.[39] noticed a similar change in the crystallinity of polyethylene after biodegradation.

Surface morphological studies

Figure 12 shows the SEM photographs of neat LLDPE and LLDPE/PVA blends before and after degradation in culture medium containing *Vibrios sp*.

There are no notable changes in morphology of virgin LLDPE when subjected to microbial attack. SEM shows that surfaces of LLDPE/PVA blends before biodegradation exhibit a rough surface with no surface defects. The surface roughness increases

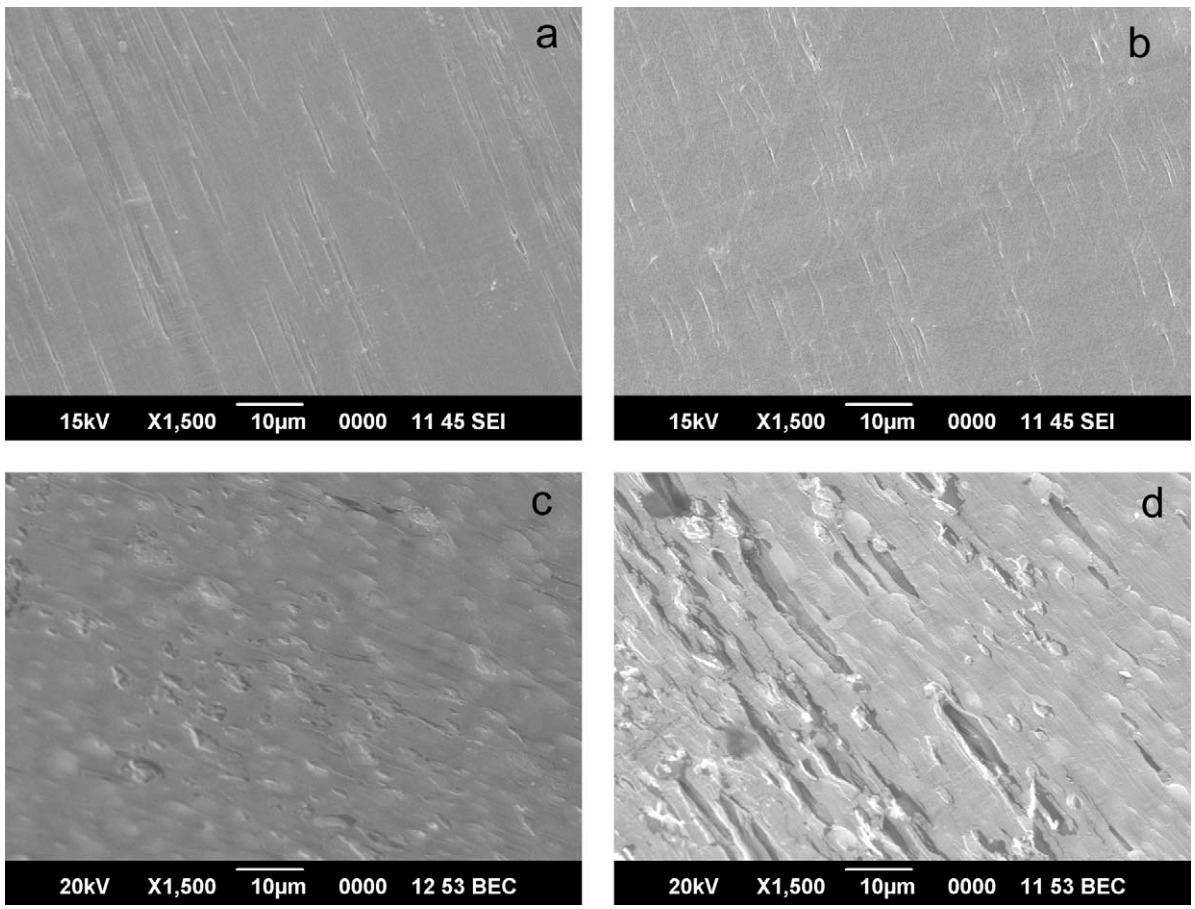

Figure 12 SEM photographs: (a) neat LLDPE before degradation; (b) neat LLDPE after degradation; (c) LLDPE/PVA blend before degradation; (d) LLDPE/PVA blend after degradation.

in the blends with degradation. There are cracks and grooves visible in the blends after degradation in culture medium containing *Vibrios*. These defects are the result of consumption of the biodegradable component by microbes. Such surface destruction can facilitate the transport of degrading factors to the polymer bulk and further accelerate degradation occurring in the environment.

CONCLUSIONS

The blending of PVA with LLDPE has lead to some loss of mechanical properties such as tensile strength and elongation at break. But the modulus of the blends increased as the PVA content is increased. TG analysis showed that, addition of PVA does not adversely affect the thermal stability of LLDPE as the maximum degradation temperature of the blend is almost the same as that of neat LLDPE. When these blends are subjected to biodegradation in soil environment and a culture medium, microbial activity is indicated by fall in tensile properties and surface defects. Biodegradation in the culture is more rapid than soil degradation. The blends are remarkably more biodegradable than pure LLDPE. The study has established that blending with PVA has improved the biodegradability of LLDPE. The FTIR analysis and SEM studies of the degraded samples give ample evidence for degradation of LLDPE/PVA blends. Biodegradation resulted in an increase of crystallinity in LLDPE/PVA blends which is obtained from DSC analysis. Hence, these blends are promising materials for environment friendly polymer applications.

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