



Deterioration study of Cellulose (15%) + Polyethylene glycol (7%) in LDPE Bio-composite thin film

Amit L. Gadre^{1,a*}, Ganesh R. Yerawar², Manisha C. Golchha³, Vijaya Sangawar⁴, Dhananjay. P. Deshmukh⁵ & Bharat B. Madavi¹

¹Department of Physics, A. D. Mahavidyalaya, Bharsingi (M.S.) INDIA

²Department of Physics, Arts, Commerce and Science College, Arvi (M.S.) INDIA

³Department of Physics, Brijlal Biyani Science College, Amravati (M.S.) INDIA

⁴Polymer Research Laboratory, G.V.I.S.H. Amravati (M.S.) INDIA

⁵Department of Physics, Late R. Bharti Arts, Comm. & S. S. R. Bharti Science College, Arni (M.S.) INDIA

* Corresponding Author Email: amitgadre09@gmail.com

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Abstract:

Plastics take literally hundreds of years to decompose and poisoning the environment and the wildlife living in it in the meantime. The aim of this research paper to prepared and characterized study of Cellulose (15%) + PEG (7%) added in LDPE bio-composite thin film in compostable soil burial period of 90 days. This bio-composite film were characterized by weight loss and percent study, UTS and PEB measurements, SEM and XRD before and after degradation in order to study biodegradation of CEL (15%) + PEG (7%) added in LDPE thin film samples in natural condition. The CEL (15%) + PEG (7%) in LDPE bio-composite thin films after 90 days of soil burial are found to be deteriorated and makes an environment-friendly degradable material in natural condition.

Keywords: Biodegradation, LDPE, PEG, CEL, XRD, SEM, UTS, PEB.

Introduction:

Plastic pollution has contributed to a heightened level of ecological detriment as a result of the excessive use of polymeric materials. The Low Density PE, High Density PE and PP polymer materials are widely used as packaging materials, such as bags, packaging bottles, containers, and other packaging materials in modern word. These petroleum-derived polymers which are not biodegradable are crafted in a way to fit each purpose in packaging supplies, biomedical, and agricultural purposes, hence the difference in their durability [10]. In view of this, energetic, chemical and biological polymer degrading techniques have been studied extensively during last three decades [12].

Usage of certain microorganisms and enzymes to degrade polymers are classified as the biodegradation method of polymer. The degradation of conventional plastics such as polyethylene, polypropylene, polystyrene, poly (vinyl chloride) and poly (ethylene terphthalate) by the action of natural herbs polymers such as starch, cellulose, lignin is a significant way to accelerate polymer biodegradation [7]. Microorganism breaks the polymeric chain and consumes materials through aerobic and anaerobic process.

LDPE is widely used packaging material, is the worst offender, being highly resistance to degradation and Polyethylene glycol (PEG) is non-

hazardous and biocompatible additive to support degradation [14]. During degradation, the PEG molecules are reduced by one glycol unit at a time after each oxidation cycle and Cellulose (CEL) is organic material likely consumed by microorganisms which enhances the rate of degradation in compostable soil environment. Thus, the aim of the present investigation to study the biodegradation of CEL (15 %) + PEG (7 %) added in LDPE composite thin film in compostable soil burial natural condition.

Materials and Methods:

Commercial grade LDPE with density 0.924 g/cm^3 at 23°C , melt flow index 4 gm/10 min ; melting range $105\text{--}109^\circ\text{C}$ was supplied from Nutan Gujarat Industrial Estate, Vadodara (India). Xylene as solvents of AR Grade and PEG with average molecular weights $3500\text{--}4000$, freezing point $53\text{--}50^\circ\text{C}$ and having pH in between 4.5 to 7.5 is used as provided without further purification. Cellulose powder with pH in between 5.0 to 7.0 obtained from Loba Chemicals, Mumbai. The compostable soil (1:1 proportion) has pH 8.43 before burial.

Sample Preparation:

The Cellulose added PEG/LDPE composite thin films were synthesized by solution evaporation technique [6]. Concentration of Cellulose (15 %) + PEG (7 %) added in LDPE and the sample is coded as (LP7P15C0 and LP7P15C9) before and after 90 days of degradation respectively. The thickness of the film is in range of $15\text{--}30 \mu\text{m}$. A German make microscope supplied by Paul Waechter, Model No. 641640 was used for thickness measurement [5].

Soil Burial Technique:

A soil burial technique is carried out in natural environment to degrade the sample. For this, a thin rectangular sheet of sample (about $2.5 \text{ cm} \times 12 \text{ cm}$) of $15\text{--}30 \mu\text{m}$ thickness is buried

at the depth of 5 cm in compostable soil (Soil and compost is in 1:1 proportion) having pH 8.43 before and pH 8.34 after 180 days. The compostable soil having pH 8.43 (with $N=588.0$, $P=27.06$, $K=249.09$) before burial of samples. The moisture content was maintained by adding water in soil after regular interval. The samples were removed from soil after 90 days and washed with distilled water and then acetone bath and dried at room temperature.

Result and Discussion:

Weight loss and percent study:

Weight loss of all CEL + PEG added in LDPE thin film samples determined from the weight measurement before and after 90 days of degradation carried out on K-Roy Monopan Balance having accuracy $\pm 0.1 \mu\text{gm}$ and are enclosed in Table 1.

Table 1: Weight loss and weight loss percent of LP7P15C0 and LP7P15C9 samples.

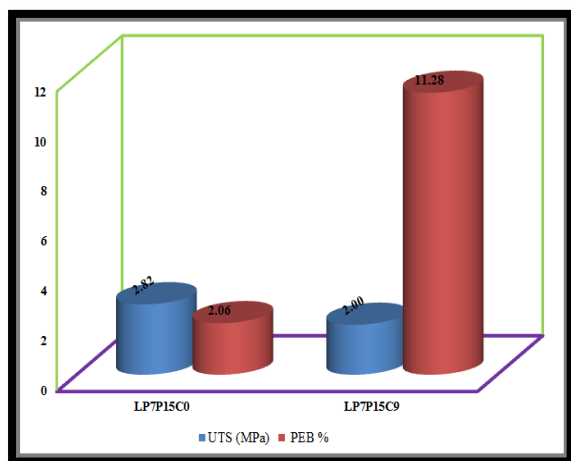
Sample Code (days)	Weight (mg)	Weight Loss %
LP7P15C0 (0 day)	1.560	0
LP7P15C9 (90 days)	1.240	20.51

Table 1 show the weight of thin film sample of LP7P15C0 was 1.560 mg before (0 day) burial in compostable soil. But after 90 days of compostable soil burial, the weight of thin film sample decreases and it becomes 1.240 mg for LP7P15C9 sample with weight loss percent of 20.51% for 90 days of degradation in compostable environment. Thus weight loss and weight loss percent was found to be decreasing after 90 days of degradation. The growth of microorganisms within the polymer leads to an increase in weight where as a loss of

polymer integrity leads to a weight loss. The weight loss is proportional to surface of polymer and it is an indication that biodegradation initiated at the surface [8, 17].

Tensile Strength and Percentage of Elongation Break:

Measurement of mechanical properties such as Ultimate Tensile Strength (UTS) and Percentage Elongation Break (PEB) of samples (LP7P15C0 and LP7P15C9) shown in Bar Graph 1 were carried out using an ASTM D 882 Universal Testing Machine (UTM), Nova Surface-Care Centre Pvt. Ltd. Mumbai.



Bar Graph 1: UTS and PEB values for LP7P15C0 and LP7P15C9 before and after degradation.

Bar Graph 1 shows that ultimate tensile strength of LP7P15C0 (0 days) sample found to be 2.82 MPa and percentage elongation at break value found to be 2.06 % before degradation. But after 90 days of burial in compostable soil, it is found that, UTS value of LP7P15C9 sample gradually decreases and reduced to 70.92 % of before burial sample. PEB value of CEL (15%) + PEG (7%) in LDPE bio-composite sample found to be increase after 90 days of degradation.

Decrement in tensile strength after compostable environment is due to chain scission and cross linking reactions in polymer. Tensile strength may not be

markedly affected by sample embrittlement, whereas, elongation is much more sensitive and can provide evidence of oxidative degradation of polymer [15, 18].

Scanning Electron Microscopy (SEM) Analysis

SEM of all thin film samples were recorded on scanning electron microscope (SEM, ZEISS EVO18), R.T. M. Nagpur University., Nagpur (India). The Photograph of LP7P15C0 (0 day) before weathering sample shows a plane transparent heterogeneous structure with traces of cellulose (CEL) observed on the surface of PEG/LDPE matrix.

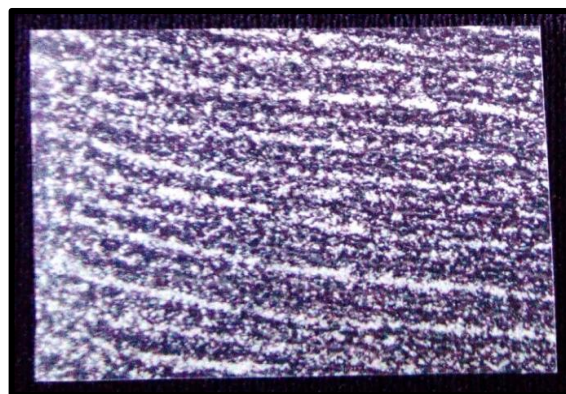


Fig. 1: Photograph of LP7P15C0

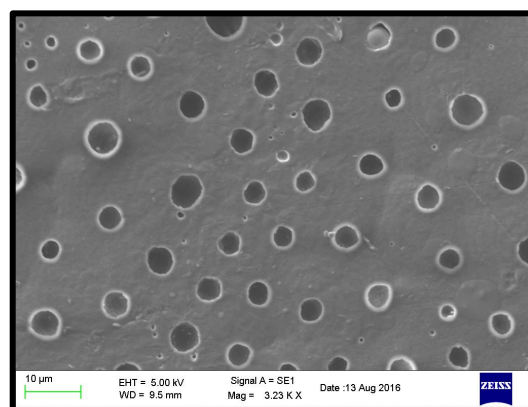


Fig. 2: SEM of LP7P15C9

In SEM photograph LP7P15C9 small pores or circles of irregular shape and size are seen on the surface of thin film after 90 days of compostable soil degradation. These pores found on

compostable sample after burial are created by micro-organisms present in compostable soil indicate that, CEL/PEG added in LDPE matrix was first attacked by the microorganisms such as fungi and bacteria present in compostable soil. The SEM photograph of CEL/PEG added in LDPE sample study shows that, addition of plasticizer (PEG) to composites enhances their dispersion in continuous LDPE phase and facilitates direct contact between cellulose fiber and LDPE matrix. Tensile strength of LDPE-Cellulose added PEG composites decreases as cellulose content increases [16]. This result is also coinciding with weight loss and mechanical properties result of bio-composite sample.

X-ray Diffraction (XRD)

X-ray diffraction (XRD) data of all thin film samples were recorded on Bruker AXS D8 Advance, STIC Cochin University, Cochin, Kerala (India).

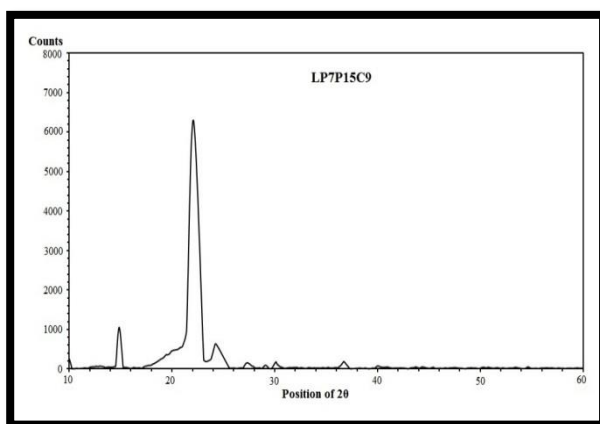


Fig. 3: XRD patterns for LP7P15C0 samples.

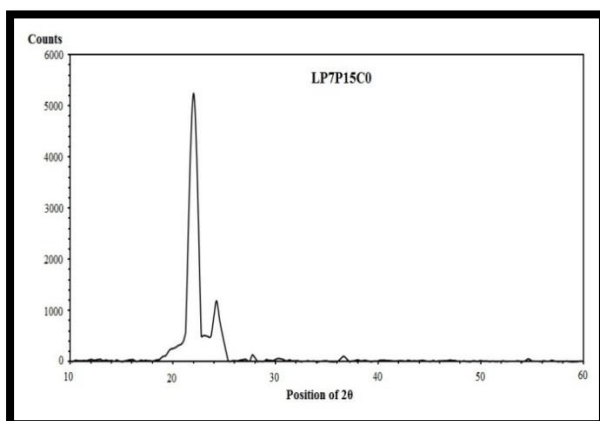


Fig. 4: XRD patterns for LP7P15C9 samples.

The XRD pattern of LP7P15C0 sample shows first prominent peaks at $2\theta = 21.99^\circ$ and second small peak at $2\theta = 24.25^\circ$ corresponding to d-spacing of 4.04 \AA and 3.67 \AA respectively. But after 90 days of compostable sample LP7P15C9 shows first prominent peaks at $2\theta = 22.07^\circ$ and second small peak at $2\theta = 24.31^\circ$ corresponding to d-spacing of 4.023 \AA and 3.66 \AA respectively. The LP7P15C9 sample shows that XRD peak shifts to higher 2θ values and corresponding d-spacing values decreases with increasing days of degradation. So after compostable soil burial of CEL (15%) + PEG (7%) in LDPE degraded samples it is revealed that, intensity of XRD peak slight increases after 90 days of degradation than before burial samples. Thus biodegraded samples show crystalline phase along with amorphous phase after degradation. Decrement in d-spacing and intensity of XRD peak with weathering duration in compostable soil decreases crystallinity and support biodegradation of CEL and PEG/LDPE matrix [1, 4].

Weight loss and percent study revealed that weight of CEL (15%) + PEG (7%) in LDPE samples goes on decreasing after 90 days of degradation. The growth of microorganisms within the polymer leads to an increase in weight where as a loss of polymer integrity leads to a weight loss. The weight loss is proportional to surface of polymer and it is an indication that biodegradation initiated at the surface. Mechanical properties result indicate that UTS value of LP7P15C9 sample gradually decreases and reduced to 70.92 % of before burial sample and PEB value found to be increase after 90 days of degradation. Decrement in tensile strength after compostable environment is due to chain scission and cross linking reactions in

polymer. Tensile strength may not be markedly affected by sample embrittlement, whereas, elongation is much more sensitive and can provide evidence of oxidative degradation of polymer. SEM study shows that, small pores or circles of irregular shape and size are seen on the surface of thin film after 90 days of compostable soil degradation. It is an indication that, CEL/PEG added in LDPE matrix was first attacked by the microorganisms such as fungi and bacteria present in compostable soil. XRD results revealed that, after compostable soil burial of CEL (15%) + PEG (7%) in LDPE degraded samples, intensity of XRD peak slight increases after 90 days of degradation than before burial samples. Thus biodegraded samples show crystalline phase along with amorphous phase after degradation.

Conclusion:

From above Mechanical property (UTS and PEB), SEM, XRD and Weight loss study it is conclude that, Cellulose and plasticizer PEG added in LDPE affect LDPE matrix and support biodegradability of bio-composite film in compostable environment.

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