

Studies on Mechanical, Optical, Barrier Properties and Characterization of LDPE-PLA Blend Films with Micro Clay

D.K. Thirumalazhagan¹, S. Soundararajan^{2*}, SK Nayak³

Abstract

Low density poly ethylene (LDPE) and Poly lactic acid (PLA) in the ratio of 70:30 wt% were blended along with ethylene glycidyl meth acrylate (EGMA) as compatibilizer (2.5%), benzophenone as photodegradable additive (1%) and microclay (1,3,5%) as filler. Blending took place in a twin-screw extruder and then the resulting blend was processed into a film using blown film extrusion. Mechanical, optical, barrier and degradative properties of film were tested as per ASTM Standards. The sample with 5% microclay has better mechanical properties than samples with 1% and 3% microclay; however, the 5% sample also has lower luminous transmittance and higher haze. OTR and WVTR were lowered with the addition of clay. SEM shows that the microclay is distributed/dispersed well. The EGMA works as good reactive compatibilizer and hence uniform flow, uniform phase behaviour and uniform properties is obtained. Hence this LDPE-PLA blend is miscible one. LDPE-PLA blend with microclay will be photo /biodegradable in the presence of Benzophenone and PLA in the environment because LDPE in presence of benzophenone is photodegradable and PLA is biodegradable.

Keywords: LDPE, PLA, EGMA, Microclay, Mechanical, optical, Barrier properties and SEM/DSC

INTRODUCTION

The urgent demand for plastics with higher biodegradability had been established by the serious environmental challenges. Despite the fact that biodegradable polymers such as Polylactic acid have a higher biodegradability, they may not be employed on their own due to technological and economic constraints. On contrary, the conventional petrochemical polymers like PE (Poly Ethylene), PP (Poly Propylene), have superior properties but with less environmental degradation.

***Author for Correspondence**
S. Soundararajan

¹B. Tech Student, Department of Plastics Technology, Central Institute of Petrochemicals Engineering and Technology (CIPET), Guindy, Chennai, Tamil Nadu, India

²Manager-Technical, Department of Plastics Technology, Central Institute of Petrochemicals Engineering and Technology (CIPET), Guindy, Chennai, Tamil Nadu, India

³Professor, Director General-CIPET Head Office, Department of Plastics Technology, Central Institute of Petrochemicals Engineering and Technology (CIPET), Guindy, Chennai, Tamil Nadu, India

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As a result, blending petrochemical polymers with biodegradable polymers is a viable strategy to address this problem. Blending of a non-biodegradable polymer with a biodegradable Polymer is a well-established approach employed by many researchers [1–10] for obtaining degradable materials for sustainable development and specific end-uses. Polymer films comprising conventional polymers and biodegradable polymers is found to be an effective way to make environmental friendly plastics while reducing the dependence on use of non-biodegradable plastics as reported by Yoo et al., in 2010 [11].

Poly(lactic acid) similar to polystyrene is comparatively brittle and stiff polymer with low deformation and break. The research by Jacobsen et al., in 1999 [12] have shown that addition of plasticizers such as polyethylene glycol PEG has significantly increased the elongation at break of PLA. However, it also revealed that plasticization of PLA has sacrificed the inherent stiffness of the material limiting its applications for structural applications like rigid Packagings.

In previous study, Yuan and Ruckenstein in the year 1998 [13] has investigated the use of polyurethane as impact modifier to toughen PLA but the solution blending technique used in this research was not economically viable to produce blends on an industrial scale without solvent which may pollute environment. Thus, a cost effective impact modifier with feasible processibility needs to be developed. Previous study by Anderson et al in the year 2003 [14] shown that semi-crystalline PLA has relatively good immiscibility with LLDPE (Linear low density poly ethylene). Hence, these two materials were chosen as matrix and impact modifier. They had performed melt blending on PLA/LLDPE in an effect to toughen PLA and found that the impact strength of PLA was improved with an addition of LLDPE and concluded that they had successfully toughened PLA [14].

Since the cost of PLA is quite high like specialty plastics, it is not economically feasible to use it alone for day to day use as a flexible packaging material without blending with a low-cost commodity flexible packaging polymers like LDPE. The real viscosity of the blend decreased with increasing PLA content below the line of mixing rule and exhibits shear-thinning behaviour over the spectrum of the examined shear rates in PLA-LDPE blends, indicating incompatibility between the two polymers [15].

Further, it was reported that the degradation behaviour of LDPE/PLA blend and they pointed out that the blend of LDPE and PLA degrades faster than the pure LDPE. Ethylene can also be obtained from Bio sources like molasses which after fermentation gives ethanol 7–9% from which ethylene can be obtained by dehydration [16]. The ethylene can be polymerized to LDPE by conventional free radical Polymerization mechanisms using High pressure process. But this LDPE is also not biodegradable like synthetic LDPE. However, this route is of Renewable origin. The common conventional food packaging materials are using a thin layer of plastic film/laminates, which is made of a synthetic polymer, such as Low-Density Poly Ethylene (LDPE). Poly-Lactic Acid (PLA) is a biodegradable polymer that can be substituted to many synthetic polymers. Because the polarity of LDPE and PLA differs, the first step in the research is to graft them with maleic anhydride (MAH) to increase miscibility through reactive compatibility. The FTIR spectra revealed an interaction between LDPE and MAH, as well as an LDPE/LDPE-g-MAH/PLA blend. Increasing PLA content in the PLA/LDPE blend, increased the water absorption and biodegradability and found that blend with 20% PLA content was the optimum composition for environmentally food packaging. The research on polyethylene polymer and PLA composite by Madhu et al., (2014) [17] has used PE-g-MAH and Maleic anhydride graft polyethylene and found that PE-g-MAH was the most appropriate compatibilizer for polyethylene/PLA composite.

In our previous study, using a twin screw extruder with LDPE-g-MAN compatibilizer (3%) and Benzophenone (3%), LDPE was melt mixed with PLA (90:10, 80:20, 70:30, 60:40, 50:50 percent) (wt percent) (Photo degradable additiveA blown film extruder was used to make tubular blown films. The tensile strength improved as the percentage of PLA in the machine direction grew until it reached 40%, after which it declined. The strength was reduced in the transverse direction as the percentage of PLA was increased. The permeability of oxygen gas was similarly reduced as the proportion of PLA increased. With an increase in the proportion of PLA, the luminous transmittance decreased and the Haze increased. In the presence of a compatibilizer, SEM pictures of 60:40 percent LDPE-PLA and 50:50 percent LDPE-PLA reveal that the blends are compatible and distributed well [18]. The LDPE-PLA blends will be highly Photo/Bio-degradable and hence will not contaminate/polute in the environment.

In another study, LDPE-Poly-lactic acid (PLA) (60:40%) was melt blended with nanoclay (1, 2, and 3%) and benzophenone (3%) in a twin screw compounding extruder with maleic anhydride-grafted LLDPE (LLDPE-g-MAN; 3%) as a compatibilizer, and blown films were extruded and used for mechanical, optical, and permeability tests. As the nanoclay percentage was increased (up to 2 wt%), the tensile strength improved and the elongation at break decreased. Tear strength has been improved. The haze level was raised, but the luminous transmittance was reduced. The transmission of water vapour and the permeability of oxygen gas were both reduced. PLA is biodegradable while LDPE with benzophenone is photodegradable. As a result, LDPE-PLA (60:40%) with benzophenone (3%) is photo-/biodegradable in this investigation. Mechanical qualities such as tensile strength, rip strength, and barrier properties improved when nanoclay was added. Furthermore, nanoclay improves the compatibility apart from LLDPE-g-MAN [19].

Most of the commercially available films were produced in LDPE which takes more time to degrade which results in environmental concern. PLA being a biodegradable polymer does not possess the flexible properties equivalent to LDPE. LDPE is having lower cost than that of PLA. Therefore an LDPE-PLA blend will be produced with optimum cost. In this Present study, we have blended LDPE/PLA (70:30%) by compatibilizing with EGMA compatibilizer and with different proportions of microclay (1, 3, 5 wt %) as filler to study the mechanical and barrier properties of LDPE-PLA blend films.

MATERIALS AND METHODS

Materials

The material, that is used, is blown film grade low density poly ethylene (LDPE) (trade name: LDPE 24FS040) with MFI 4 g/10 min was supplied by Reliance Polymers. The Biopolymer used is Poly Lactic Acid (trade name: RAWPLA 3052D) of MFI 14 g/10 min was supplied by Natur Tec India Pvt Ltd, Chennai, TN. The compatibilizer used is Ethylene Glydicyl Meth Acrylate of MFI 5 g/10 min was supplied by M/S Pluss Polymers Pvt Ltd, India. The Photodegradable additive used is Benzophenone was obtained from SD Fine-Chem limited, Mumbai. Microclay is added mainly to increase barrier properties which in turn results in addition of Microclay increases rheological & mechanical properties. Improved tensile and wear properties were observed in microclay-filled films when compared to regular films. Table 1 Shows the composition of Material samples.

Table 1. Composition of Material Samples

Material	Sample		
	<i>I</i>	<i>II</i>	<i>III</i>
LDPE	70% (by weight)	70% (by weight)	70% (by weight)
PLA	30% (by weight)	30% (by weight)	30% (by weight)
EGMA	2.5%	2.5%	2.5%
MICROCLAY	1%	3%	5%
BENZOPHENONE	1%	1%	1%
SILICONE OIL	0.5%	0.5%	0.5%

Twin Screw Extrusion

The samples are heated and mixed in temperature range between 130–190°C as given below in Table 2. The LDPE/PLA with EGMA is mixed in appropriate ratios with microclay and the strands were extruded and cut into granules using chopper.

Table 2. Temperature zone of for Twin Screw compounding

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Die Zone
130 °C	140 °C	150 °C	160 °C	170 °C	180 °C	190 °C

Blown Film Extrusion

The films of different compositions are produced using a blown film extrusion process. The set temperatures of barrel zones are as given in Table 3.

Table 3. Temperature zone of blown film extrusion

Zone 1	Zone 2	Zone 3	Zone 4	Die Zone
160°C	165°C	170 °C	180°C	180°C

MACHINE SPECIFICATION

Blown Film Extrusion machine Specification (Machine Name : Konark, Odisha)

Lay flat width 100 mm-300 mm. Screw L/D is 25 & Screw Diameter is 65 mm.

Die Dia is 325 mm. Die Gap-2.2 mm. Production Capacity : 350 Kg/hr.

Testing of Film Properties

The properties were determined as per ASTM standard [20] except WVTR which was determined as per BS standard. The tensile strength and elongation at break were determined using a UTM (Universal Testing Machine) as per ASTM D882 standard. The tear strength was determined as per ASTM D 1922 standard using an Elmendorf Tear Tester. Dart impact was determined as per ASTM D 1709 standard using falling weight impact-o-meter The COF (coefficient of friction) was determined as per ASTM D 1894 standard with a coefficient of friction tester. Luminous Transmittance (Clarity) was determined as per ASTM D 1003 standard using a clarity meter. MFI was determined as per ASTM D 1238 using a Lloyd MFI Tester (UK). Density was determined as per ASTM D 792 using extrudate obtained from MFI tester and with a weighing balance. WVTR test was determined as per BS 2782 standard.

Biodegradability Test

Biodegradability Test was determined using Soil Burial method in the ambient atmospheres. The film whose biodegradability has to be determined is cut as per specimen dimension (50 mm wide strip) and weighed before allowing it to compost in the soil. After 28–150 days, the loss in weight of the film is measured. This determines the ability of the material to biodegrade in a depth of 50 mm from soil surface.

RESULTS AND DISCUSSION

The test results are shown in Tables 4–7 and Figures 1–5 and are discussed below.

Physical Properties

Density (ASTM D 792)

Density is the ratio of mass to volume of the sample. The samples are weighed as per ASTM standard and the results are tabulated below in Table 4.

Table 4. Density and MFI of LDPE-PLA blend (70:30%) with microclay

S.N.	Sample	Density (gm/cc)	MFI (gm/10 min)
1	LDPE-PLA+MC1%	0.9954	3.1660
2	LDPE-PLA+MC3%	0.9968	2.7528
3	LDPE-PLA+MC5%	1.0142	2.4728

From the results it is clear that the LDPE-PLA film with microclay has higher density than normal LDPE film since microclay has high density due to inorganic in nature of silicate compound.

Thermal Properties

Melt Flow Index-MFI(ASTM D 1238)

The MFI values are determined using MFI tester as per ASTM standards and the results for the three samples are tabulated above in Table 4. From the Table 1 it is noted that as the microclay percentage increases MFI decreases since the filler restricts the flow of polymer and of high viscosity in nature.

Mechanical Properties

Tensile Strength (ASTM D 882)

Tensile strength is determined using Universal Testing Machine (UTM) as per ASTM standards and the tensile strength for all three formulations are tabulated below in Table in Table 5.

Table 5. Mechanical Properties of LDPE-PLA (70:30%) blend film with microclay

SL NO	Properties	unit	Test Results		
			+Microclay	LDPE-PLA	
			1%	3%	5%
1.	Tensile St, M/D	Kg/cm ²	109.02	178.25	206.56
	Tensile St, T/D	Kg/cm ²	12.30	18.20	71.36
2.	Eb. M/D	%	14.06	13.75	9.38
	Eb. T/D	%	5.06	4.20	3.54
3.	Tear St, M/D	gf/m	4.17	4.20	4.37
	Tear St, T/D	gf/m	4.21	5.13	6.40
4.	Dart Impact St	g	50	48	45
5.	Static Coefficient of Friction,	-	0.126	0.133	0.145
	Dynamic Coefficient of Friction	-	0.124	0.132	0.143

From the Table 5, Tensile strength of the samples is better in machine direction(M/D) than their corresponding transverse direction (T/D). Tensile strength increases with corresponding percentage of microclay due to the reinforcing action of microclay filler. From the values we can say that the percentage of elongation is less when microclay is added. The Elongation at break is higher in machine direction than in transverse direction.

Tear strength is the ratio of force required to tear the sample to the thickness of film. From the Table 5, Tear strength values are high in transverse direction than machine direction due to perpendicular orientation of the polymer chains to the applied force, The increase in percentage of microclay increases the hardness, rigidity, toughness and hence increases Tear strength. The Dart impact strength got reduced than pure LDPE (80 g). This is due to the brittleness nature s of amorphous PLA in the LDPE matrix. The brittleness makes the film more easy for crack propagation. As microclay content increases, the falling weight impact resistance decreases slightly.

A material's coefficient of friction is the ratio of the force acting between the material surfaces to the pulling force. Friction has two components static and dynamic. From the values it is clear that the friction coefficient value increases when microclay content increases. The dynamic coefficient of friction is little lower than the static coefficient of friction in all the compositions as expected.

OPTICAL PROPERTIES

Luminous Transmittance And Haze (ASTM D 1003)

Luminous transmittance and Haze determines the transparency characteristics of the film. The high the transmittance value high the transparency The values are determined as per ASTM standards and tabulated below in Table 6.

From the values we can say that as luminous transmittance values decreases, haze increases, due to the light scattering by the filler particles of microclay. Hence the above trend was obtained.

Table 6. Optical properties of LDPE-PLA (70:30%) Blend with microclay.

S.N	Sample LDPE+PLA+MC	Luminous Transmittance (%)	Haze (%)
1.	MC 1%	83	48.5
2.	MC 3%	82	48.6
3.	MC 5%	80	70.2

BARRIER PROPERTIES

Water Vapour Transmission Rate-WVTR (BS 2782)

WVTR determines the water vapour permeability into the film. The test results /values are tabulated below in Table 7.

The values clearly shows that the addition of microclay decreases the WVTR. In other words, microclay shows improvement in barrier to water vapour i.e., it has high barrier property. This is because the microclay filler closes the voids in film matrix like in nanoclay filled films (18) but little higher by about 4–5 times than that of LDPE-PLA (60–40%) with Nanoclay 1% and 2% filled films which are about 0.0733 & 0.0600 grams/sq m/day respectively. But, microclay cost is ten times lower than that of nanoclay.

Table 7. WVTR of LDPE-PLA (70:30%) Blend with microclay.

S.N	Sample LDPE+PLA+MC	WVTR (grams/sq m/day)
1.	MC 1%	0.20
2.	MC 3%	0.19
3.	MC 5%	0.17

CHARACTERIZATION

Fourier Transform Infrared Spectroscopy

FTIR is a powerful tool for identifying the types of chemical bonds in organic and inorganic molecules by producing an infrared absorption spectrum that is like a molecular fingerprint. It can be utilized to identify the components of an unknown mixture. From the graph, the frequency value 3405.18 cm^{-1} indicates the presence of hydroxyl ($-\text{OH}$) group in PLA. The frequency value 2915.35 cm^{-1} indicates the presence of methyl ($-\text{CH}_3$) group in LDPE and the frequency value 1754.57 cm^{-1} indicates the presence of ester ($-\text{COO}$) group in PLA. Figure 1 shows a typical FT-IR spectrum of LDPE-PLA (70:30%) with microclay 5%.

Differential Scanning Calorimetry-DSC (ASTM D 3418)

Differential scanning calorimetry (DSC) is a thermoanalytical technique in which the difference in

the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature and the result is shown below in thermogram (Figure 2).

The thermogram clearly explains the compatibility and melting behavior between LDPE and PLA. Initially at 62°C PLA undergoes glass transition i.e., it changes from brittle to ductile. Then at 113°C LDPE undergoes melting and at 150–160°C the PLA polymer molecules melts. The T_g of LDPE is very lower at about 100 Deg C.

The EGMA works as good reactive compatibilizer and reduces interfacial tension between the two Polymers as shown by SEM where uniform matrix are obtained.

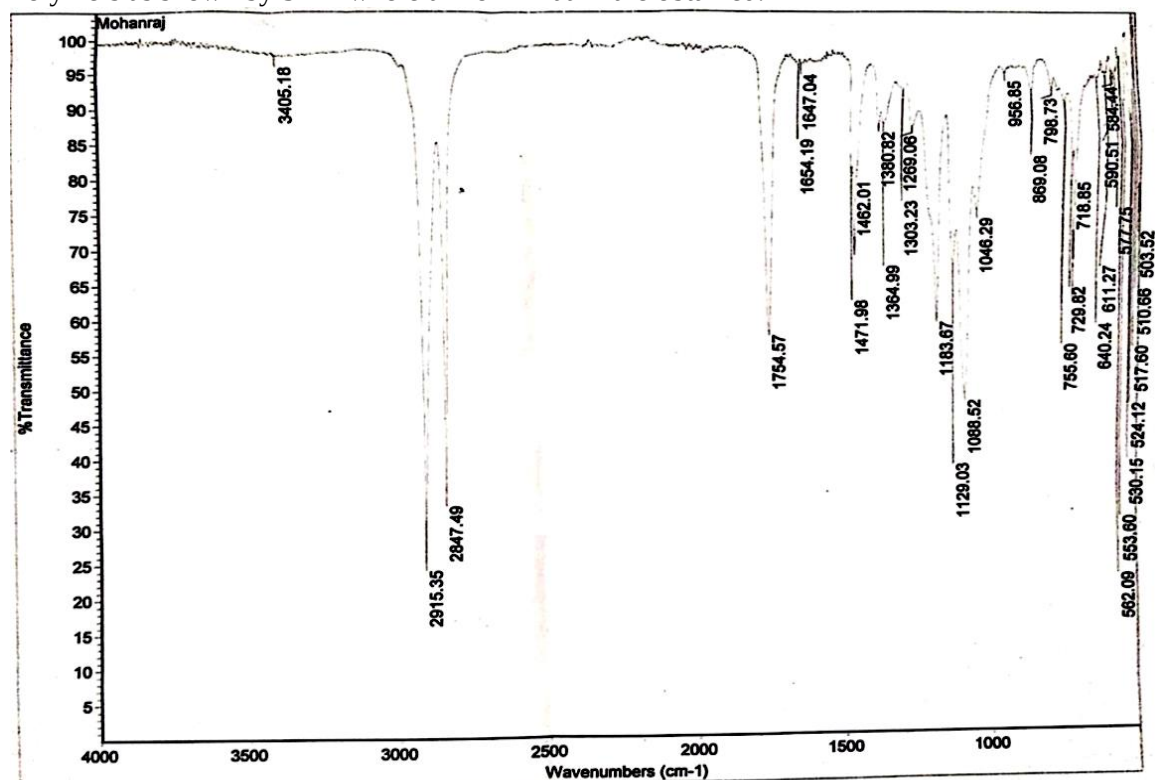


Figure 1. FTIR of LDPE-PLA (70-30%) with microclay(5%).

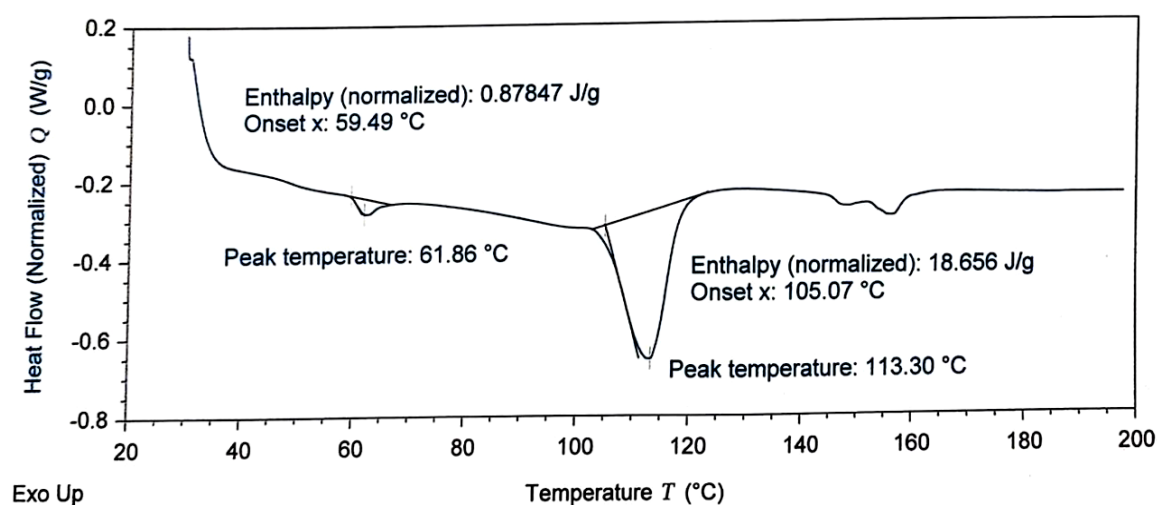


Figure 2. DSC of LDPE-PLA (70:30%) with microclay (5%).

Scanning Electron Microscope-SEM

A scanning electron microscope is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. SEM provides a very detailed 3-dimensional image at much higher magnifications than light microscope are shown below in Figures 3, 4 and 5.

The SEM micrographs clearly show the structure of LDPE-PLA film with microclay. The microclay is distributed and dispersed uniformly in LDPE-PLA matrix. The EGMA works as good reactive compatibilizer and reduces interfacial tension between the two Polymers and hence uniform phase behaviour is obtained.

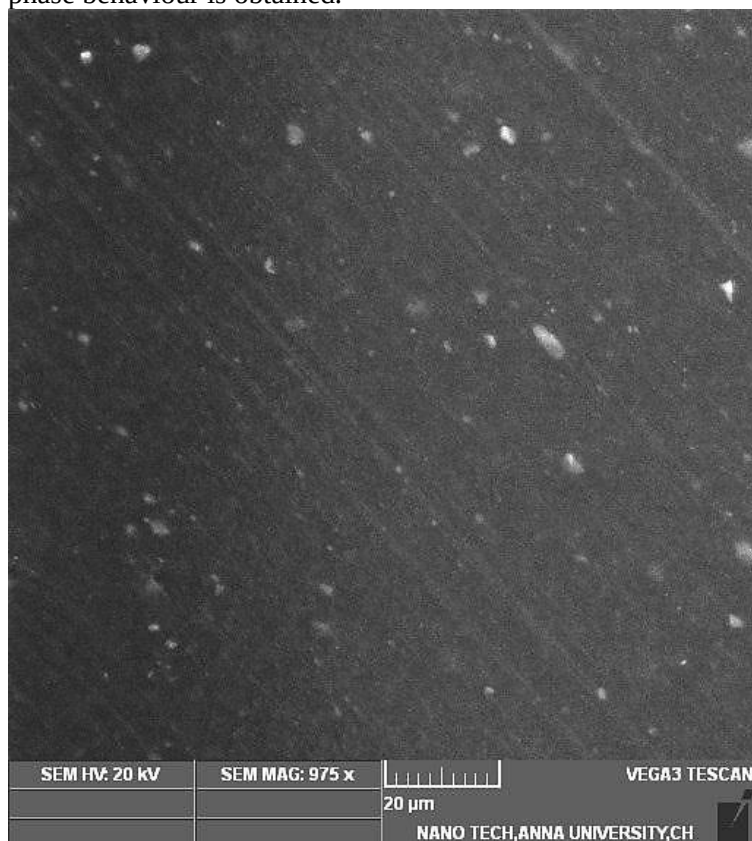


Figure 3. SEM of LDPE-PLA (70–30%) with microclay-1%.

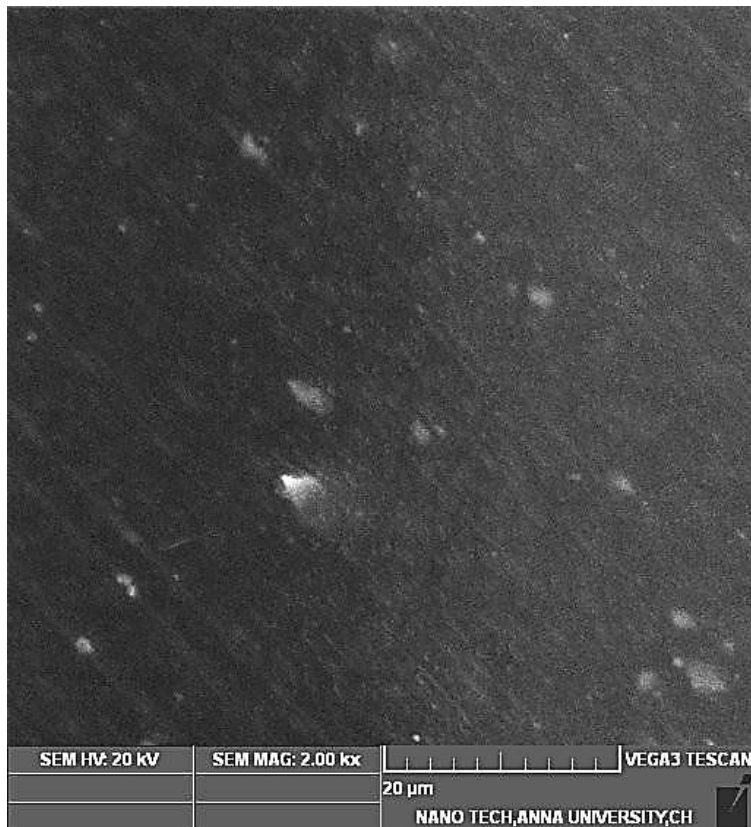


Figure 4. SEM of LDPE-PLA (70-30%) with microclay-3%.

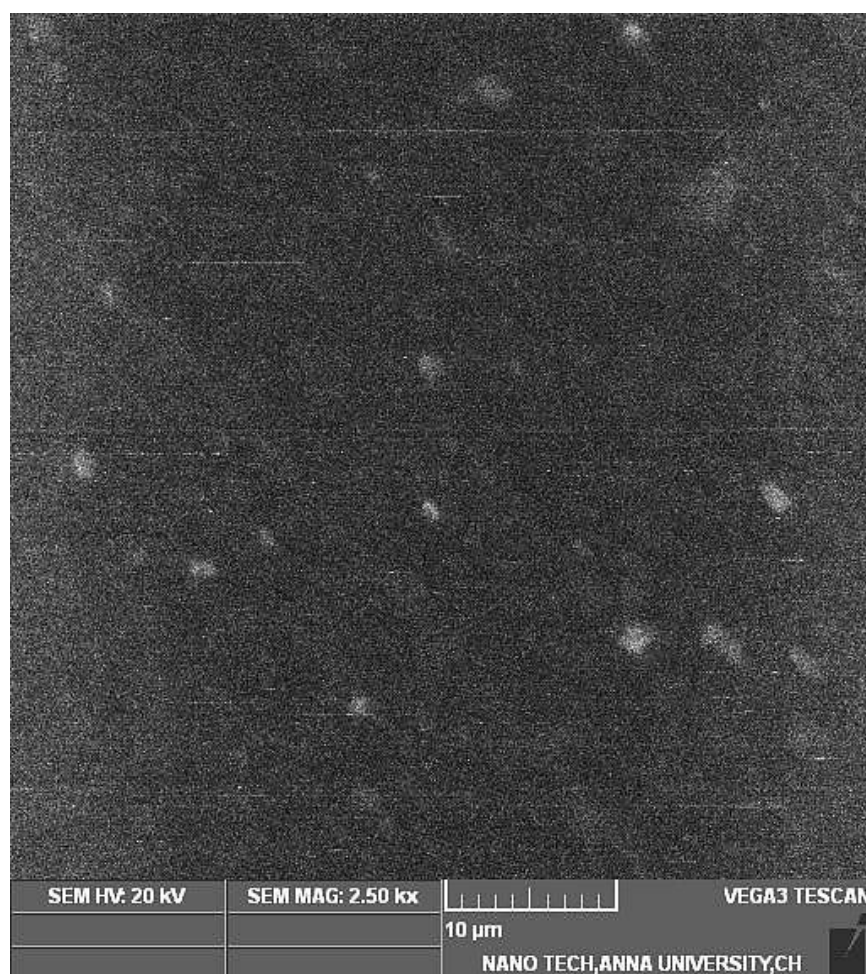


Figure 5. SEM of LDPE-PLA(70-30%) with microclay-5%.

BIODEGRADABLE TEST (SOIL BURIAL TEST)

Soil burial test is carried out biodegradability has to be determined After 28 days, the loss in weight of the film is measured. This determines the ability of the material to biodegrade. After 28 days the film is taken weighed and the percentage of degradation is found to be 14–16 %. The result is very good than LDPE-film but to get 100% degradation more time is needed. In presence of benzophenone, poly ethylenic part of LDPE-PLA will be Photodegradable when exposed to UV-Light of solar radiation and then biodegradable once molecular weights are lowered sufficiently like 2000-3000 for microorganism attack. When combining these two polymers LDPE and PLA by blending we can make a Photo/Bio-degradable blend since PLA is Bio degradable. The LDPE-PLA blends can also be recyclable. If not recycled the blend packaging materials will be Photo/bio-degradable in the environment. Also Thermal energy can be recovered by pyrolysis of the LDPE-PLA-Microclay Packagings.

CONCLUSION

Low Density Poly Ethylene (LDPE) and Poly Lactic Acid (PLA) in the ratio of 70:30 wt% with Ethylene Glycidyl Meth Acrylate (EGMA) compatibilizer and microclay (1,3,5%) as filler are blended/extruded using Twin Screw Extruder and then pelletized. The Pellets are processed into film using Blown Film Extrusion. Of the three samples it is found that the sample with 5% microclay has better mechanical properties than samples with 1% and 3% microclay, even though it has poor optical properties, as luminous transmittance values decreases, haze increases, due to scattering by the filler particles of microclay. The addition of microclay decreases the WVTR. In other words, microclay shows very good barrier to water vapour with lower cost. From SEM it is known that microclay is

distributed/dispersed well. The EGMA works as good reactive compatibilizer and hence uniform flow, uniform phase behavior and uniform properties in LDPE-PLA blends was obtained. Hence this LDPE-PLA blend is miscible one. PLA-LDPE blend with microclay will be photo /bio degradable. LDPE part in presence of benzophenone photo-sensitizer additive is photodegradable in presence of sunlight. PLA part is inherently biodegradable due to polyester groups in the main chains in presence of water.

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