

Research Article

# Studies on Biodegradability, Morphology, Physicomechanical and Thermal Properties of Pp/Potato Starch Bio-Composite

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## Abstract

*In this study to determine the possibility of using starch as biodegradable filler in the thermoplastic polymer matrix, starch-filled polypropylene (PP) composites are prepared by extrusion of PP resin with 5, 10, 15, and 20 wt % of potato starch in corotating twin-screw extruder. The injection molded test specimens are tested for physicomechanical properties such as tensile and flexural properties, Izod impact strength, density, moisture and water absorption. Characterization of the thermal properties by melt flow index (MFI), vicat softening point (VSP), differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) techniques has also been done. The results show that, with increase in starch content, tensile modulus, flexural strength, flexural modulus, moisture absorption, water absorption and density of the PP composites increased but, tensile strength and elongation, impact strength, hardness and MFI of the PP composites decreased while retaining the VSP. For the biodegradability test. Poly propylene (PP)/potato starch (PS) blend films are prepared using the two roll mill machine. The films are investigated by soil burial method over a period of 4 months. The percentage weight loss of the bio-composites in the compost soil burial test is increasing with increasing PS content. The biodegradability of the bio-composites is enhanced with increasing starch content because the PS is easily attacked by microorganisms. Any changes in the various properties of the PP/PS before and after degradation are monitored using FTIR spectroscopy, a scanning electron microscope (SEM) for surface morphology, a thermo gravimetric analyzer (TGA) for rapid determination of starch content and a differential scanning calorimeter (DSC) for crystallinity. It can be concluded from the results that use of these bio-composites will reduce the environmental problems associated with waste pollution and the study findings support the predicted application of bio-composites as green-composites or eco-materials.*

**Keywords:** Polypropylene Potato Starch Bio-composite, Mechanical properties, Thermal properties, Morphology, Soil burial test

## 1. Introduction

In recent times, biodegradable materials have gained importance particularly for the protection of the environment from ever increasing plastic waste (Franco, *et. al.* 2004), (Okada, 2002). A number of biological materials may be incorporated into biodegradable polymer (Avella, *et. al.* 2000) (McCrery, *et. al.* 1978). The main characteristic of biodegradable polymers is that they are biodegradable through the action of the microorganism in appropriate environmental conditions (Contat, *et. al.* 2001). When in contact with the biodegradable polymer, the microorganisms produce enzymes that break the material in progressively smaller segments reduces its average molecular mass, favoring its degradation in the environment (Shogren, *et. al.* 2003). Biodegradable

materials can be completely degraded into natural ecosystems such as active sludge, natural soil, lake and marine.

Petroleum-based synthetic polymers are widely used in modern society. Many of the physical and chemical properties of plastics make them ideal materials for a variety of products and applications. Various approaches to render synthetic polymers degradable have been considered. However, the annual worldwide disposal of approximately 150 million tons of petrochemical plastics in commonly used commodities such as polyolefin in packing, bottle and moulding products is a significant environmental problem, especially with the continuously increasing production and consumption of these materials (Okada, 2002), (Wu, 2003). Most widely used alkane-derived plastics have poor biodegradability and may have lifetime of hundreds of years when buried in typical solid-waste sites.

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The most attractive renewable natural polymer resource is starch because of its low cost, wide spread availability and potential for mass production from renewable resources (Guohua, *et. al.* 2006), (Yun, *et. al.* 2008). Research on biodegradable plastics based on starch began in the 1970s and continues even today at various laboratories all over the world. Starch has been considered a suitable material source due to its inherent biodegradability, availability and relative low cost (Tang, *et. al.* 2006). Starch satisfies the requirements of having adequate thermal stability with minimum interference in melt properties and negligible disturbance of product quality. Starch is the mixture of amylase and amylopectin. The availability of starch is just after cellulose. Potato is the world's fourth largest food crop, following rice, wheat and maize. China is now the world's largest potato producing country and nearly a third of the world's potato is harvested from China and India. Potato starch obtained from the tuber of *Solanum tuberosum* L. and it is a very fine white powder. It is insoluble in cold water and alcohol. The most important industrial sources of starch are wheat, corn, tapioca, potato and rice. Polymeric composite containing starch has been developed for different applications and is the subject of several patents (Bastioli, *et. al.* 1990), (Lay, *et. al.* 1992), (Vaidya, *et. al.* 1977). Incorporation of starch into a polyolefin matrix was proposed by Griffin as an effective means of accelerating the deterioration of plastics under biotic environmental exposure conditions (Griffin, 1975), (Griffin, 1987). The microbial consumption of starch component, in fact, leads to increased porosity, void formation and loss of integrity of the plastic matrix. PP is a popular thermoplastic material widely used in automobile, electrical equipment, furniture and packaging applications because of its excellent and versatile properties but PP is very resistant to hydrolysis and is totally nonbiodegradable. There is great potential interest in incorporating biodegradable material such as starch into conventional plastics such as polypropylene.

The present study reports the preparation of PP/starch composites to examine the possibility of using potato starch as filler in PP matrix and biodegradability study by soil burial method. The changes in the properties of the PP/PS before and after degradation were monitored using weight loss, FTIR spectroscopy, a scanning electron microscope (SEM) for surface morphology, a thermo gravimetric analyser (TGA) for rapid determination of starch content and a differential scanning calorimeter (DSC) for crystallinity. The driving force behind the use of the starch is its low cost, annually renewable resource utilization and environmental benefits. From these results, we suggest the predicted applications of bio-composites that are considered as an alternative to non- biodegradable plastic products.

## 2. Experimental

### Materials

PP injection-grade H200MK obtained from Reliance Industries, Mumbai, India, was used as a polymer matrix. Its melt flow index (MFI) and density as reported by the manufacturer were 20 g/10 min and 0.919 g/cm<sup>3</sup>, respectively. Potato starch granules (insoluble) of 60 mesh size with sulphated ash 0.30% obtained from M/S S D Fine-Chem, Mumbai, India, were used as filler.

### Compounding and sample preparation

Starch powder is mixed with PP granules and extruded in a twin-screw extruder (JSW 30a, Japan) with L/D ratio of 36/1 in the temperature range of 170–230° C. The extruded strands are pelletized and stored in sealed packs containing desiccant. Four levels of filler loading (5, 10, 15, and 20 wt %) are designed in sample preparation. Tensile, flexural, Izod, vicat softening point (VSP), and water-absorption specimens are prepared using ENGEL 80T automatic injection-molding machine with 50-ton clamping pressure in the temperature range of 180–215° C and an injection pressure of 90 bars. After molding, the test specimens are conditioned at 23° C ± 2° C and 50% RH for 24 h according to ASTM D 618 before testing. Similarly four levels of filler loading (5, 10, 15 and 20 wt %) are designed in film preparation. The film of 0.10 mm thickness is prepared in the two roll mill machine with L/D ratio of 1:40 in the temperature range of 170-175° C.

### Testing methods for physicomechanical and thermal characterization

Tensile and flexural strength tests are carried out as per ASTM D 638 and ASTM D 790 respectively on universal testing machine UTM (Lloyds, LR 100 K). Izod impact strength test is carried out as per ASTM D 256 A on RESIL IMPACTOR (CEAST, Italy). VSP test is carried as per ASTM D 1525 test method on HDT-VICAT Junior (CEAST, Italy). MFI analysis of various formulations is measured using extrudate pellets on melt flow indexer (Lloyds MFI tester, Type 7273) at 190° C and at 2.16 kg load as per ASTM D 1238. Rockwell hardness is measured using EIE HARDNESS TESTER, India. Density is measured using a Mettler PM200 electronic weighing balance as per ATM D 792 displacement method.

### Biodegradability test

Biodegradability test (Fig. 1) of all the blends is performed by the soil burial method (Potts, *et. al.* 1972). For this test, a number of plastic containers of about 250 ml capacity are filled with compost. Rectangular polymer samples (in triplicate) are cut into 30 x 50 mm pieces and buried in the soil at a depth

of about 5 cm. The compost is kept moist by sprinkling water at regular time intervals to maintain 45-50% humidity. The excess water is drained through the hole at the bottom of the container. The containers are stored at about 30-35° C. The degradation of the samples is studied at regular time intervals by removing the samples carefully from the soil and washing them gently with distilled water to remove the soil adhering on the surface. The samples are dried at 60° C under vacuum until a constant weight is obtained. Weight loss of the polymer with respect to time is recorded as a measure of degradation. The soil burial test is studied by evaluating the weight loss of the film over time. The weight loss is calculated using Equation (1)

$$\text{Weight Loss (\%)} = (M_o - M_d)/M_o \times 100 \quad (1)$$

$M_o$  = Initial mass;  $M_d$  = Degradation mass at each designated day



**Fig. 1** Soil burial test

#### Scanning Electron Microscopy

The surface morphology of the PP/PS films before and after biodegradation is observed by means of Nova Nano FEG-SEM 450. The micrographs of the samples are taken at different magnifications to identify cracks, holes and other changes on the surface of the samples during degradation.

#### Transmission electron microscopy

The diffraction analysis of the sample is determined by transmission electron microscopy by JEOL JEM 2100. Resolution of the instrument is 1.4 Angstrom Unit and the accelerating voltage is 200kV. The sample is prepared by ultra-microtome method and the filament used is LaB<sub>6</sub> (Lanthanum hexa boride), made in Japan.

#### Atomic force microscopy

AFM imaging is performed on flat bio-composite films of Polypropylene using NT-MDT NTEGRA Prima Scanning Probe Microscope in tapping mode, operated with 100 X 100μ scanner and NSG tip having size at the

edge around 10 nm and frequency of 1.01 Hz. The coated area of composite film is subjected to 50μ X 50μ scan under SPM. The topography image is developed with Nova software supplied with the instrument. Average roughness analysis and particle size distribution in the scanned area are studied.

#### Thermal Properties

DSC analysis is performed using Mettler Toledo (Star DSC1 SW 9.30). The instrument is calibrated with indium (calibration standard, purity >99.999%) for melting point and heat of fusion. A heating rate of 10 °C/min is employed in the range of 25 °C to 500 °C. Analysis is performed under a nitrogen purge (50 ml/min). An empty pan is used as a reference.

#### Glass transition temperature study

DSC analysis for glass transition temperature is carried out using PYRIS DIAMOND DSC (Perkin Elmer, USA) at a heating rate of 10° C/min over a temperature range of -40-200° C.

#### Infrared Spectroscopy

Fourier transform infrared (FTIR) spectra of PP/PS films before and after biodegradation, are obtained in SHIMADZU CORP (model A210044 01377) in the spectral region between 4500 and 450 cm<sup>-1</sup>.

### 3. Results and discussion

#### Physicomechanical properties

##### Tensile and flexural properties of PP/PS composites

The results [Table 1] shows that the tensile strength and tensile elongation decreased from 42.63 to 29.82 MPa and 19.77–8.42%, respectively. The reason behind the reduction in tensile strength and elongation at break can be attributed to the facts that the weakness of interfacial adhesion due to hydrophilic nature of starch that is not compatible with hydrophobic PP and less-effective cross-sectional area of PP matrix that is continuous phase toward spherical particulate starch granules as the starch contents rose. This is a general phenomenon observed in thermoplastics filled with natural filler (Ismail, *et. al* 2001), (Ismail, *et. al* 2002), (Ismail and Jaffry, 1999). With increment in the filler loading, thereby increasing the interfacial area and worsening interfacial bonding between the filler and the matrix (Thakore, *et. al.* 1999), (Ahamed, *et. al.* 1996).

The tensile modulus is increased from 845.58 to 932.82 MPa. Similarly the flexural strength increased from 43.44 to 47.95 MPa and flexural modulus increased from 1355 to 1521 MPa as the starch content increased from 0 to 20%. Starch granules are not melting and retain their shape as rigid filler during

**Table 1** Effect of Starch Addition on Tensile and Flexural Properties of PP Composites

Blend Composition PP/PS	Tensile strength (MPa)	Tensile modulus (MPa)	Tensile elongation (%)	Flexural strength (MPa)	Flexural modulus (Mpa)
100/00	42.63	845.58	19.77	43.44	1355
100/05	38.31	884.37	12.78	45.70	1382
100/10	36.10	885.93	10.82	45.79	1441
100/15	31.93	924.43	9.59	46.39	1501
100/20	29.82	932.82	8.42	47.95	1521

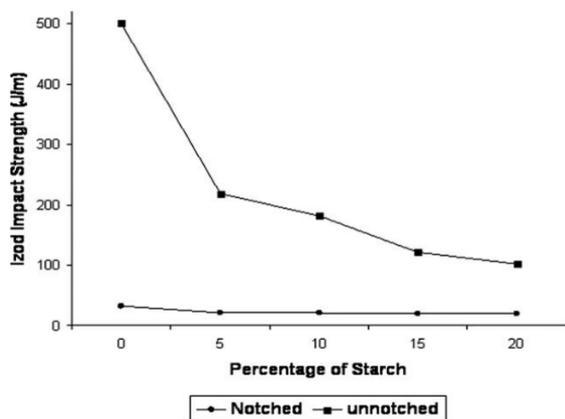
**Table 2** Effect of Starch Addition on Moisture, Water Absorption, Density and Hardness of PP Composites

Blend Composition PP/PS	Moisture absorption (%)	Water absorption (%)	Density (g/cc)	Rockwell hardness
100/00	0.013	0.035	0.9121	102
100/05	0.023	0.076	0.9214	101
100/10	0.032	0.105	0.9360	100
100/15	0.050	0.151	0.9581	99
100/20	0.072	0.242	0.9806	97

processing. Starch granules may be stiffer than the PP matrix in which they are dispersed (Nawang, *et al.* 2001). With the increment in the starch content, particles are crowded and reduced the particle–matrix interaction. The material became more rigid and stiffer than material without starch.

*Impact strength of PP/PS composites*

From Figure 2, unnotched Izod impact energies are considerably larger than notched Izod impact energies. The notched impact strength decreased from 33.07 to 18.78 J/m with increasing the amount of starch filler and unnotched one drastically decreased with filler loading from 501.37 to 101.37 J/m. This large difference notched and unnotched values can be explained by the facts that unnotched Izod impact energies are not only a measure of crack propagation but also crack initiation and notched Impact behaviour is controlled to a greater extent by factors affecting the propagation of the fracture initiated at the predominating stress concentration at the notched tip.



**Fig. 2** Effect of starch addition on Izod impact strength of PP composites

The decrement in the Impact energies with increasing starch content may be due to the low-interfacial interaction between starch and PP which would lead to mechanical rupture at the blend interface. Similarly, poor interfacial bonding induces micro spaces between the filler and matrix polymer which causes numerous micro cracks when the impact occurs, inducing easy crack propagation.

*Moisture, water absorption, density, and hardness of PP/PS composites*

It is seen from the Table 2 that the moisture absorption of PP composite increased from 0.013 to 0.072% and water absorption increased marginally from 0.035 to 0.242% with increase in starch content from 0 to 20%. With increment in the filler amount, potato starch being more hydrophilic than PP, leading to an enhancement of hydrogen bond formation between hydroxyl groups in the starch and moisture or water. Starch is encapsulated by PP matrix and are not exposed to the surface to have higher water absorption. These results are key factors to promote the biodegradation rate.

The density of PP increased from 0.9121 to 0.9806 g/cc with the increase in starch content from 0 to 20 wt %. Density increase may be due to the higher density of the starch filler (1.575 g/cc) (Isleib, 1958). Due to the incompatibility, micro void formation, poor interfacial adhesion or phase separation between PP and starch Rockwell hardness decreases with increase in starch content.

*MFI and Vicat softening point*

Starch particles are more viscous than PP, so the MFI values of PP/starch composites decreased from 19.83 to 13.23 g/10 min as the starch content increased from 0 to 20 wt % [Table 3]. Starch granules retained their

**Table 3** Effect of Starch Addition on Melt Flow Index and Vicat Softening Point of PP Composites

Blend Composition PP/PS	Melt flow index (g/10 min.)	Vicat softening point (°C)
100/00	19.83	147.6
100/05	16.58	147.6
100/10	16.12	147.8
100/15	15.21	147.4
100/20	13.23	147.7

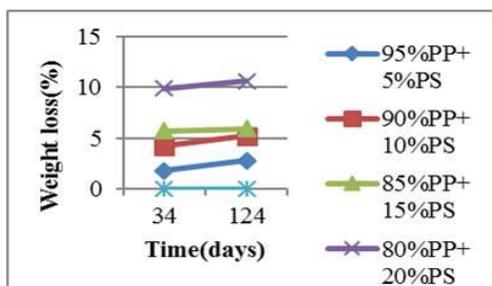
shape and functioned as rigid particulate fillers during processing and restricted the melt flow. The inter particle space becomes small with increment in the starch content and subsequently the particle-particle interactions getting pronounced which results in agglomeration formation, leading to immobilization of the matrix. As the size of agglomerates increases, flows of matrix have been confined due to trapping of the matrix molecules in the filler particles.

VSP of virgin PP does not change with variation in starch content because starch is rigid filler inside the relatively flexible PP matrix. So, there is retention of the elevated temperature performance of the PP bio-composite.

*Soil burial test*

Fig. 3 shows the weight loss of pure PP and PP/PS films in soil. In the figure, biodegradability increased up to 10.6% as the burial time increased in the soil for 120 days for 20% PP/PS bio-composite. All the films in soil degraded rapidly in the first 30 days. This rapid degradation was due to the composting process, which occurred in two stages: an active composting stage and a curing period. In the first stage, the temperature rose and remained elevated as long as there was available oxygen, which resulted in strong microbial activity.

In the second stage, the temperature decreased but the film continued to compost at a slower rate (Albertsson 1986). In the soil burial test pure PP showed almost no loss in weight.

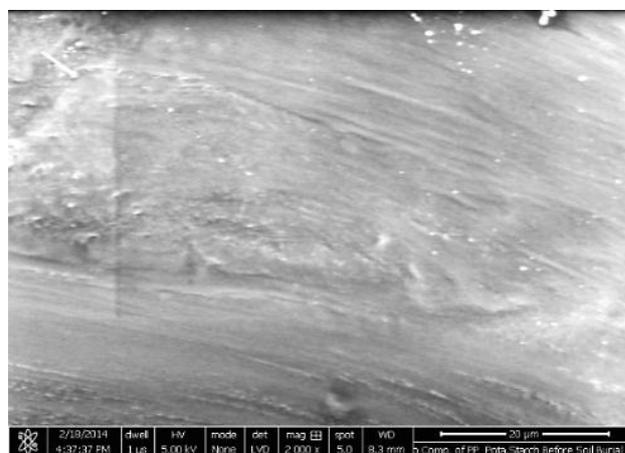


**Fig. 3** Weight loss in soil burial test

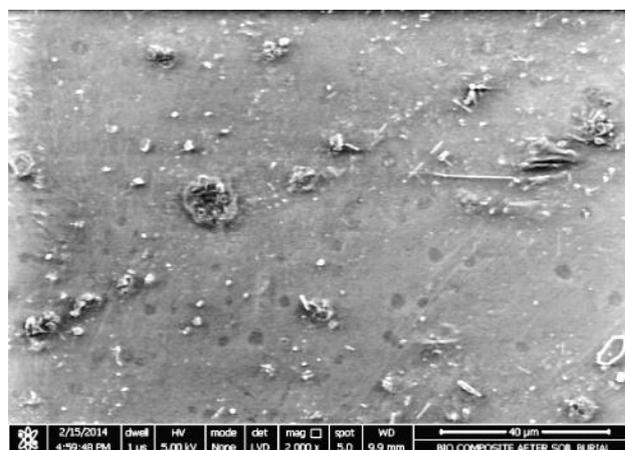
*Surface morphology*

Scanning electron microscopy (SEM) has been used widely to study the surface morphologies. Enhanced resolution of samples by SEM permits higher magnification probing of surfaces without loss in detail.

In addition, due to the depth of the field of SEM, the micrographs retain a three-dimensional appearance of the samples. SEM was used to study the surface morphological changes after the biodegradation of the samples. Fig. 4 was used to exemplify the effect of soil on the PP/PS (80/20) blend. After soil burial the blend exhibited weight loss due to microbial growth on the starch. Micrographs [Fig. 4(a) and (c)] show continuous phase before biodegradation and [Fig. 4(b) and (d)] show cracks, voids and damages on the surface after biodegradation. This is a general phenomenon observed in other-thermoplastics filled with natural filler.

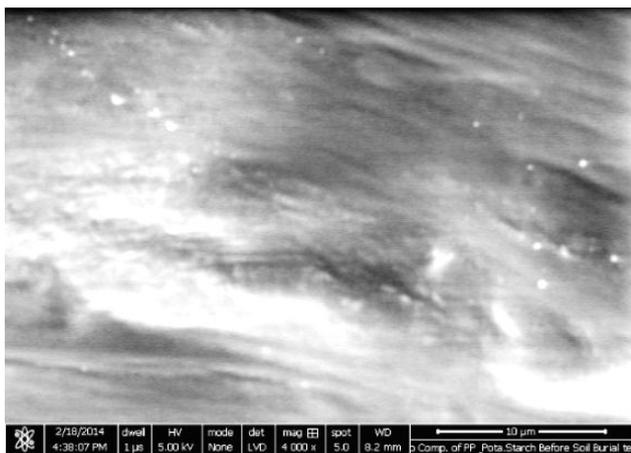


(a)

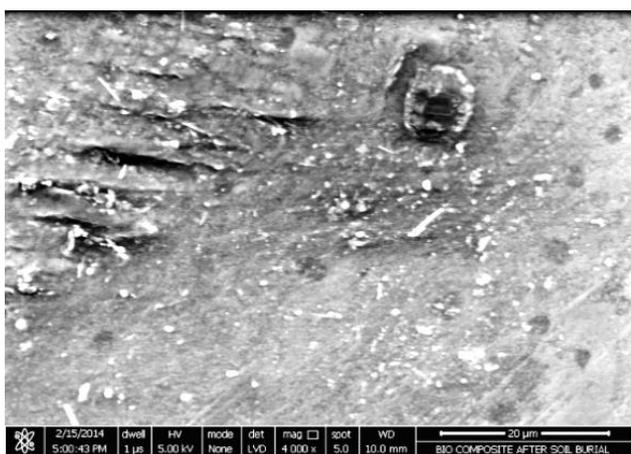


(b)

**Fig. 4 (a) & 4 (b)** SEM micrographs of PP/PS 20% bio-composite (for the magnification of 2000x) before & after biodegradation



(c)

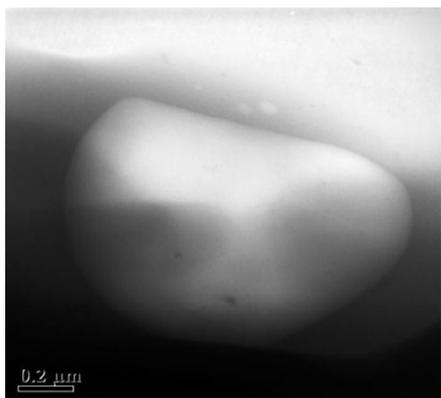


(d)

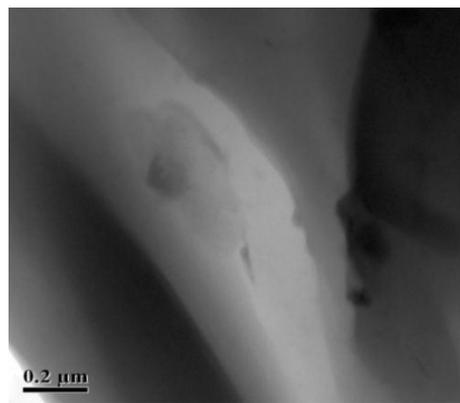
**Fig. 4 (c) & 4 (d)** SEM micrographs of PP/PS 20% bio-composite (for the magnification of 4000x) before & after biodegradation

*TEM Studies*

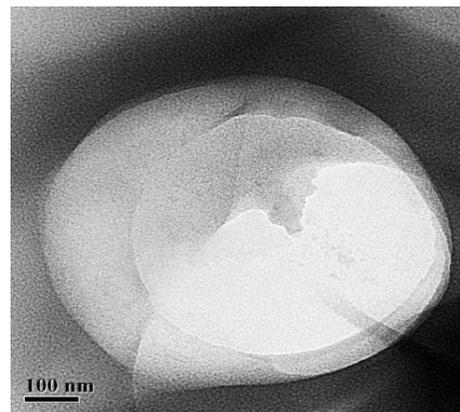
Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through.



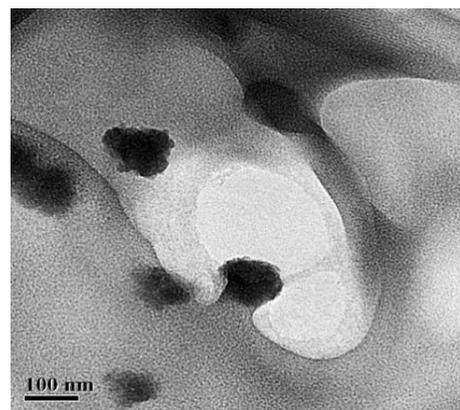
(a)



(b)



(c)



(d)

**Fig. 5 (a), (b)** TEM (0.2 micro metre) of micrographs of PP/PS (80/20) after biodegradation & **Fig.5 (c), (d)** TEM (100 nm) of micrographs of PP/PS (80/20)

An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film. In this experiment it was observed [Fig. 5(a) to 5((d)] that after biodegradation there is loss of starch content from the composite. This observation is also supported by other researchers for bio- composite biodegradation (Thakore, *et. al.* 2001).

Atomic Force Microscopic Studies

Atomic Force Microscopic Studies (AFM) has been used widely to study the surface morphologies. In addition, due to the depth of the field of AFM, the micrographs retain a two- dimensional as well as three- dimensional appearance of the samples. AFM was used to study the surface morphological changes after the biodegradation of the samples.

Fig. 6 to 11 was used to exemplify the effect of soil on the PP and PP/PS (80/20) blend. After soil burial the blend exhibited weight loss due to microbial growth on the starch. In Fig. 6 (a) and 6 (b), there is no change for PP films in before and after biodegradation whereas comparing the Fig. 7 (a) & 7 (b) it can be stated that some sort of degradation has been taken place after soil burial method in PP/PS composite which is clearly reflected by the change in surface roughness. Similarly for three dimensional images the continuity in the phase has been maintained for PP films in before [Fig. 8 (a)] as well as after biodegradation [Fig. 8 (b)] but it is showing an increasing damage on the surface of PP/PS films by comparing before [Fig. 9 (a)] and after [Fig. 9 (b)] degradation images. For PP films after degradation counts are increasing and domain sizes are decreasing [Fig. 10 (a) and 10 (b)] but not much change in the roughness has been seen. For PP/PS films after degradation counts and domain size both are decreasing [Fig. 11 (a) and 11 (b)] and a change in the roughness has been seen.

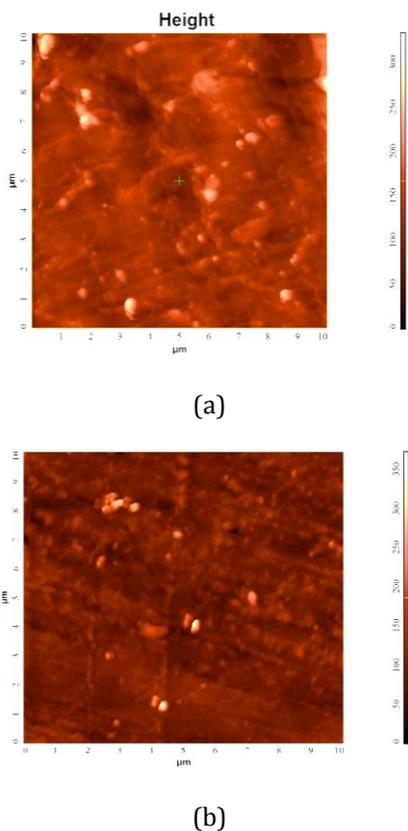


Fig. 6 AFM (2D) of PP (a) before degradation & (b) after degradation

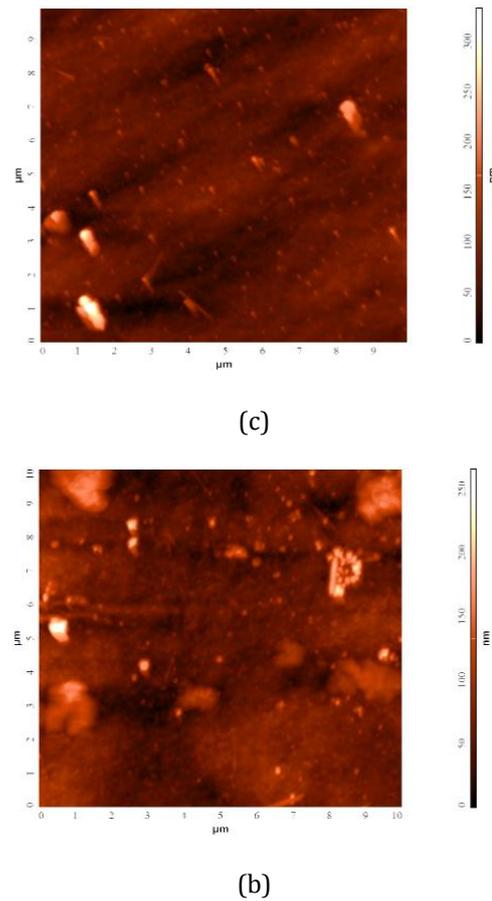


Fig. 7 AFM (2D) of PP/PS (80/20) bio-composite (a) before degradation & (b) after degradation

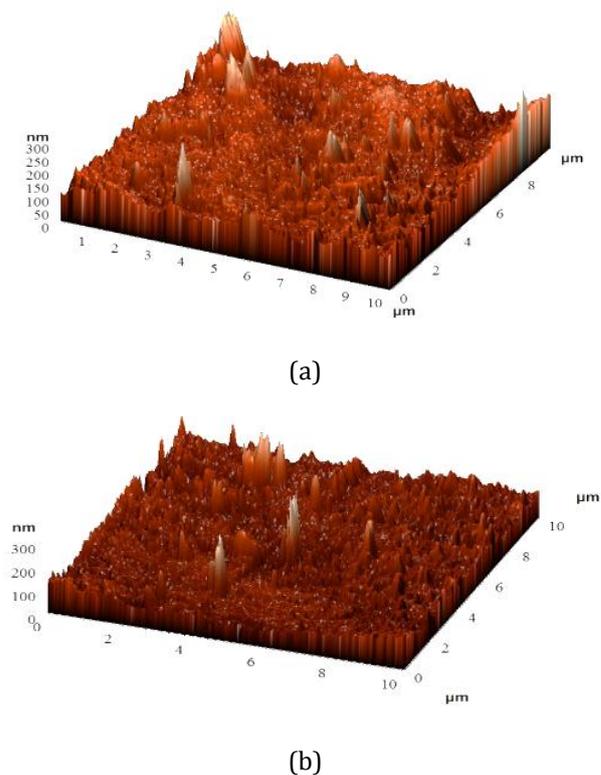
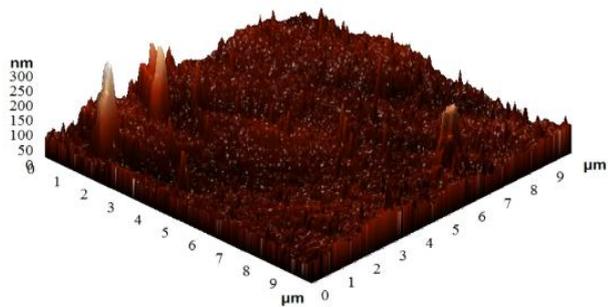
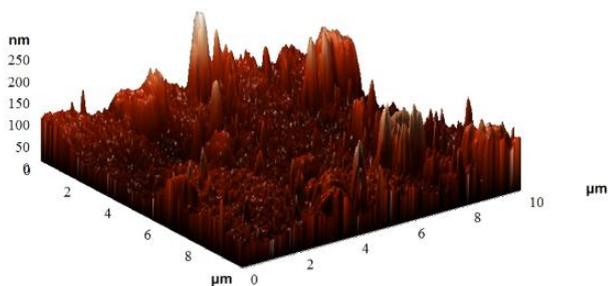


Fig. 8 AFM (3D) of PP (a) before & (b) after degradation

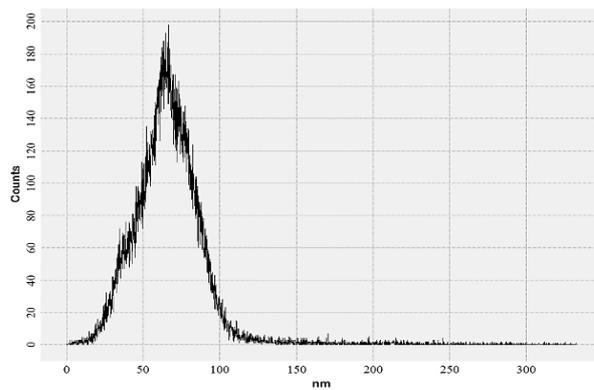


(a)

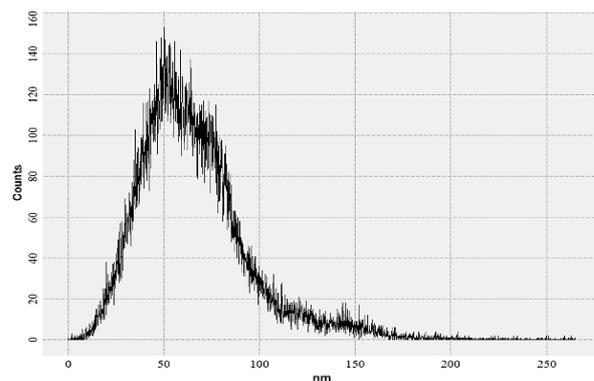


(b)

**Fig. 9** AFM (3D) of PP/PS (80/20) bio-composite (a) before & (b) after degradation

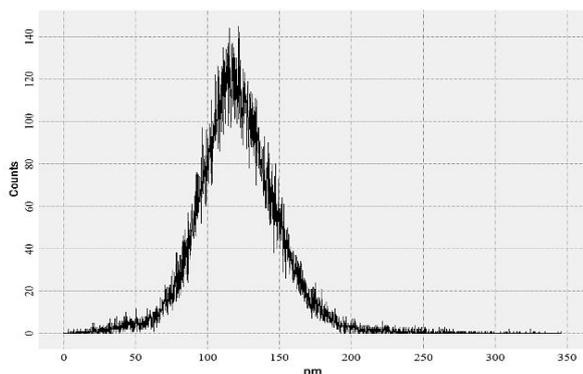


(a)

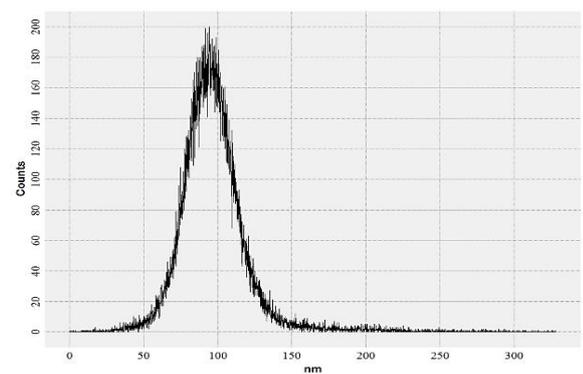


(b)

**Fig. 11** AFM (Histo) of PP/PS (80/20) (a) before & (b) after degradation

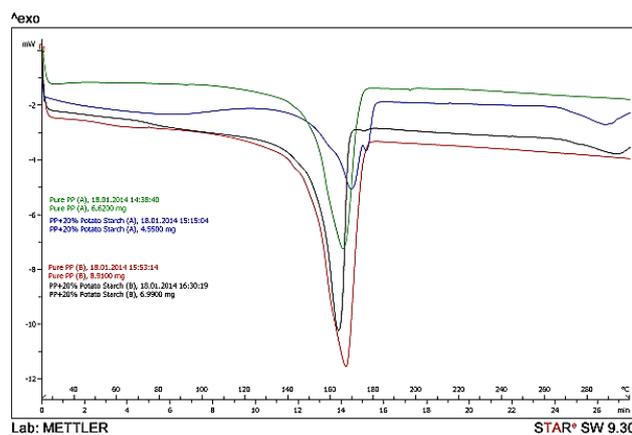


(a)



(b)

**Fig. 10** AFM (Histo) of PP (a) before & (b) after degradation



**Fig. 12** DSC of PP before degradation (A), after degradation (B) & PP/PS (80/20) before degradation (A), after degradation (B)

The percent crystallinity was calculated on the assumption that the heat of fusion of 100% crystalline PP is 207.1 J/g. As the heat of fusion is directly proportional to the amount of crystalline PP in the sample, it decreases as filler percentage increases. An apparent decrease in the heat of fusion [Fig. 12] was due to the decrease in the weight fraction of crystalline PP.

$$X_c (\% \text{ Crystallinity}) = [\Delta H_f / w \Delta H_{f100}] \times 100$$

$\Delta H_f$  = latent heat of fusion of the composite sample;

$\Delta H_{f100}$  = latent heat of fusion of a PP with 100%

crystallinity;  $w$  = weight fraction of PP in the composite;

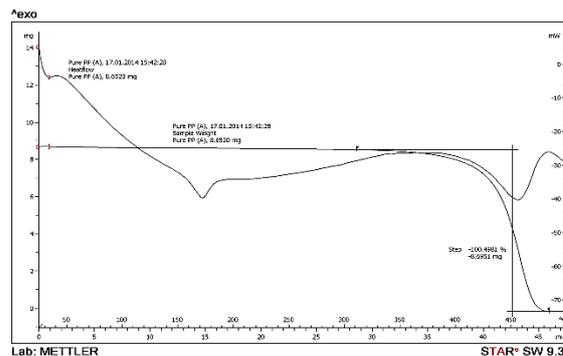
After 4 months of exposure to nursery soil environment, the biodegraded samples were again analyzed for crystallinity. The results obtained for PP and PP/PS blend are tabulated in Table 4. Heat of fusion and crystallinity increased after bio degradation. During biodegradation process, generally the amorphous fraction of material is exposed to microorganism attack. Therefore, the microbial degradation results in an increase in the overall degree of crystallinity of the sample. This is similar to the results obtained for most filled thermo- plastic and in agreement with that reported by researchers before, stating that the crystallinity of starch composite increased after biodegradation (Albertsson, 1986), (Kestelman, *et. al* 1972).

**Table 4** Effect of Soil Burial Test on Crystallinity, Melting Temp. and Heat of Fusion

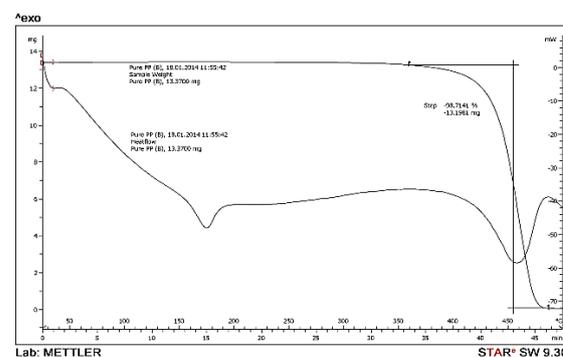
Blend Composition PP/PS	Melting Temperature (Tm) (°C)		Heat of Fusion (J/g)		% Crystallinity	
	A	B	A	B	A	B
100/00	165.49	166.62	79.81	83.15	38.53	40.15
100/20	169.42	163.19	70.90	74.07	42.79	44.71

*Thermogravimetry analysis*

The TGA curves of PP and PP/PS show that a considerable change has occurred in the thermal behavior of PP upon filler addition during biodegradation. In the Fig, 13 (a) and (b), the TGA curve of PP sample (Fig. 13) shows no weight loss in the temperature range of 250-350°C whereas there is a very distinct weight loss is seen in PP/PS sample after bio-degradation in the same temperature interval for the comparison of Fig. 14 (a) with Fig. 14 (b), suggesting weight loss is due to partial or complete pyro lytic volatilization of the starch in the blend [Table 5]. This loss in weight might therefore be used to quantify the starch content in the sample, or residual starch content after degradation. In PP/PS blend with starch content 20%, there is significant decrease in the amount of starch (about 12.16%) was observed after soil burial test from TGA data.

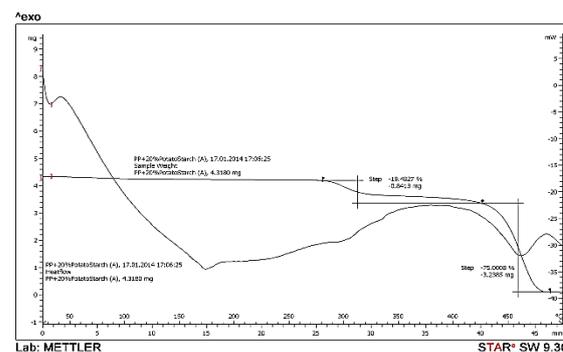


(a)

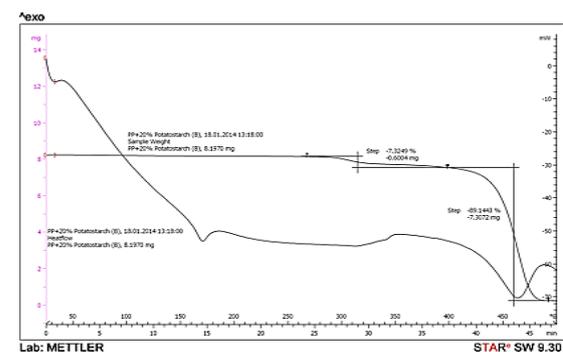


(b)

**Fig. 13** TGA of PP (a) before & (b) after degradation



(a)

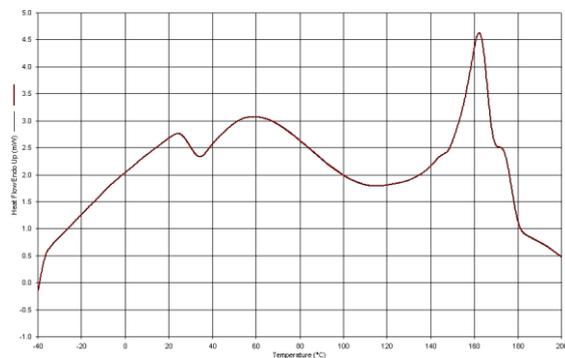


(b)

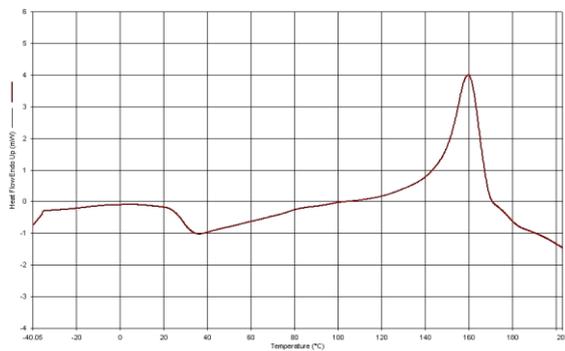
**Fig. 14** TGA of /PS (80/20) bio-composite (a) before & (b) after degradation

**Glass transition temperature ( $T_g$ )**

It is used when describing temperature curves of polymers. At low temperatures, the polymer occurs in a glassy state (it appears as a rigid and brittle solid). With increasing temperature it is in the glass transition range which is particularly important and is mostly specified in terms of just one temperature value which is referred to as glass transition temperature. It is assumed to be approximately in the middle of the temperature range in which the glass transition is taking place. In this range the molecules are showing more and more mobility and the polymer occurs in a soft-elastic, rubber-like state. For PP/PS films there is a change in  $T_g$  ( $-10^\circ\text{C}$ ) in the enthalpy value after degradation [Fig. 15.(a) and (b)] in the descending direction so it can be concluded that after degradation in the film as the starch is coming out so it would be easier for the flow of the material.



(a)



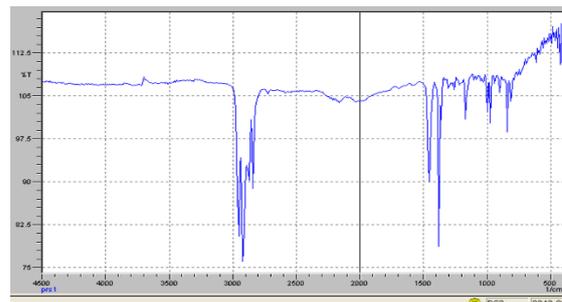
(b)

**Fig. 15**  $T_g$  of PP/PS (80:20) bio-composite (a) before & (b) after soil burial

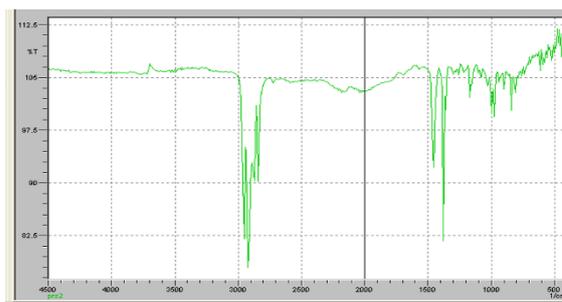
**Fourier Transform Infrared Spectroscopic Studies**

The FTIR spectra of PP film show characteristic C-H stretching bands within the  $3000-2840\text{ cm}^{-1}$  region, medium-strong C-H bending bands within the  $1465-1450\text{ cm}^{-1}$  region and C-H out of plane bending within the  $740-719\text{ cm}^{-1}$  region [Fig. 16(a) and (b)]. All the bands are also present in PP/PS (80/20) [Fig. 17 (a) to (b)]. The characteristic bands for starch in PP/PS blend

films showed a broad O-H stretching absorbance in the  $3600-3000\text{ cm}^{-1}$  region and strong set of C=O stretching band in the  $1190-960\text{ cm}^{-1}$  region.

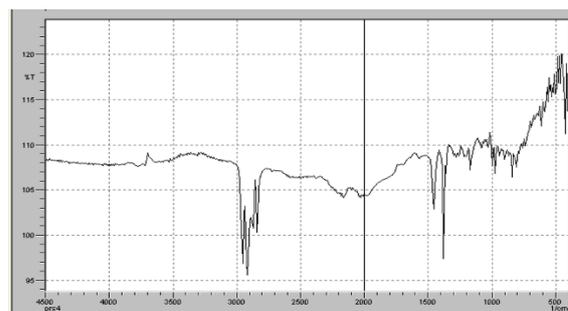


(a)

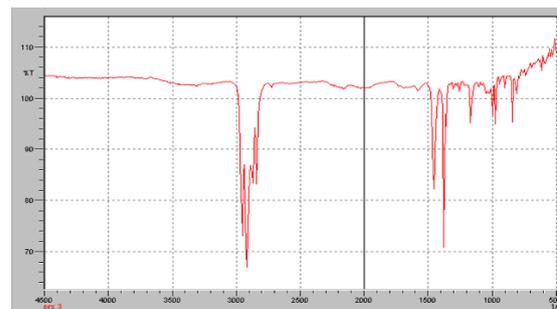


(b)

**Fig. 16** IR of PP (a) before & (b) after degradation



(a)



**Fig. 17** IR of PP/PS (80/20) bio-composite (a) before & (b) after degradation

The other bands independent of PP are the O-H bands though this band is unreliable for qualitative analysis due to their dependence on water content (Smith,

1979). In the blends the 1640  $\text{cm}^{-1}$  band, which is a measure of the water content, vary between degraded and undegraded samples. This could be attributed to a change in the absorbed water starch band as starch is removed rather than any change in the polymer matrix. A decrease in the O-H stretching band in the 3600-3000  $\text{cm}^{-1}$  region was also observed. The other significant trend that was observed in PP/PS blend before and after soil burial test was a slight change in the C-O stretching band (960-1190  $\text{cm}^{-1}$ ) after degradation, indicating that the starch is preferentially removed, leaving the PP matrix [Fig. 17.(b)].

## Conclusions

The tensile modulus of PP/starch composites increased from 845.58 to 932.82 MPa, flexural strength increased from 43.44 to 47.95 MPa and flexural modulus increased from 1355 to 1521 MPa along with the increase in moisture and water absorption and density. But, tensile strength and elongation, impact strength, MFI and hardness decreased. The decrease in tensile strength and elongation with addition to starch filler to PP matrix follows the general trend of filler effects on polymer matrix. The blends containing a higher percentage of starch degraded rapidly in the first 30 days, during which the maximum starch content was accessed (10.6%). Over the next 4 months a gradual decrease of starch occurred. In morphological study has also showed cracks, voids and damages on the surface of the bio-composite after biodegradation. In DSC study, PP/PS blend with starch content 20%, there is significant decrease in the amount of starch (about 12.16%) was observed after soil burial test. Heat of fusion and crystallinity increased after bio degradation. During biodegradation process, generally the amorphous fraction of material is exposed to microorganism attack. Therefore, the microbial degradation results in an increase in the overall degree of crystallinity of the sample. In the AFM, TEM, SEM studies it was observed that after degradation there is changes in the morphology as well as surface roughness and it is also clear from IR that starch is coming out of the bio-composite. So, the utilization of starch in the PP matrix may reduce the cost as well as bring environmental benefits.

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