

Implicating Nanoparticles as Potential Biodegradation Enhancers: A Review

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Abstract

Nanoparticles have entered the scientific world with varied potential applications, of which studies are often conducted in relation to pharmaceuticals as potent drug delivery systems. They perk up pharmacokinetic and pharmacodynamic properties. But, in the era of innovation the studies have revealed a novel side of nanoparticles that has shown their unique ability in enhancement of polymer degradation. Petroleum-plastics like Low Density Polyethylene (LDPE) being an unavoidable necessity, has crossed high levels of environmental pollution due to its poor waste disposal and thus, calls for better ways of its degradation management. The conventional methods have shown a limited degradation with environmental constraints which have been overcome by microbial and enzymatic process of biodegradation. Nanoparticles influence the growth profiles of LDPE degrading microorganisms to augment the biodegradation rate, a major conundrum faced. The review primarily focuses on researches related to the degradation aspect where nanoparticles have acted as enhancers of biodegradation.

Keywords: Plastics; Biodegradation; Nanoparticles

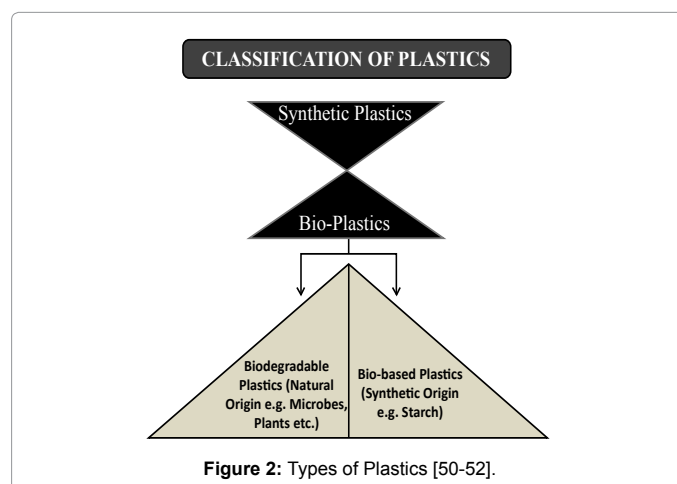
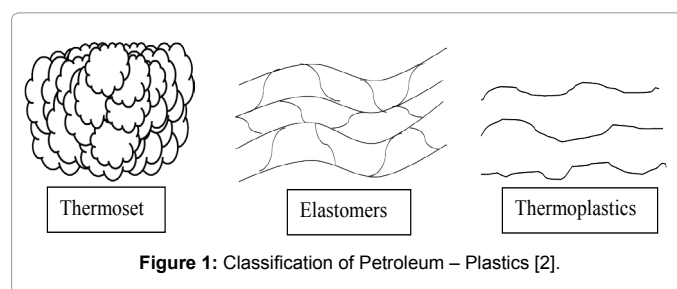
Introduction

Petroleum plastics are the most unavoidable face of our daily necessities. These are the organic polymers with high molecular mass and long linked chains of hydrocarbons with a few nitrogen, oxygen or sulfur. Their properties depend on the chemical structures, where side-chains play an important role [1] and thus, exist of various types (Figure 1). Additives are further added to get superior properties and to make them appealing (like fillers, colorants etc. [2]) but at the same time become non-degradable and hazardous to health in some cases [3].

Polyethylene wastes are normally discarded as landfill or thrown in water bodies as garbage material to decompose/degrade [4]. These synthetic plastics get aggregated at the rate 25 million per year [5] and cause an increased encumbrance on the ecosystem [6], which is presently being dealt with recycling, land filling and incineration. But these approaches have different constraints with regard to environment like recycling is a very environmentally-attractive solution, but a very small part of the plastics can be recycled where the remaining goes to the burial sites [7]. Landfills are less in number and are rarely satisfactory, whereas, incineration causes highly toxic fume generation that is released into the environment causing air pollution.

Thus, scientists look forward for an 'environment friendly' way for either replacing the synthetic plastics with bio-plastics (Figure 2) or biodegrading the petroleum plastics due to the inability of excluding them from daily life. Microbial [8] and Enzymatic [9] processes have been studied well so far in soil and water used for degradation [10-12], where usually this degradation occurs photolytically and chemically [13] (Figure 3). The changes in the molecular mass, melting temperature etc. are evident for the results of degradation [14]. A new advancement in the study is to supplement microbial degradation with nanoparticles. Nanoparticles are found to be influential on the bacterial growth profile, thus has been incorporated for degradation enhancement.

The particles of size ranging between 1-100 nm are called nanoparticles, and they are capable enough to act as a complete unit, which were given by National Nanotechnology Initiative of USA, as ultrafine particles, later gave that agglomerates of ultrafine particles is



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known as Nanopowder and their single domains are Nanocrystals [15].

This review focuses on selected nanoparticles used for biodegradation of synthetic plastics.

There are varied nanoparticles that enhance thermal, mechanical and physicochemical stability along with biodegradability [4,7,16]. Nanoparticles are used as nanoclays by forming the nanocomposites that have ultra large surface area to volume ratio, also known as clay nanocomposites. Using nanoparticles as fillers help in enhancing the material properties along with degradation. They help in achieving the activation energy faster for a process than the neat polymer [4] (Figure 4).

Bio-degradation by Microorganisms with Nanoparticles as Enhancers

Nanobarium titanate [17]

NBT is supplemented in minimal broth, to influence the growth cycle of LDPE degrading bacterial consortia. It influences lag phase, exponential phase and stationary phase and acts by reducing the

duration of lag phase and increasing the duration of exponential and stationary phase. For the accelerated growth of bacterial consortia NBT is a supportive nutritional component and thus assist the consortia in plastic waste biodegradation.

The preferred particle size is 38nm with bacterial consortia of *Microbacterium* species strain MK3, *Pseudomonas putida* strain MK4 and *Bacterium* Te68R strain PN12 [11] (Table 1). Experiment performed with both positive and negative control with and without nanoparticles, where, nanoparticles are sonicated at 50-60 Hz for 2.5 minutes with 0.3 second cycles.

UV spectrophotometer is used for monitoring bacterial growth cycle. Consortia is removed from the LDPE by centrifugation and then evaporated to remove water. The residue is analyzed and characterized using FTIR (Table 2) and TG-DTG-DTA (Tables 3 and 4), where LDPE is taken as control. As the polymeric structure changes, a shift in λ_{max} is observed from 209-225.3 nm. This shift takes 4 days in the absence of NBT. In the presence of NBT, the spectrum shifts from 209-224.11 nm in 2 days, showing an improved degradation action.

Fullerene 60 Nanoparticles

The Fullerene 60 nanoparticles [18] have shown a similar degradation study when used in minimal broth Davis without dextrose to influence the growth cycle of LDPE degrading bacterial consortia. These nanoparticles used at 0.01% (w/v) concentration due to the fact that they are detrimental to bacterial growth at higher concentrations like 0.25, 0.5, 1%... etc. LDPE used with Fullerene 60 at a concentration of 5 mg/ml [19] have worked with these nanoparticles on two different consortia [5]. The bacterial strains are LDPE degrading, isolated from degrading polyethylene. They even have capability of degrading HDPE, epoxy and epoxy silicon blends [8,17,20,21]. It acts as growth accelerator for enzymes responsible for plastic waste degradation.

The bacteria growth curve analysis using UV Spectrophotometer is done to know the effect of fullerene 60 on individual strains and on consortia. The degradation process is carried out in presence of sonicated nanoparticles and the degraded product is obtained after the consortia has reached stationary phase. The degraded product obtained after centrifugation and evaporation is analyzed and characterized using FTIR (Table 2) and TG-DTG-DTA (Tables 3 and 4), where LDPE is taken as control. The polymeric structure changes making a shift in λ_{max} is observed from 209 nm, constant for 2 days, to 220 nm, after 3 days and to 223 nm after 4 days, in absence of Fullerene 60 nanoparticles. Whereas, in the samples with fullerene 60 λ_{max} was found to shift from 209-224.97 nm on 1st day itself, showing an improved rate of degradation.

Supermagnetic ironoxide nanoparticles

In a comparative analysis carried by Kapri et al. [5] Supermagnetic Iron Oxide Nanoparticles (SPION) have been studied in the similar way as that of NBT and Fullerene 60 nanoparticles. The SPION of particle size 10.6 nm was used for enhancing the consortium growth. The consortium considered here is Consortium I, mentioned in Table 1. The nanoparticles here have been synthesized by co-precipitation method with ammonia using ferrous chloride and ferric chloride solutions [18,20-31]. The apt particle size is obtained by XRD. The same degradation process was carried out and the analysis was done by UV, FTIR (Table 2) and Thermal Analyzer (Tables 3 and 4).

Where, UV spectrophotometer gave similar results for pure LDPE as in the case of NBT [17] and Fullerene 60 [18]. But LDPE with SPION

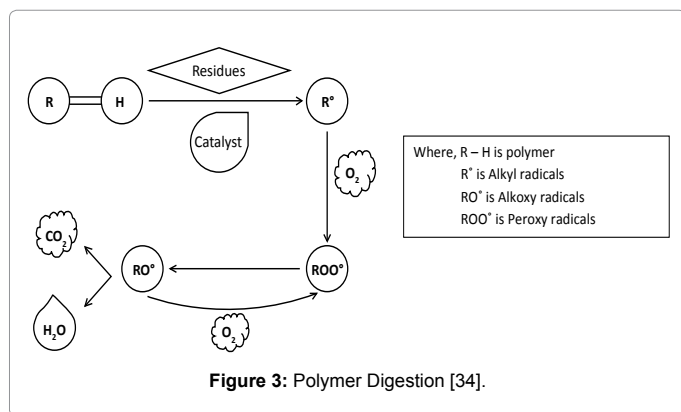


Figure 3: Polymer Digestion [34].

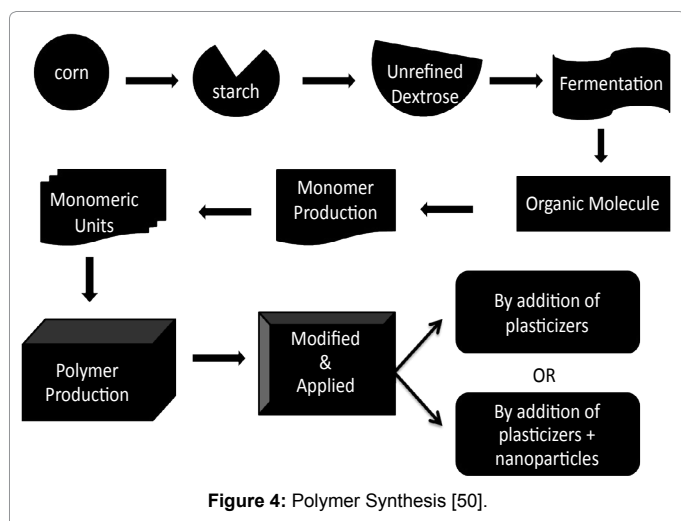


Figure 4: Polymer Synthesis [50].

Consortium I [17]	Consortium II [5]
<i>Microbacterium</i> species strain MK3,	<i>Pseudomonas aeruginosa</i> strain PS1
<i>Pseudomonas putida</i> strain MK4	<i>Pseudomonas putida</i> strain PW1
<i>Bacterium</i> Te68R strain PN12	<i>Pseudomonas aeruginosa</i> strain C1

Table 1: Composition of Consortia.

Pure LDPE [5,17-19]	LDPE + Consortia I [5,17-19]	LDPE + Consortia II [18]
Combination bands δCH_2 2151.8 cm^{-1} & ρCH_2 2368.0 cm^{-1}	Combination bands δCH_2 2370.3 cm^{-1} & ρCH_2 2370.0 cm^{-1}	Combination bands δCH_2 2361.0 cm^{-1} & ρCH_2 2401.8 cm^{-1}
δCH_2 1465.2 cm^{-1}	δCH_2 1443.5 cm^{-1}	δCH_2 1426.9 cm^{-1}
$>\text{CH}_2$ deformation 1595.1 cm^{-1}	$>\text{CH}_2$ deformation 1658.9 cm^{-1}	$>\text{CH}_2$ deformation 1663.9 cm^{-1}
C-O frequency not observed	C-O frequency 1023.7 cm^{-1}	C-O frequency 1023.0 cm^{-1}

Table 2: Comparative analysis of FTIR results for Pure LDPE and LDPE+Consortia.

Samples	TG	
	Temperature of Occurrence	Step Weight Loss in % (at respective temperatures)
Pure LDPE [17,18]	400-460°C	One step steep weight loss
LDPE + Consortia I [17]	61°C	5.33%
	200°C	9.47%
LDPE + Consortia I + NBT [17]	60°C	3.88%
	200°Cs	9.01%
	274°C	11.32%
	380°C	14.34%
LDPE + Consortia I + Fullerene 60 nanoparticles [18]	126°C-155°C	12.5%
	230°C	15.7%
LDPE + Consortia I + SPION [5]	98.1°C	1.9%
	112°C	4.4%
	130°C	5.2%
	Above 200°C	8%

Table 3: Thermal Gravimetric analysis data of pure LDPE and LDPE with nanoparticles.

	DTG		DTA	
	°C	mg/min	°C	μJ/mg
Pure LDPE [17,18]	451	1.79	107 457	129 53
	56 205	0.2116 0.0714	58 207,248 362	143 120 5.03
LDPE + Consortia I + NBT [17]	56	0.1423	47	22.5
	190	0.0367	117	13.3
	273	0.0384	144	44.8
	368	0.0185	222	135
LDPE + Consortia I + Fullerene 60 nanoparticles [18]	144°C	0.3220	146°C	0.167
	220°C	0.0500	221°C	0.115
Where, in the case of Fullerene 60, the first step was recorded as rapid and second step as steady. Consortium I gave better results with both NBT and Fullerene 60 [16,18].				
LDPE + Consortia I + SPION [5]	44°C	0.048	46°C	18.3
	110°C	0.052	113°C	12.5
	144°C	0.221	145°C	100
	218°C	0.050	222°C	90.2

Table 4: DTG – DTA data of pure LDPE and LDPE with nanoparticles.

a sharp shift in λ_{max} was observed from 209 nm to 222.6 nm in a day and then it shifted to 225.62 nm in next 2 days.

The result obtained after the analysis have been provided in Tables 3 and 4. Where, the pure LDPE shown degradation in One-step weight loss and LDPE with SPION shown degradation of above 8% in Four-step weight loss.

C-O frequencies (Table 2) are not seen in case of Pure LDPE but have been observed in both LDPE with only consortia and LDPE with consortia+nanoparticles due to inclusion of O-atoms in hydrocarbon chain in latter case, where in the case of LDPE+Consortia+Nanoparticles similar peaks with reduced transmittance were observed with C-O frequency of 1044.3 cm^{-1} [17] and 1,102.1 cm^{-1} [5] In case of NBT (difference in the two values reported in two separate researches conducted by same authors, it is due to difference in the sample used), 1088.7 cm^{-1} and 1087.0 cm^{-1} in case of Fullerene 60 with Consortia I and II, respectively [18] and 1101.1 cm^{-1} in case of SPION [5]. Consortium I and Consortium II has shown a weak peak of C-O frequency at

1023.7 cm^{-1} and 1023.0 cm^{-1} , respectively, proving the sole efficiency of the consortium. This proves that the nanoparticles and consortia have helped to capture O_2 in the hydrocarbon chain which has further helped in degradation by bioactive hydrolysis [5]. C-O bond is fastened by enzymes possessed by Consortia (Figure 4).

The *in vitro* biodegradation has even been continued to *in situ* processes [5,32,33]. Although no significant changes were observed through FTIR, and thermal profiles were also more or less same, this might be due to the fact that degradation is a surface phenomenon and has little affect on the inner layers of film. Molecular mass changes were significant in case of biodegradation with nanoparticles in comparison to the control i.e., untreated LDPE. The *in situ* analysis data has been provided in Table 5 and the SEM micrographs with Consortium I and II+LDPE and Consortium+LDPE +Nanoparticles have been depicted in Figures 5 and 6.

For higher degradation rate and stability of Polyethylene, Polyethylene nanocomposites (PE nanocomposites) can be prepared

either with the methods mentioned above. These have higher stability in terms of heat of distortion temperatures, flame resistance, Young's modulus, barrier properties and thermal expansion coefficient [34].

As the studies carried out with PE Nanocomposites under biotic conditions and UV irradiations, says they help the polymer to bind O₂ for a longer duration, and even causes photodegradation, so that it can decrease the degradation time by being available to polymer[34].

Doped and undoped TiO₂ with LDPE

TiO₂ is a semi-conductor and has been reported to have Photocatalytic Activity [35], which efficiently acts on gaseous and water contamination and is therefore used in purification. It is also been reported for removal of volatile organic compounds from indoor air, e.g. Dichloromethane...etc [36]. It has been implicated on LDPE for attaining effective biodegradation due to its photocatalytic activity [35]. The UV irradiation is responsible for efficient initiation of activity. But the absorbance it gives is below 380 nm and not in visible range, causing a reduction in the activity *in vivo*. Thus, doping of TiO₂ with metal dopants has been applied to overcome it by causing a shift in absorbance by modifying its chemical appearance and helps in absorbing near visible range and causes the photocatalytic activity initiation. There are a number of dopant metals that can be used. Iron, Silver and Iron with Silver have been mentioned as dopants for LDPE degradation [35] (Table 6).

Doped TiO₂ nanoparticles can be prepared by addition of metals at 1% concentration of TiO₂ in 100 ml Distilled Water and then kept for 24 hours incubation at room temperature, after which these are dried in oven at 100°C for 12 hours. These are ground in mortar and calcinated at 500°C for 3 hours. Similarly, Fe and Ag mix TiO₂ nanoparticles

are prepared by adding the dopants in 0.5% concentration of each with respect to TiO₂ amount. Whereas, in the case of undoped TiO₂ nanoparticles, TiO₂ is directly calcinated in a furnace for 3 hours at 500°C.

The LDPE-TiO₂ film were prepared and then characterized by XRD using Scherer Equation, SEM and EDS, where SEM has shown uneven distributions of dopants before degradation and Energy Dispersive Spectroscopic (EDS) analysis shown that the amount of TiO₂ is not consistent.

The photolytic and photocatalytic degradation of the undoped and doped TiO₂ samples is carried under UV light rays for 300 h, respectively. The direct photons from the UV rays irradiated the nanocomposite surface, which causes TiO₂ to enter excited level and thus cause chain scission, branching and cross linking by oxidation reactions. Pure LDPE has shown some cavities due to escape of volatile components in polymer and doped PE has shown greater cavity due to greater escape of amorphous TiO₂ nanoparticles under SEM, hence higher degradation. FTIR analysis proved the presence of C-O and -OH frequencies with respect to pure LDPE due to incorporation of oxygen species and hydrolysis, respectively and also, the increased thickness caused dispersion of film, which further verified the results.

Higher degradation is caused in doped TiO₂ nanocomposites due their capability to act both as holes and electrons, due to which they are able to adsorb more oxygen compared to undoped TiO₂ and further release hydrogen peroxide to attack the remaining matrix; although the undoped TiO₂ degrade the sample by photolytic activity, which is showing a greater degradation than pure LDPE. So, according to the degradation ability of all three following can be deduced:

Samples	Incubation Period (in Months)	Observations based on obtained SEM micrographs
Untreated LDPE [5,32]	0	Smooth Surface
	1	Smooth Surface
	2	Smooth Surface
	3	More or Less Smooth Surface
LDPE + Consortia [5,32]	0	Smooth Surface
	1	Inflicted Degradation
	2	Cracks and Fissures (visible only at higher magnifications)
	3	Cracks and Fissures widened with their increased frequency of occurrence
LDPE + Consortia + Nanoparticles [5]	0	Smooth Surface
	1	Inflicted Degradation
	2	Cracks and Fissures (Even visible at lower magnifications)
	3	Deepened Cracks, Fissures with high number of whitened areas and cavity formation giving rise to globular white areas due to bacterial penetrations (Even visible at lower magnifications)

Table 5: *In situ* biodegradation data of LDPE samples from soil in a period of 3 months.

Nanocomposites	XRD	EDS			Weight Reduction		
		Ti (%)	Fe (%)	Ag (%)	UV (%)	Artificial (%)	Darkness
Pure LDPE	-	-	-	-	3.32	0.65	NO R E D U C T I O N
Undoped TiO ₂ + LDPE	42.52-26.72	100	-	-	10.6	6.51	
Fe/TiO ₂ + LDPE	42.52-26.72	99.08	0.92	-	13.49	11.9	
Ag/TiO ₂ + LDPE	42.52-11.27	99.09	-	0.91	13.75	14.8	
Fe + Ag/TiO ₂ + LDPE	42.52-20.09	98.99	0.48	0.53	14.34	13.18	

Table 6: Analyzed data for TiO₂ nanocomposites [35].

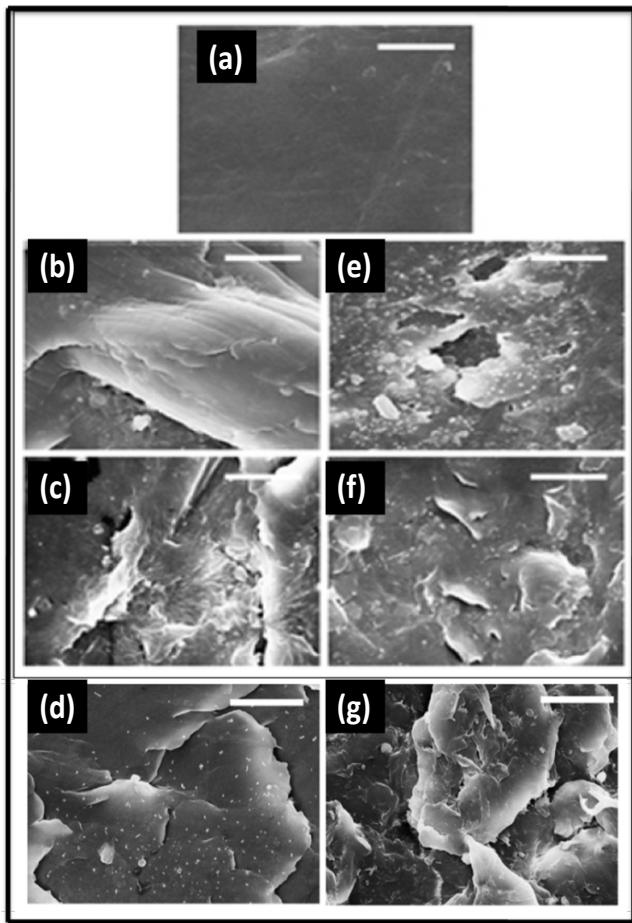


Figure 5: *In vitro* biodegradation of LDPE in presence of Consortium I (b and c) and Consortium II (e and f) depicting before and after in comparison to untreated LDPE (a). (d) and (g) depicts the biodegradation in presence of Nanoparticles SPIO and NBT, respectively with Consortium I. [5,17].

Doped TiO_2 +LDPE>Undoped TiO_2 +LDPE>Pure LDPE

Hazardous effects of TiO_2

A number of authors have conducted studies using TiO_2 and have documented it as environment friendly and safe [37-41]. But a research conducted has shown the amorphous TiO_2 and SiO_2 particles as hazardous when inhaled and have lead to water pollution affecting the fishes in the tested areas. The research has even found a certain cardiovascular diseases and cytotoxicity related to these particles and can cause Central Nervous disorders by translocation through the nasal cavity. The soil also gets polluted by TiO_2 , as it releases fumes while degradation and is even responsible for Cadmium accumulation in fish [42-46].

So to reduce the hazardous effects the nanocomposites must be recycled and should be used to the fullest of the life cycle. Although it is not an option to be considered for TiO_2 films nut surely a suitable alternative for SiO_2 . A research related to the field has been found beneficial without compromising the polymer properties [47]. A better fixation of nanoparticles in the polymer will help the alternative in proving it to be better in use. In both the cases, using the antioxidants and changing surface properties [42,43,48,49] of nanoparticles might

reduce the related hazards. If the larger sized particles are used, they are less hazardous [43].

Inference and Future Prospects

The weighty accumulation of plastic waste and less effective measures for its management; LDPE degradation has become a serious issue to be dealt with. Microbial degradation has been proved to be effective in certain ways. So, the researchers have found nanoparticles as enhancers of microbial degradation ability.

Nanobarium Titanate, Fullerene 60 and Supermagnetic iron oxide (SPION) have been documented to degrade LDPE with the help microbial activity enhancement. Whereas, Doped TiO_2 and Undoped TiO_2 nanoparticles have been embedded in the LDPE matrix while its synthesis to enhance degradation by known photocatalytic activity of TiO_2 that occurs in presence of UV light and doping helps to shift the absorption range, to perform photocatalytic degradation in the presence of visible light.

Notwithstanding to environmental unfriendliness, TiO_2 has been found to be cost-effective and efficient in degradation enhancement. Thus, measures for reduction of environmental hazards have been designed, where a few documented are use of antioxidants, changing surface properties, use of larger sized particles and a better fixation in the matrix.

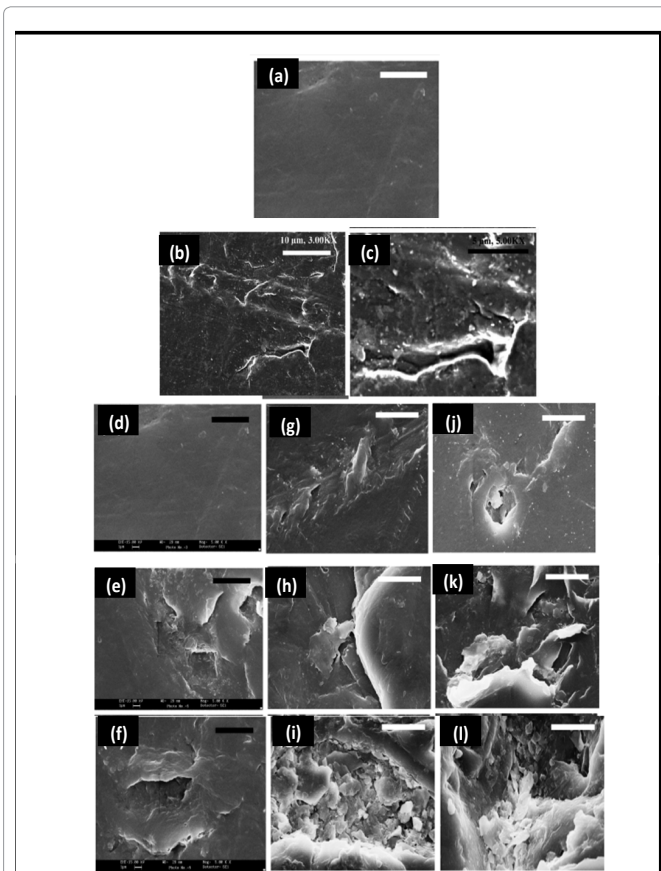


Figure 6: In situ biodegradation of LDPE in presence of Consortium I (b and c) and Consortium II (e and f) depicting before and after in comparison to untreated LDPE (a). (d) and (g) depicts the biodegradation in presence of Nanoparticles SPIO and NBT, respectively with Consortium I [5,32].

Nanoparticles, being a new field in the advancement of study have not been much explored. Therefore, are of great interest, as found to be valuable in researches conducted. So, this aspect has got potential to be explored with other documented nanoparticles like Cobalt ferrite and other silica based nanoparticles that influence the bacterial growth profiles. The degradation mechanisms are also not known in much detail and consequently open a new direction for biodegradation related studies, to make the area sound and helpful at commercial level. Therefore, the implications of nanoparticles have got wide applications as biodegradation enhancers of synthetic plastics and can act as a benign solution.

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