

Degradation of Polyfurfuryl Alcohol-based Biopolymer by Soil-burial and Photo-degradation Methods

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Abstract

Recently, polyfurfuryl alcohol (PFA) based material has been gaining attention. Despite its use as an intermediate in various industries, the degradation process of PFA has rarely been reported. In this study, neat PFA (PF) and polylactic acid (PLA) incorporated PFA (PF-PL) based thermoset biopolymers were prepared by casting method. The degradation of the prepared biopolymer specimens was carried out under environmental conditions via soil-burial test and photo-degradation method for 21-months. The extent of degradation of PF and PF-PL was assessed by evaluating weight loss, structural and morphological change by Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM), respectively. Weight loss percentage in case of photo-degraded samples was found to be much higher compared to soil buried specimens. SEM micrographs showed a blistered surface with visible cracks on the surface of soil buried and photo-degraded samples. FTIR spectra of photo-degraded samples showed a new peak at 673 cm^{-1} indicating the furan ring opening during the degradation process. Significant variation in mechanical properties of PF and PF-PL specimens after soil-burial test also indicated biodegradable nature of the biopolymers. Approximately 45% and 63% of loss in tensile strength was obtained in PF and PF-PL soil buried specimens, respectively. All the obtained data revealed the fragmentation of biopolymers, hence supporting the biodegradable nature of PFA-based biopolymer.

Introduction

In the last two decades, the world has become more aware and concerned about the environment. It is a well-known fact that the surplus use of synthetic polymers has adverse effects on our ecosystem. Disposal of these polymers has become the biggest threat due to its recalcitrant nature. Only about 20 lakh tonnes of plastics were produced per year till 1950. Since then, the production reached 381 million tonnes by 2015, which is an increase of almost 2000 times [1]. About 10 lakhs of plastic drinking bottles are consumed worldwide, every minute and, there is the utilization of around 5 trillion single-use plastic bags every year. For the last 60 years (starting 1950), an estimated 8.3 billion tonnes of plastic have been produced and, 60% of the produced plastics have been dumped into the natural environment [1]. The primary source of plastic production is non-renewable sources e.g., oil, natural gas and, coal. If this trend continues, plastic production could consume almost 20% of all oil production in the next few decades [1]. Above data is only related to plastics, mainly comprising polyethylene, polystyrene, polypropylene and polyvinyl chloride. The actual situation is even more drastic as there are enormous varieties of other synthetic polymers in use. Hence, the focus has been shifted from using synthetic to natural polymers [2].

Synthetic thermoset resins along, with epoxy and phenolic resins, hold significant industrial interest. Polyfurfuryl alcohol (PFA) is one of the thermoset biopolymers having potential to meet the requirements of green polymers [3]. Furfuryl alcohol (FA) is the building (monomeric) unit of PFA biopolymer which can be obtained from wastes of sugarcane i.e., bagasse, corn cobs, and other agricultural wastes containing pentoses. The uncontrolled exothermic reactions during FA polymerization prevent the commercialization of PFA. Kumar and Anandjiwala in 2011 reported an acid catalyzed polymerization of FA with step-wise curing of FA to PFA and hence eliminating the risk of explosion [4]. Thus, PFA-based polymers can now be

explored to prepare eco-friendly products [5]. FA-based thermoset resins have a vast array of applications as wood adhesives, binder, polymer concretes, solvent, corrosion-resistant coatings, etc. [6]. Due to heat resistant nature and good mechanical properties, PFA has high demand in foundry industries, metal casting cores, and molds. PFA also plays a key role in the production of carbonaceous products and bio-based nanocomposites [6, 7]. Nowadays, PFA is also being employed in wood stabilization, PFA- silica materials, sol-gel based nanocomposites [8].

The most alluring aspect of PFA- based biopolymers is the capacity of FA to degrade by micro-organisms in general and enzymes in particular [9]. To assess the potential of any biopolymer as commodity materials, it is important to study its degradation aspect in environmental condition [10]. Intrinsic degradation property can ultimately aid to plan the safe disposal method for used biopolymers. Also, the extent of stability of biopolymer can help to target the end-use applications of the products. In addition to this, any kind of modification (e.g. addition of additives or stabilizers) can also be apprehended for preparing an improved biopolymer with required properties [11]. Many physical and chemical ways for degradation of phenolic resins containing aromatic ring have been reported; but these methods are energy consuming and uneconomical. Hence, alternative methods like soil-burial test and photo-degradation method are being focused on degradation of both neat and modified polymer containing aromatic rings.

According to few reports, soil-burial test is considered as a viable way to study the degradation process of biopolymers [12]. In this method, polymers get attracted by various micro-organisms (bacteria/fungi), which release enzymes capable of degrading chemical bonds of polymer chains leading to the depolymerization step. It was reported by Wang et al. that specific enzymes cause primary cleavage in polymer chain, and microbes utilize the resulting residual fragments as carbon source [13]. Photo-degradation of polymers is an advanced oxidation process capable for degrading aromatic compounds [13]. According to various reports, UV radiation is one of the most destructive environmental factors. Literature states that UV radiation causes photooxidative reaction leading to polymer chain breakage with the release of free radicals. This depolymerization results in decrease in the weight and mechanical properties of the polymer. The present study attempts to degrade neat PFA (PF) and PLA incorporated PFA (PF-PL) through soil-burial method and photo-degradation. Degradation was monitored structurally and morphologically by FTIR and SEM, in addition, to change in weight and mechanical properties. The prime concern is to design such polymers which retain their strength while in use and can be biodegradable afterward. This study will provide essential information about the extents of degradability and stability of biopolymer specimens during the degradation process so that appropriate use can be planned.

Materials And Methods

Materials

Furfuryl alcohol (FA) from Sigma Aldrich, Germany, was used as a monomer for the preparation of biopolymer specimens (PFA). *p*-toluene sulphonic acid (PTSA) supplied by Sigma Aldrich, Germany, was used as a catalyst in FA polymerization. PLA fibre was purchased from Dalian Impex International trading, China. Citric acid, sodium hydroxide, and chloroform were procured from Merck, India. Silicon mold was used for the preparation of biopolymer specimens. For biodegradation of biopolymers in soil, the soil sample was aseptically collected from industrial effluent contaminated areas near the Indian Institute of Carpet Technology (IICT), Bhadoi (Uttar Pradesh), India.

Preparation of Neat and PLA Incorporated PFA Specimen

For assessing the biodegradability of FA-based biopolymers, degradation of both PF and PF-PL biopolymers has been studied. As reported by Kumar and Anandjiwala, the process of PFA formation involves condensation reaction of FA in the presence of PTSA as an acid catalyst [4]. For preparing PF samples, 10 ml of PTSA solution (0.3% (w/v) wrt FA) was added in 50 ml of FA. The acid-catalyzed FA solution was poured in a silicon mould at room temperature and kept undisturbed for about 4 h. Then, the mould containing acid-catalyzed FA was kept in an oven at 50°C for 96 h to harden (curing). Finally, the casted samples were kept for 1 h at 100°C and at 150°C for 30 min to get completely cured neat PFA biopolymeric samples [14]. To prepare PF-PL bioplastic samples, 0.5% (w/v) of PLA fabric was added in FA. The resulting mixture was magnetically stirred for about 45 min to ensure complete mixing of PLA fabric into FA. Then, the above process was followed for the preparation of PLA incorporated PFA samples also, but as reported by Sharib et al. 1st step for curing samples was carried out in an oven at 50°C for just 22 h [14]. PF and PF-PL based biopolymeric samples have been successfully prepared for degradation study.

Degradability of PF and PF-PL Biopolymers by Different Methods

Two processes, viz. soil-burial and photo-degradation test, have been performed to assess the degradation potential of PF and PF-PL biopolymers in environmental conditions. The prepared biopolymer specimens were buried 30cms deep under the soil surface (Fig. 1). Garden soil was mixed with soil sample collected from effluent, mainly aromatic compounds exposed area of Indian Institute of Carpet Technology (IICT) Bhadoi (Uttar Pradesh), India, for this purpose. The collected soil sample was first homogenized and then mixed with garden soil. The test was planned to be carried out for 21months, and samples (in triplicates) were taken out every 3months initially and then after 6months of the incubation period for the studies related to the extent of degradation. PF and PF-PL based biopolymers after 0, 3, 6, 9, 15, and 21 months of soil-burial degradation have been designated as PF-0M to PF-21M, and PF-PL-0M to PF-PL-21M, respectively.

After the designated time, the degraded PF and PF-PL based biopolymers were taken out one after another, rinsed carefully with water, treated with 0.1M NaOH, and then dried in a hot air oven at 60°C for 24 h for the weight loss measurements after biodegradation [15]. Thus, initially degradation was

evaluated by analysing change in weight of the samples (before and after degradation) and changes in the mechanical properties.

Similarly, to find out the extent of photo-degradation of PF and PF-PL based biopolymers, samples were kept under direct sunlight for 21 months. Again, the samples were taken out at every 3 months initially and then after 6 months of the incubation period for the degradation study. Prior to keeping the samples in under sunlight (UV), samples were uniformly cut and weighed [15]. PF and PF-PL based biopolymers after 0, 3, 6, 9, 15, and 21 months of photo-degradation are designated as Ph-PF-0M to Ph-PF-21M, and Ph-PF-PL-0M to Ph-PF-PL-21M, respectively.

Analysis of the Extent of Degradation PF and PF-PL Based Biopolymer

Weight Loss: Initial weights of the prepared samples were taken in order to compare the degraded and undegraded samples. The weight of solid samples was measured after an interval of three months or six months and is designated as M_t . The obtained weight was compared to the initial weight (M_0) [16].

Percentage degree of degradation (D) was then calculated as:

$$D (\%) = \frac{M_0 - M_t}{M_0} \times 100$$

Where, M_0 = Weight of undegraded sample;

M_t = Weight of degraded samples after t months.

Mechanical Properties: Any mechanical change in a polymer can be easily assessed by monitoring variation in its mechanical properties [17]. Tensile strength, elongation at break and Young's modulus of the prepared dumbbell shaped biopolymer samples (PF and PF-PL) before and after degradation were determined using the tensile testing machine (Lloyd, LR 100 K, USA) following ASTM D638 at IIT Patna, India. The test rate was maintained between 1-3mm min⁻¹.

Water Uptake: The capacity of any biopolymer to absorb water from the environment may lead to change the basic properties of the polymer. Thus, it is crucial to find out water uptake nature of PF and PF-PL along with other mechanical properties of the biopolymer before and after degradation. Water uptake of the biopolymer specimens was analyzed using the standard protocol of ASTM D570-81 [18]. The prepared biopolymer specimens were first weighed and then immersed in distilled water for 24 h at 24±1 °C. After 24 h, excess water was removed, and weight of the specimens after 24 h was noted [19]. Water uptake was calculated in terms of percentage of increase in weight as:

$$WaterUptake(\%) = \frac{W_1 - W_0}{W_0} \times 100$$

Where, W_0 = Weight of biopolymer specimen;

W_1 = Weight of specimen after immersion in water for 24 h.

Structural and Morphological Changes: Variation at the molecular level and morphological changes have been characterized by FTIR and SEM. FTIR studies of biopolymer samples (PF and PF-PL) before and after degradation were conducted using Perkin- Elmer at IIT Patna (40 scans at a resolution of 4 cm⁻¹). FTIR spectra were recorded in order to locate change in any functional group after degradation either by soil-burial test or photo-degradation method. Spectra were taken between 400-4000cm⁻¹(wave number). Further, to detect any change on surface morphology of the biopolymer specimens after degradation, SEM was performed. Surface morphology of undegraded and degraded samples in the soil buried well as photo-degraded biopolymeric samples were carried out on EVO-SEM 15/18 (Carl Zeiss Microscopy, Ltd) at an accelerating voltage of 20 kV. The samples were coated with gold prior to subjecting it for the SEM analysis.

Result And Discussion

Evaluating Degradation Depending Upon Weight Change

The extent of biodegradation (percentage) in terms of weight change obtained for PF and PF-PL biopolymeric specimens are given in Table 1. There was less than 1% degradation of the biopolymers in the soil-burial method for both neat as well as incorporated samples. In the case of photo-degradation, there was around 2.7% degradation for PF and about 3.6% degradation in PF-PL biopolymer samples. Thus, degradation in terms of weight loss is negligible for the soil-burial test as compared to photo-degradation, and the differences in the weight loss could be related to the catalytic effect of UV radiation on the degradation process of biopolymer.

Table 1

Weight change of neat and PLA incorporated sample after 3, 6, 9, 15, and 21 months interval time of soil-burial and photo-degradation tests

Degradation time (months)	Weight change (%) after			
	Soil-burial biodegradation		Photo-degradation	
	PF	PF-PL	Ph-PF	Ph-PF-PL
3	0.70 ± 0.05%	0.53 ± 0.13%	2.65 ± 0.08%	3.61 ± 0.24%
6	0.61 ± 0.31%	0.61 ± 0.12%	2.62 ± 0.25%	3.70 ± 0.14%
9	0.74 ± 0.04%	0.71 ± 0.04%	2.77 ± 0.33%	3.70 ± 0.03%
15	0.73 ± 0.05%	0.71 ± 0.04%	2.96 ± 0.37%	3.60 ± 0.10%
21	0.76 ± 0.04%	0.72 ± 0.04%	2.98 ± 0.32%	3.65 ± 0.12%

Mechanical Properties and Water Uptake

Biodegradation of biopolymers under soil-burial test was monitored through change in mechanical properties. Obtained results (Fig. 2 and Fig. 3) depict the decrease in tensile properties like tensile strength (TS) and elongation at break (%E) with the increase in degradation time (incubation time). The reduction in TS properties increases the brittleness of biopolymeric samples which, thus, can be related to the degradable nature of PFA with time.

After comparing the maximum and lowest values obtained for TS, it was found that the decrease in TS for PF specimens was around 45%, while for PF-PL based biopolymeric samples, it was around 63% after 21 months of the soil-burial test. A similar trend of tensile properties was reported with PLA incorporated PFA samples by Sharib et al. while working with different concentrations of PLA in PF-PL specimens [14]. It can thus be assumed that the incorporation of PLA enhances the decrease in TS of PF-PL biopolymeric samples. No significant changes in the weight of the specimens were obtained after immersing the degraded samples for 24 h in distilled water. Negligible water uptake by PF and PF-PL biopolymeric samples indicates the hydrophobic nature of PFA even after 21 months of degradation.

As explained by Lucas et al., degradation of any polymeric material cannot always end up in carbon recycling and mineralization. The process of degradation is a very complex event and can end up at any step [20]. So, the change in mechanical properties of the PF or PF-PL biopolymeric specimens can be due to the fragmentation of biopolymers. Based on the obtained results, we propose the degradation pathways for PF and PF-PL samples after soil-burial tests in Fig. 4. Before degradation, the PF and PF-PL specimens were long chains of crosslinked polymer (black and red lines). After biodegradation (soil-burial test), the long chain polymer, breaks into several short-chain polymers resulting in decrease in TS as observed in Fig. 2 and 3, in addition to weight loss as shown in Table 1. The minor change in weight and reasonably good TS after the soil burial method indicates the stability of PFA-based biopolymer samples supporting its use in various sectors yet showing its biodegradable nature.

Morphological Aspects

Figure 5 to 8 shows the morphology of undegraded and degraded PF and PF-PL samples. Figures 5b-6b and 5c-6c show the surface morphology of PF and PF-PL samples after 6 months and 21 months of soil-burial tests. Small holes were found on the surface of the specimens after 6 months of the soil burial process. Images of the specimens after 21 months indicated a comparatively rough and weathered surface with a large number of holes (Fig. 5c and 6c), indicating the degradation of PFA samples which is confirmed by low weight loss as well as decrease in TS. Similar results in form of small gaps and blisters were obtained in the case of PF-PL based biopolymeric samples (Fig. 6b and 6c).

On the contrary to the soil-burial test, significant changes in the form of cracks on the surface of both neat as well as incorporated samples (Fig. 7b and 8b) were obtained in photo-degraded specimens after 6 months. SEM micrographs after 21 months showed that the biopolymer surface had been degraded into various parts as the cracks must have been increased as well as deepened with time (Fig. 7c and 8c). This could be due to exposure to UV light which may have led to the removal of formaldehyde from the PFA polymeric backbone resulting in PFA with only methylene linkages (discussed later in the pathway).

These fissures and gaps observed in SEM micrographs can be correlated with the higher weight loss as obtained after photo-degradation as compared to soil-burial degradation method (Table 1).

FTIR Studies

FTIR spectra of PF and PF-PL based biopolymeric samples after the soil-burial test are given in Fig. 9. The identifying peaks of PFA are as follows: 596 cm^{-1} is attributed to the furan rings of PFA, 788 cm^{-1} and 876 cm^{-1} are related to the in-plane deformation mode of the ring and out of plane deformation of CH bonds (Fig. 9a) The peak at 1546 cm^{-1} is related to -C=C- stretching in 2-5 di-substituted furanic ring [21]. In addition to these fingerprint regions, PLA incorporated in PFA shows a peak at 1745 , which is attributed to the C=O stretching of the ester bond (Fig. 9b) [14]. FTIR spectrum obtained after the soil-burial test (Fig. 9a and 9b) shows shifting and changes in the shape of the peaks at 596 cm^{-1} and 788 cm^{-1} . The shifting in peaks to lower frequency from 1745 cm^{-1} to 1743 cm^{-1} for PF-PL based biopolymeric samples can also be observed, which may be attributed to depolymerization of the PFA-based polymer, which indicates the start of degradation of polymers. The depolymerization of PFA-based biopolymeric specimens will result in decreased mechanical properties of the degraded samples, which are confirmed from Figures 2 and 3.

Figures 10a and 10b show the FTIR spectra for photo-degraded PF and PF-PL samples. In photo-degraded samples, major peaks were almost similar, as observed in Figures 9a and 9b for PF and PF-PL samples. However, the differences in few peaks, which are characteristic of photo-degradation of PF and PF-PL samples, are highlighted here. Results of photo-degradation of both types of specimen show the appearance of a new peak at 673 cm^{-1} . This peak attributes to mono or di-substituted benzene ring [22]. During acid-catalyzed polymerization, the color of PFA changes from brown to black after polymerization; hence, the role of a chromophoric group can also be contemplated. Recently, Tondi et al. in 2019 has given pathways for the development of chromophoric group in PFA [23,24]. Thus, we may assume that during photo-degradation of PF and PF-PL samples, photo-oxidation leads to furanic ring-opening resulting in Diels-Alder rearrangements that form benzene ring. Fig. 11b pathway shows the formation of benzene ring after Diels alder reaction [25]. However, this is possible only if the acid catalyzed reaction follows a pathway where PFA is joined by only a methylene linkage that is obtained after the removal of formaldehyde from PFA samples during photo-degradation Fig. 11a.

Conclusion

In-depth knowledge of the degradability nature of biopolymer is crucial for developing efficient PFA biopolymer and achieving the way it has been targeted to be used. Change in weight, as well as TS of soil buried specimens, can be considered as a positive indication of the degradable nature of the biopolymer. SEM images clearly showed the modification on the surface of the polymers in the form of holes after the soil-burial degradation process. It is noteworthy to mention that these results also help to predict the stability of biopolymer when used in various products. The shift in bands of degraded samples after soil-burial degradation and decrease in TS indicates that the process of depolymerization may have taken

place. In photo-degraded PFA or PLA incorporated PFA, there is a higher magnitude of weight change. SEM images of photo-degraded samples clearly showed various cracks, which can be due to the emission of formaldehyde. The appearance of a new peak at 673 cm^{-1} in case of photo-degraded samples indicates the aromatization of PFA ring. It may be assumed that the PFA samples degrade slowly under soil-burial degradation as compared to the photo-degradation process. However, further study is needed to unfold the steps required for the processing of PFA-based polymers to be utilized in different sectors. Like the addition of stabilizers or antioxidants to prevent the crack in PFA based biopolymers upon exposure to sunlight. Moreover, for degradation of the biopolymers combination of both photo-degradation and soil-burial may give more efficient degradation results. PFA-based biopolymers can be first exposed to direct sunlight, which will catalyze the depolymerization process and then subjecting it for microbial degradation via the soil-burial method.

Declarations

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Conflicts of interest/Competing interests The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work cited in this paper.

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Code Availability Not applicable

Author's contributions Statements are not mandatory

Ethics approval Not applicable

Consent to participate Not applicable

Consent for publication Only subscription based

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Figures

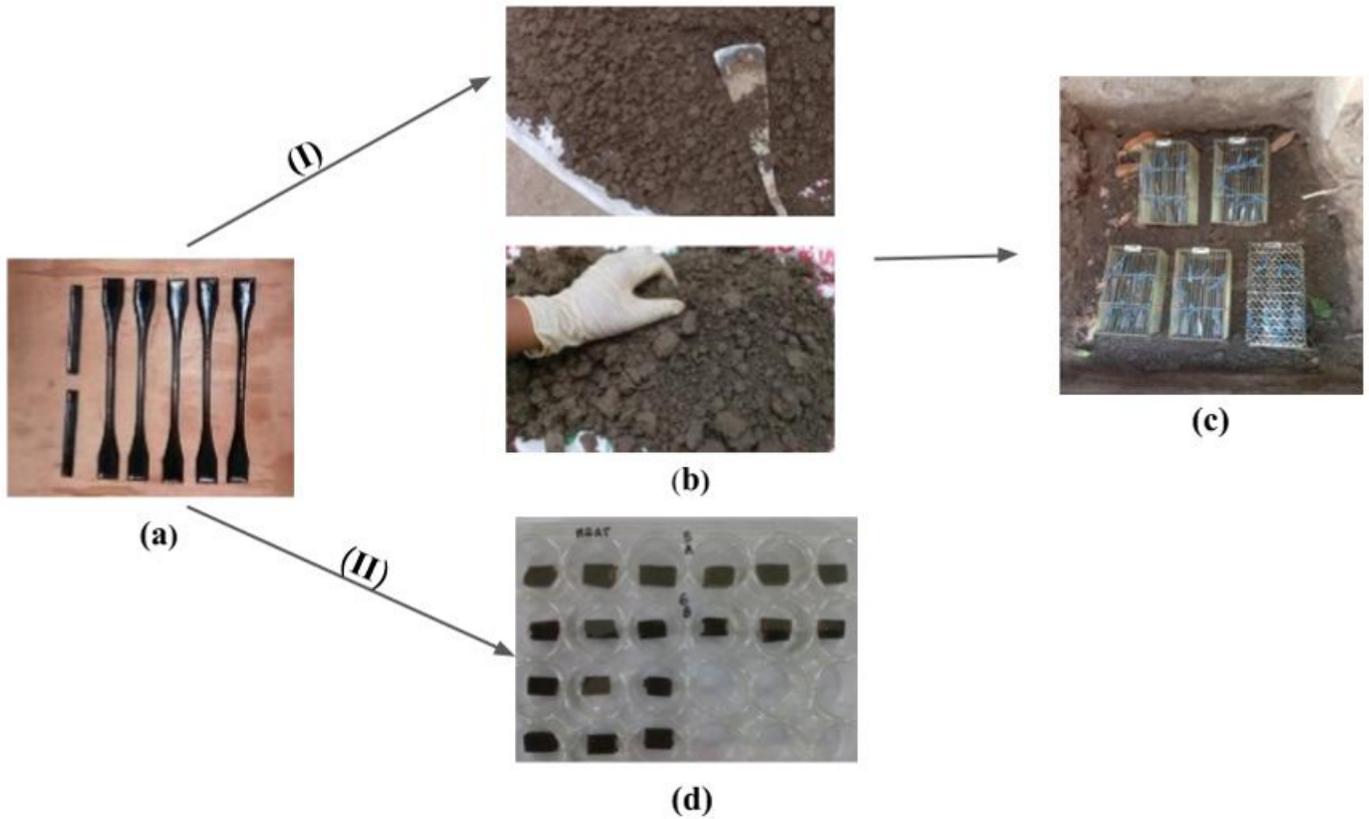


Figure 1

(a) Photograph showing prepared biopolymers, (b) Homogenization of soil sample collected from IICT, Bhadoi, India (c) Biopolymer samples tied in wire boxes and buried 30cms below the soil surface, (d) Setup showing biopolymer specimens inside a transparent box to be kept under direct sunlight. In the Figure, mode I and II represent soil burial degradation process and photo-degradation method

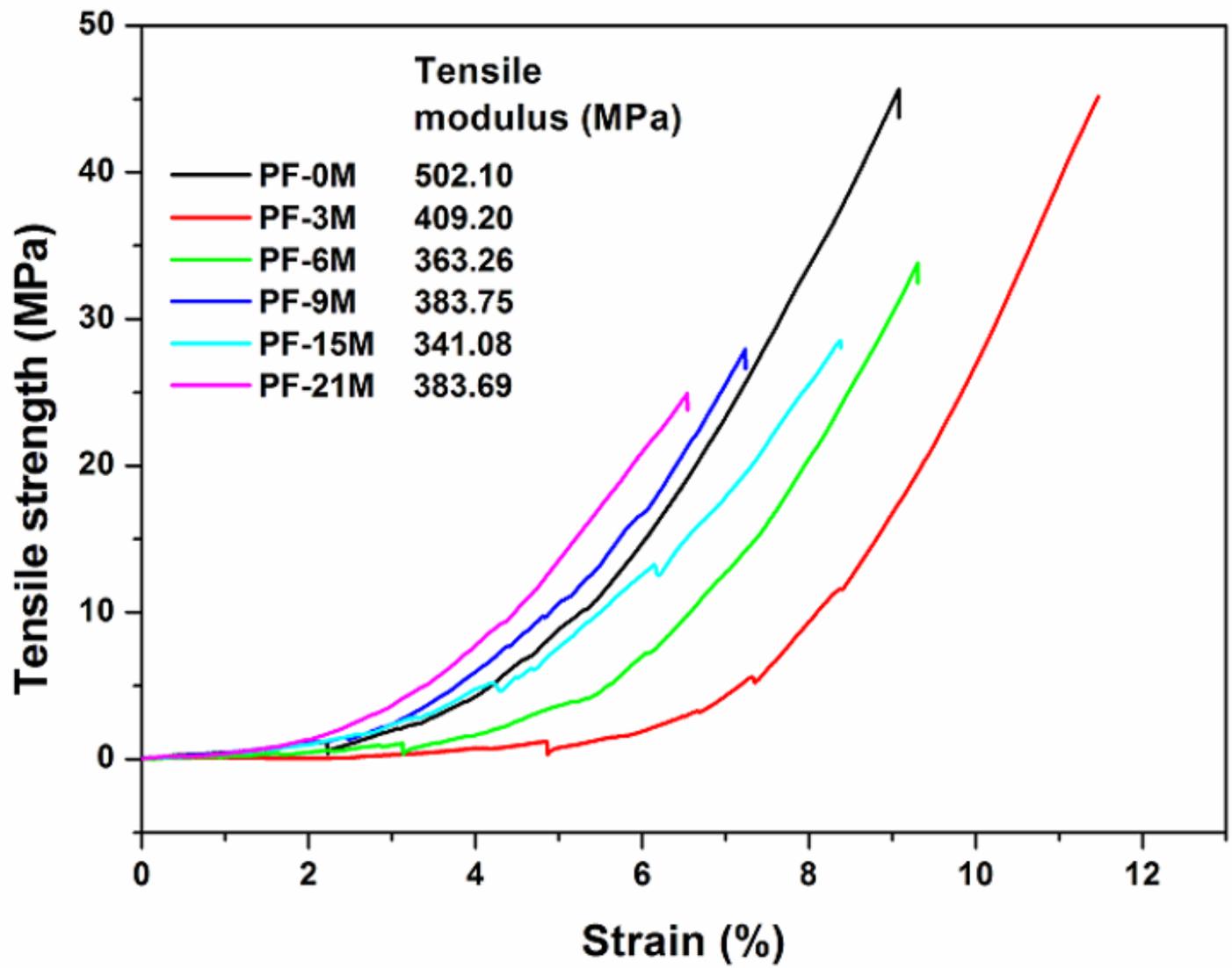


Figure 2

Change in tensile strength of PFA based samples with respect to months after soil burial test

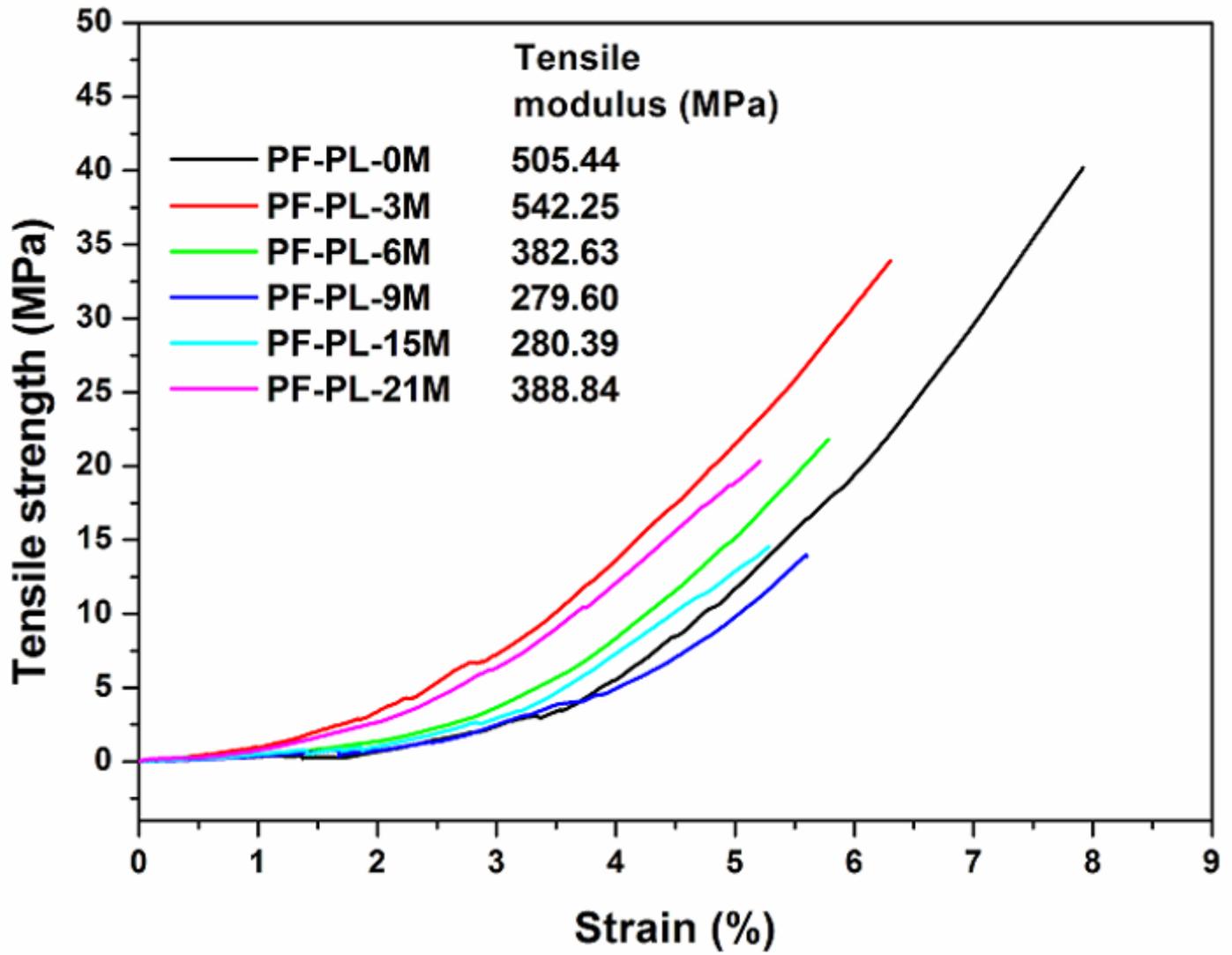


Figure 3

Change in tensile strength of PF-PL based samples with respect to months after soil burial test

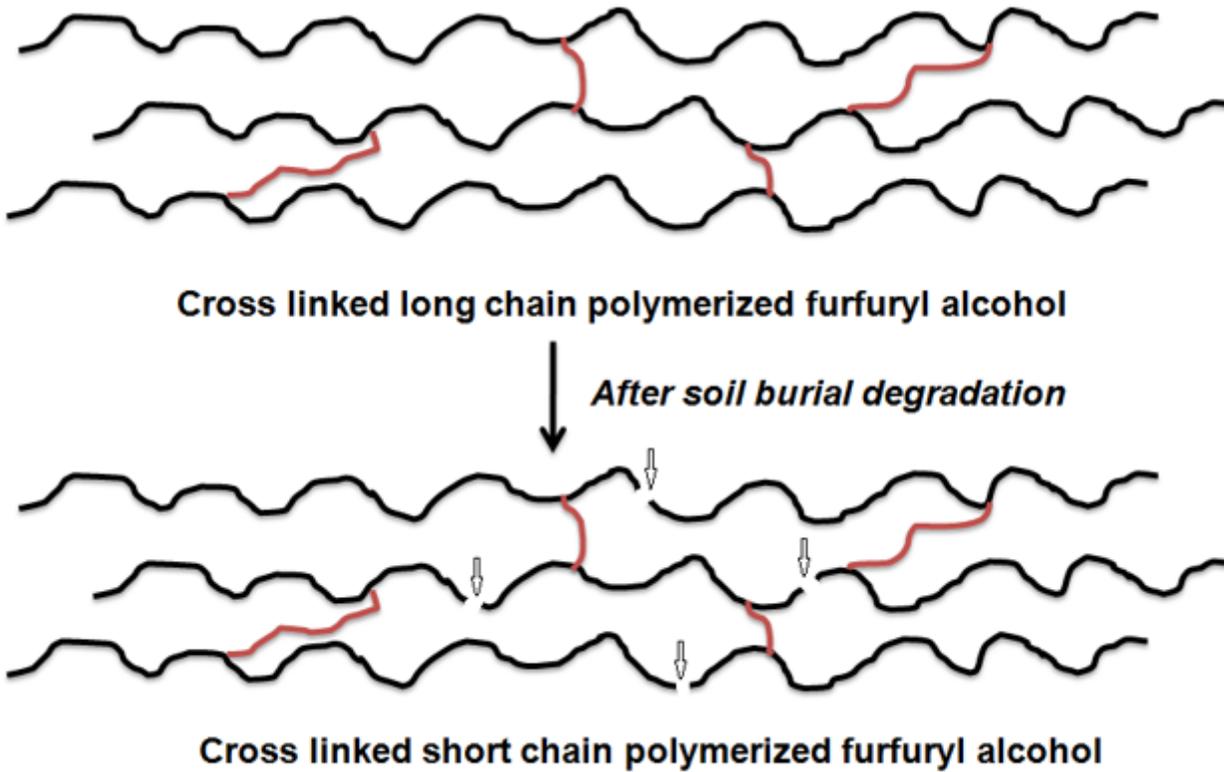


Figure 4

Scheme showing breaking of long chains to short chains of PFA after soil-burial degradation. In the scheme, black wavy lines represent PFA and red lines represent the crosslinked structure. The arrow represents the point at which chain-breaking takes place

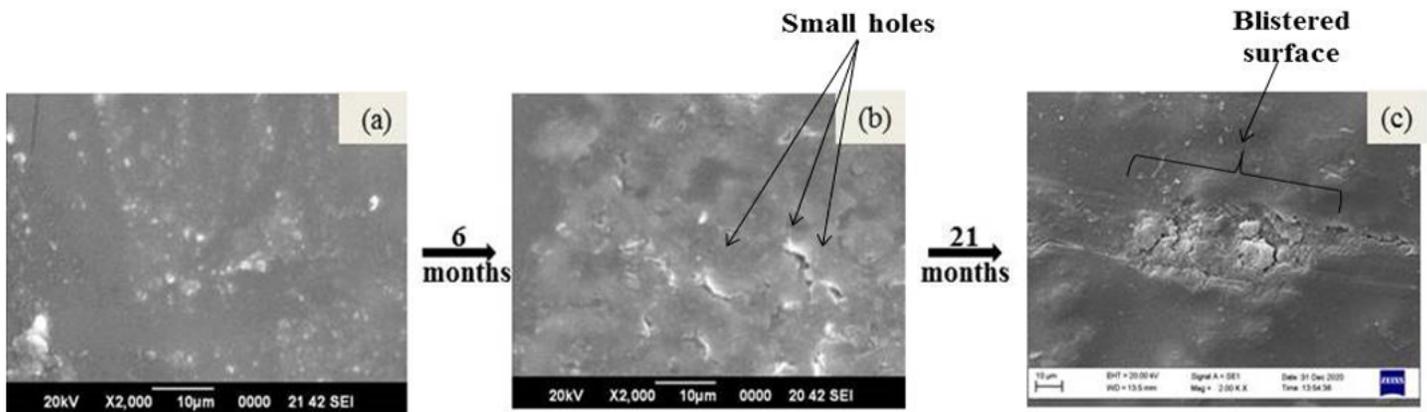


Figure 5

SEM image of neat PFA specimen after (a) 0 day (b) 6 months (c) 21 months of soil-burial.

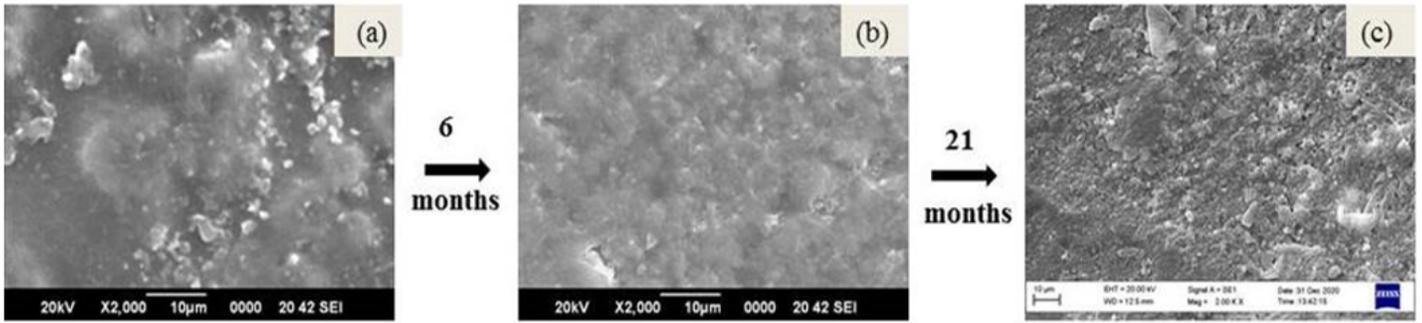


Figure 6

SEM image of PLA incorporated PFA specimen after (a) 0 day (b) 6 months (c) 21 months of soil-burial

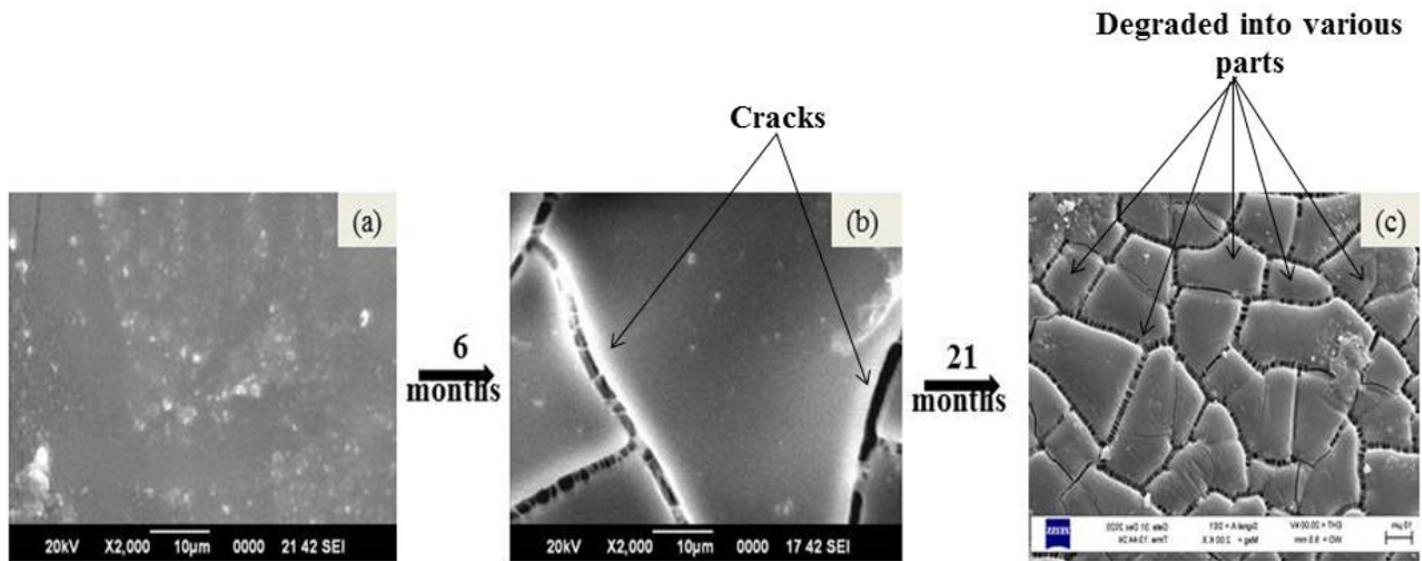


Figure 7

SEM image of neat PFA specimen after (a) 0 day (b) 6 months (c) 21 months of photo-degradation

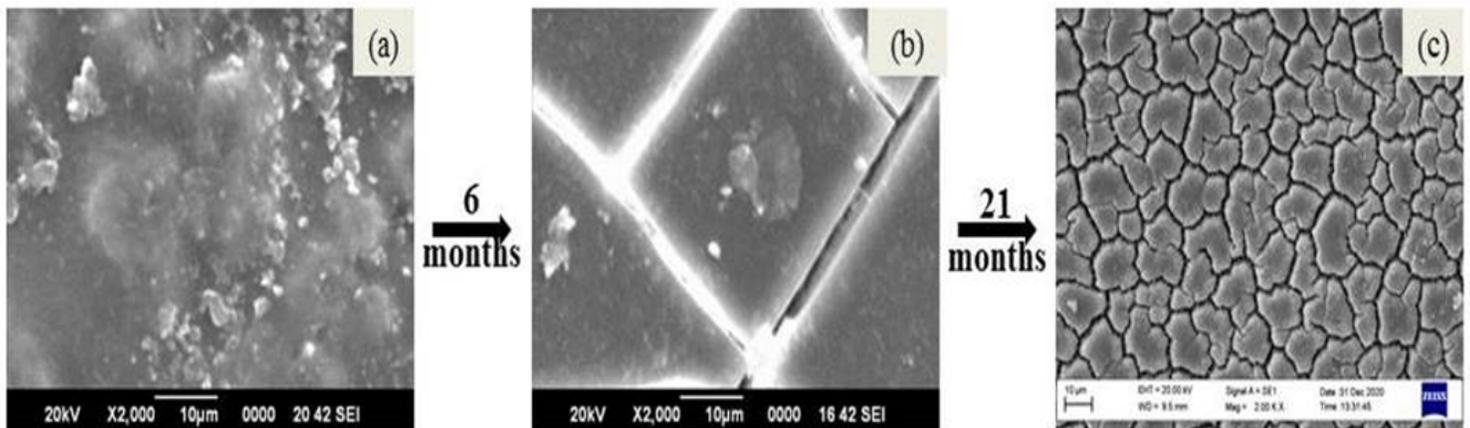


Figure 8

SEM image of PLA incorporated PFA specimen after (a) 0 day (b) 6 months (c) 21 months of photo-degradation

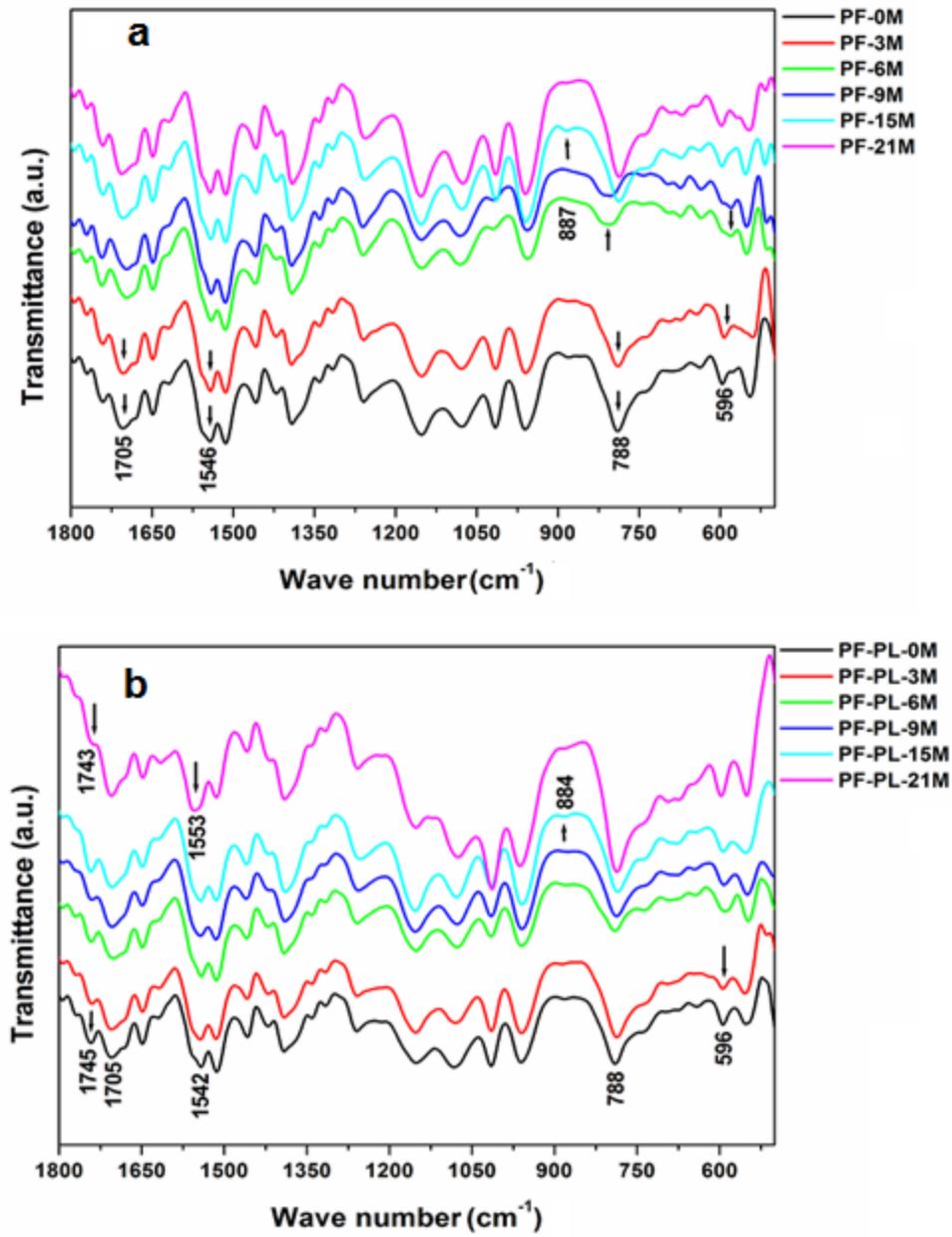


Figure 9

FTIR spectra of (a) PF and (b) PF-PL after soil-burial test

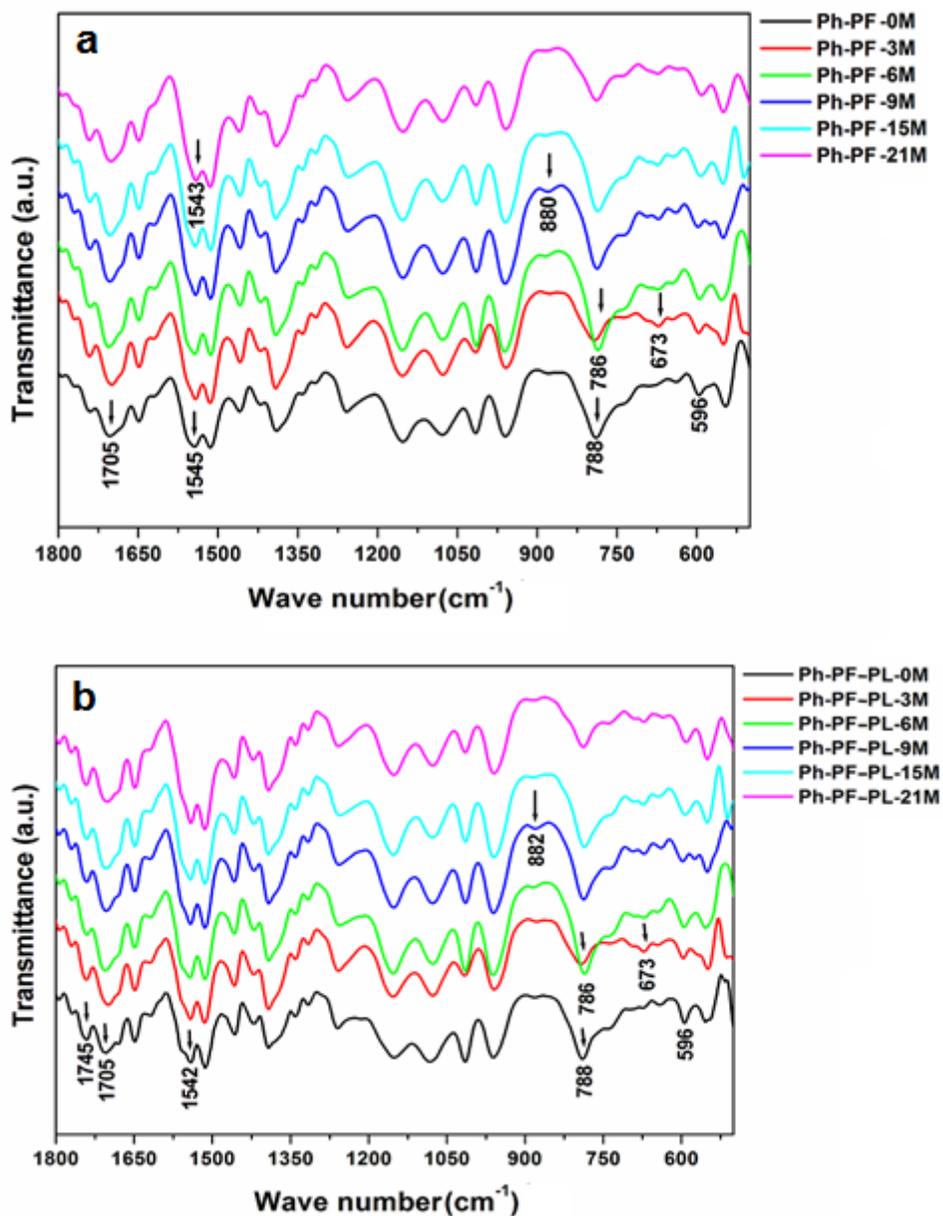


Figure 10

FTIR Spectra of (a) PF and (b) PF-PL after photo-degradation test

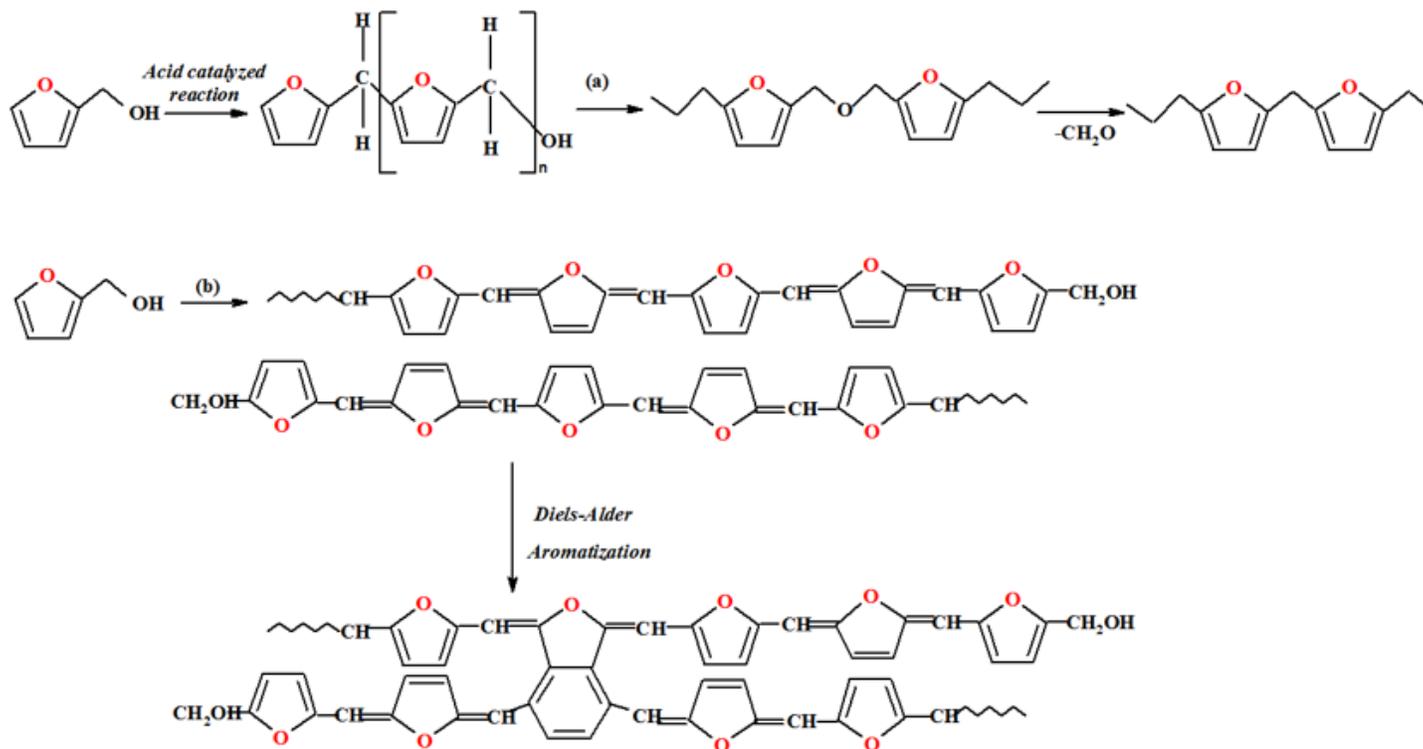


Figure 11

Arrangement of PFA (a) linear (b) conjugated and related reactions during photo-degradation (Adapted from Ref. 23 (Open Access Journal))