



Waste Cooking Oils in Biodegradable Polymer Conversion Process

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Abstract. Polymers, such as plastics and coatings, are susceptible to photo degradation initiated by heat or Ultra Violet (UV) light during processing or long-term use. In preventing the photo degradation of polymers, the sustainable polymer composite (SPC) is doped with different percentage of Titanium Dioxide (TiO₂). Hydrolyzed monomer of virgin cooking oil (VO) and waste cooking oils (WO) were mixed with hardener and solvent to form polymers namely as virgin oil polymer (VOP) and waste oil polymer (WOP). The mixture was stirred using mechanical stirrer at optimum condition for about 60 min at 50 °C resulting viscous compound. The VOP and WOP was leave to cure at ambient temperature for at least 6 h, as to ensure complete removal of solvent traces. Once dry, the film was stripped from the plate. The above steps were repeated to fabricate sustainable polymer composite (SPC) doped with different percentage of TiO₂. The samples of BP are based on VOP and WOP while BPC was namely according to TiO₂ percentage as VOP2.5, VOP5, VOP7.5, and VOP10; WOP2.5, WOP5, WOP7.5, and WOP10 respectively. Micrometer was used to measure the thickness of the sample at particular point ranging from 110 μm to 250 μm accordance to ASTM D1005-95. The above steps were repeated to fabricate sustainable polymer composite (SPC) doped with different percentage of TiO₂. The UV accelerated weathering test was conducted according to ASTM D 4587-Standard practice for fluorescent UV-condensation exposures of paint and related coatings. Furthermore, tensile test was conducted to determine the strength and ductility of a material. The mechanical properties for tensile test of polymer thin films upon UV irradiation for photo degradation were significantly reduce its reduction strength. As compared to WOP, VOP gives better result for tensile strength and elongation at break.

Keywords: polymers · photodegradation · TiO₂ · ultra-violet · hydrolyzed · monomer

1 Introduction

In recent years, increasing interest in the development of more environment friendly polymer products such as plastics is observed. This trend has been spurred not only by the realization that the supply of fossil resources is inherently finite, but also by a growing concern for environmental issues, such as volatile organic solvent emissions and recycling or waste disposal problems at the end of a resin's economic lifetime. Furthermore, developments in organic chemistry and fundamental knowledge on the physics and chemistry of paints and coatings enabled some problems encountered in vegetable oil based products to be solved. This resulted in the development of coatings formulations with much improved performances that are based on renewable resources [1].

Palm oil is one of the most widely used plant oils in the world, which is grown in mass plantation in tropical countries. The competitive environment of the industry provides the drive needed to develop plastic materials that utilize less expensive material. Palm oil differs from its major competitors (soybean, sunflower seed, and rapeseed oil) in that it is obtained from a perennial tree crop and drought impacts are less severe in comparison to oilseed crops. Palm oil which contains significant amount of saturated bonds that presumably contribute to the non-drying property of the resin synthesized.

Preparation of the alkyd resin from non-drying palm oil may expand the application of the oil in various areas as environment-friendly materials, because of its abundance and renewability. There is no study reported on the synthesis of alkyd resin based on palm oil so far. However, the use of petroleum based monomers in the manufacture of polymers is expected to decline in the coming years because of spiraling prices and the high rate of depletion of the stocks. This has inspired the technologists all over the world to investigate renewable natural materials as an alternate source of monomers for the polymer industry as substitute for the petroleum-based monomers to manufacture polymers [2].

The evaluation of the resistance to weathering of materials can be done by direct weathering outdoors, but for most purposes it is more practical in economical and time consumption terms to assess material performance by exposed to artificial light sources that accelerate the degradation. Degradation of polymeric materials by exposure to solar radiation or light is referred to as photo-oxidation. It is a free radical process, progressing even at low temperatures by the combine action of light and oxygen. Thermal oxidation is always superimposed on photo-oxidation [3].

Under the action of sunlight, polymer materials undergo a series of oxidative reactions that lead to chemical degradation, with consequences like brittleness, loss of brightness, colour change, opacity and formation of surface cracks. Besides the reduction in molecular weight, a number of changes take place in the molecules during photodegradation with the formation of chemical groups like carbonyl, carboxylic acids and hydroperoxides [4].

Products like fibres and films tend to deteriorate under UV exposure to UV light, resulting mainly in fragility and loss of transparency. The degradation and stabilization of some types of polymer, like polyethylene were extensively investigated throughout the years and hence the degradation mechanisms and their controlling factors are reasonably well established [5]. The common polymers normally photo degrade are fairly well

known, but various aspects of the mechanisms involve remain unclear. It is important to take into account very significant influence of compounding additives in modifying the chemical pathways, which are pigments, extenders, photo stabilizers and thermal stabilizers [6].

Normally polymers such as plastics are susceptible to auto-oxidation initiated by heat or UV-light during processing or long term use. In order to prevent the degradation of polymers, much kind of stabilizers have been developed and are now use widely in many applications. Normally for coloured materials, combination of UV absorbers and light stabilizer are used to achieve a measure of protection and prevent photo-oxidation [7]. The aim of this study was to investigate the effect of UV stabilizers on the photodegradation of sustainable polymer composite. Film samples were exposed in the UV weatherometer and tested for mechanical properties and physical changes.

1.1 Motivation of Research Study

Environmental pollution and destruction on a global scale have drawn attention to the vital need for totally new, safe and clean chemical technologies and processes, the most important challenge facing chemical scientists for the 21st century. A large amount of waste cooking oil has become an environmental issue around the world. The Energy Information Administration in the United States estimated that some 100 million gallons of waste cooking oil is produced per day in USA, where the average per capita waste cooking oil was reported to be 9 lb [8]. In the European countries, the total waste cooking oil production was approximately 700,000–1,000,000 tons/year [9].

Synthetic polymers are produced from petrochemicals that cost very expensive. It also can burden to the environmental because it non-degrade and harming wildlife when they are dispersed in nature. Management of such oils and fats pose a significant challenge because of their disposal problems and possible contamination of the water and land resources As large amounts of waste cooking oils are illegally dumped into rivers and landfills, causing environmental pollution, so the use of waste cooking oil to produce Sustainable Monomer as sustainable polymer substitute offers significant advantages because of the reduction in environmental pollution.

The potential of biodegradable polymers has been recognized for a long time since they could be an interesting way to overcome the limitation of the petrochemical resources in the future. The fossil fuel and gas could be partially replaced by green agricultural resources, which would also participate in the reduction of CO₂ emissions [10]. Due to concerns over sustainability, environmental issues and raw material costs, the use of renewable resources such as waste cooking oil is very attractive to industries. It is because it most valuable to develop as raw materials for sustainable polymer composite and offers great choice with biodegradability, clean, safer and it relatively low cost with acceptable mechanical properties and wide range of application such as in coating field.

Sustainable polymer composites have been developed from natural oils like epoxidized palm oil that has been used as monomer for production of resins. These vegetable oils have their own particular advantages like they are renewable products derived from natural oils and fats and are more readily biodegradable than the corresponding products made from petroleum sources.

Polymers, such as plastics and coatings, are susceptible to photodegradation initiated by heat or UV-light during processing or long-term use. In order to prevent the photodegradation of polymers, different types of stabilizers have been developed and are now used widely in many applications. With the increasing use of polymers in exterior applications, there seems to be a growing need for excellent UV stabilizers such as TiO₂ to protect polymers from photodegradation [11].

Such varnishes, lacquers or paints are usually made of highly crosslinked polymers which must exhibit a great resistance to solar radiation, moisture, pollutants and chemicals in order to ensure a long lasting protection. By lowering the mobility of the polymer chains, the network structure reduces the extent of both the production of initiating species (cage effect) and the propagation step, thus making crosslinked polymers more resistant to photo-oxidation. The durability of organic coatings can be further enhanced by the addition of light stabilizers [12].

The used of metal oxide as stabilizer with sustainable polymer in a coating application such as paint is a major concern in this study as the use of TiO₂ give no harm to environment. The stability of the sustainable polymer composites were tested under UV light using accelerated weathering tester which equivalent to the real outdoor weather condition.

2 Methodology

2.1 Preparation of BP and BPC

Sustainable monomer from virgin vegetable oil (VO) and waste vegetable oil (WO) was used as received from E1, Sustainable Polymer Engineering, Advance Manufacturing and Material Centre (AMMC), Faculty of Mechanical and Manufacturing Engineering (FKMP), Universiti Tun Hussein Onn Malaysia (UTHM). Sustainable monomer of VO and WO was mixed with Methylene diphenyl diisocyanate (MDI) and solvent to form virgin oil sustainable polymer (VOP) and waste oil sustainable polymer (WOP). The mixture was stirred using mechanical stirrer at optimum condition for about 60 min at 50 °C resulting viscous compound. The compound was then cast into a container spread to uniform thickness. The VOP and WOP was leave to cure at ambient temperature for at least 6 h, as to ensure complete removal of solvent traces. Once dry, the film was stripped from the plate. Micrometer was used to measure the thickness of the sample at particular point ranging from 110 μm to 250 μm accordance to ASTM D1005-95 [13].

The above steps were repeated to fabricate sustainable polymer composite (SPC) doped with different percentage of TiO₂. The samples of BP are based on VOP and WOP while BPC was namely according to TiO₂ percentage as VOP2.5, VOP5, VOP7.5, and VOP10; WOP2.5, WOP5, WOP7.5, and WOP10 respectively.

2.2 Preparation of BP and BPC Thin Film for Ultra Violet Irradiation Exposure, Tensile Test and Carbonyl Index

VeSawit Palm oil was used as the original palm oil to produce the origin biopolymer (B). The conversion of the monomer starts with the catalyst preparation to generate the

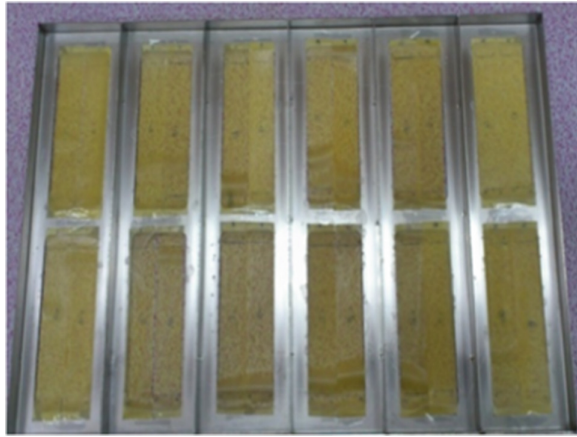


Fig. 1. Biopolymer thin film samples on a rack for UV light exposure.

epoxies from the unsaturated fatty compound, and the second reaction is the opening of the acid catalyst ring of the epoxies to form polyols or bio monomer [11–18] [19–21]. Then, bio-monomer was mixed with cross linking agent known as 4, 4'-Methylene Diphenyl Diisocyanate (MDI). The mixture was stirred together at 50 °C for 30 min and the resulting viscous compound was poured into the mould designed according to the BS EN ISO 527-2: 1996 and left until tack-free and clamped using Heat Transfers Machine. The sample was left at room temperature for 24 h to cure and produced biopolymer notated as B.

UV Accelerated Weatherometer Haida International Equipment Ltd. was used to irradiate sample in UV light. The UV accelerated weathering test was conducted according to ASTM D 4587-Standard practice for fluorescent UV-condensation exposures of paint and related coatings [14]. The UV Weatherometer used UV irradiation was carried out using an array of UV fluorescent lamps emitting light in the region from 280 to 320 nm with a tail extending to 400 nm.

The BP and BPC thin film samples were placed on a rack with a rack holder as shown in Fig. 1 in the UV weatherometer chamber at 50 °C with different exposure time. The samples were analyzed after 250, 500, 750, 1000, 2000 and 3000 h of UV exposure to examine the changes in mechanical and physical properties.

Tensile test was conducted to determine the strength and ductility of a material. Tensile strength, elongation at break and strain at break were measured by Universal Testing Machine (LLOYD Instruments LR30K), with load range; 20N, cross head speed; 500 mm min⁻¹, gauge length; 100 mm and efficiency within ±1%. All specimens were tested at room temperature. A minimum of five samples was analyzed in order to obtain average result. Original length and area of the gauge sample was measured using vernier calipers. The sample was mounted between two jaws of the tester.

Stress tests for each material were according to ASTM D882 [15] which is the standard test method for tensile properties of thin plastic sheeting are very similar to the common ASTM D638. The plastic material is pulled until it breaks in order to measure elongation, tensile modulus, tensile yield strength, and tensile strength at break. However,

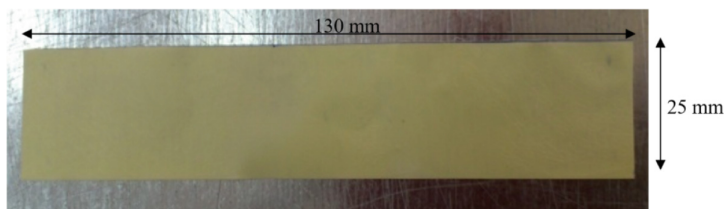


Fig. 2. Biopolymer as thin film sample for tensile test.

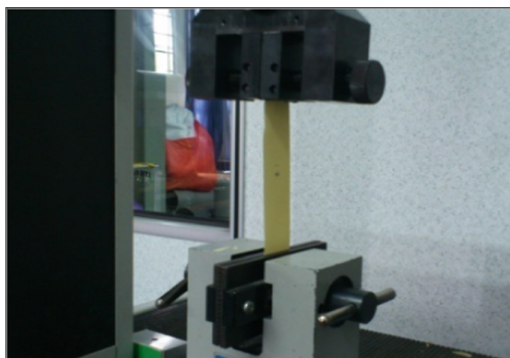


Fig. 3. The sample is mounted between two jaws of the tester and in line with the direction of pull.

it is designed specifically for thin sheeting and film less than 1 mm (0.04") thick where the BP and BPC films samples thickness were 0.11–0.25 mm.

The ASTM D882 test specimens are rectangular strips of and are not “dumb bell” or “dog bone” shaped as shows in Fig. 2. Therefore, it does not need to use an extensometer to measure the elongation as the system built in crosshead displacement indicator is sufficient. The required information to do the tensile test are specimen dimension (mm x mm), gauge length (mm), cross-head speed (mm/min), specimen thickness, load cell (N) and instrument name and model.

Care must be taken to ensure that the film is in line with the direction of pull as shown in Fig. 3. The average tensile strength for the films was calculated and the results were compared with unexposed sustainable polymer thin films samples.

Meanwhile, the carbonyl index (CI) is calculated according the ratio of area of peak centered at carbonyl (C = O) region from 1800–1680 cm^{-1} and area of the peak centered at alkene (CH) group of 2930–2850 cm^{-1} (which is the characteristic vibration of stretching band of CH). The reference band is chosen as out of plane bending of –CH at 2722 cm^{-1} ($\text{CI} = \text{A}_{1730}/\text{A}_{2722}$). The CI versus time data was plotted for different temperatures to follow the variation of degradation. One can notice the presence of NH group around 3400 cm^{-1} , carbonyls in the region of 1800 cm^{-1} a alkene (CH) group close to 2930–2860 cm^{-1} . At least two replicates were tested for each FTIR spectrum and the average values of the CI. The values of CI were used of compare the thermal-activity of different polymer thin films [16].

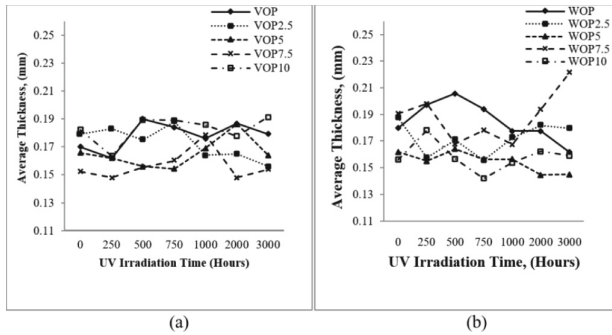


Fig. 4. Average thickness for BP and BPC of (a) VOP and (b) WOP.

3 Results and Analysis

3.1 Mechanical Properties of BP and BPC Upon UV Irradiation

The BP and BPC with average thickness of 110–250 μm with standard deviation ± 0.02 using micrometer according to ASTM D1005-95. The average thickness, t_{avg} for VOP and WOP is 0.148 to 0.91 mm and 0.44 to 0.221 mm respectively. Figure 4, shows the plotted graph of average thickness of BP and BPC in the thickness range of thin film between 110–250 μm .

Throughout the thickness monitoring of BP and BPC for VOP and WOP, the plotted average thickness is still in the range of 110–250 mm after UV irradiation up to 3000 h. This is therefore, the result of the mechanical test such as tensile stress and elongation at break is acceptable. The study by Salem (2001) only states that the thickness of commercial LDPE films that produced by using co-extrusion machine of approximately 50 μm .

After BP and BPC thin film were exposed to UV light for several hours, the samples were mechanically test using UTM tensile test according to ASTM D882. Five samples were tested to get the average value of each UV irradiated BP and BPC at 250, 500, 750, 1000, 2000 and 3000 h. The mechanical properties of BP and BPC were study according to the tensile strengths and maximum elongation. The average value of tensile strength, Young modulus and elongation of exposed samples to UV light at 250, 500, 750, 1000, 2000, and 3000 h.

Figures 5 and 6 show the tensile strength as a function of UV irradiation time for BP and BPC of VOP and WOP respectively. BP of VOP shows rapid loss in tensile strength from 250 h until 3000 h about 20%. It is due to the UV degradation, which causes a breakdown of chemical bonds, resulting in embitterment and also increased cross linking and chain scission, which lead to shrinking and crack. But the addition of TiO_2 into the BP can improve the mechanical performance. This result is in good agreement with result obtained by Salem (2001) [16]; Saleh and Shnean (2009) [17].

The tensile strength from 0 h to 250 h UV irradiated BP and BPC of VOP and WOP is increased. From 250 to 500 h UV irradiation, the tensile strength for BP and BPC of VOP and WOP were decreases. Meanwhile tensile strength of BP and BPC of VOP and WOP increases from 500 to 750 h of UV irradiation except for VOP and VOP5. For

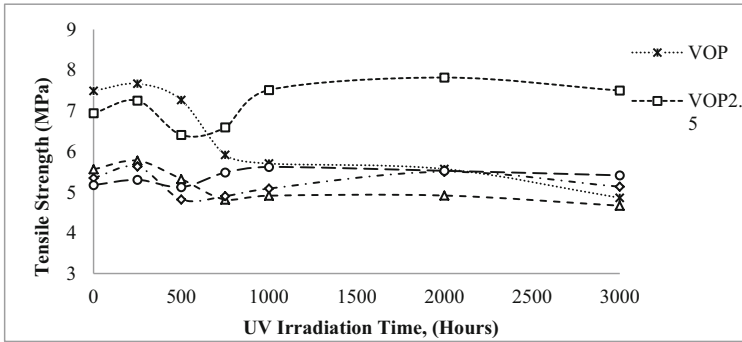


Fig. 5. Tensile strength against UV irradiation time for BP and BPC of VOP.

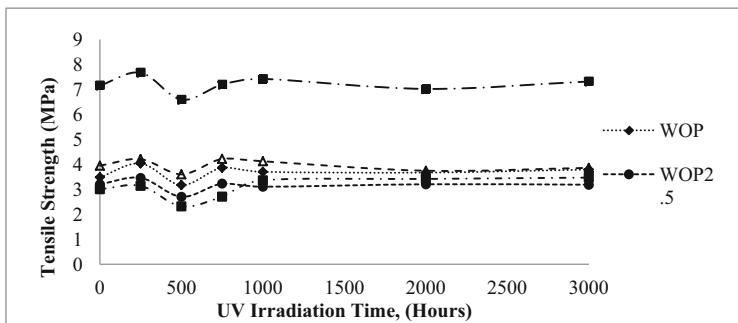


Fig. 6. Tensile strength against UV irradiation time of BP and BPC for WOP.

VOP, the tensile strength was decreases from 500 h until 3000 h of UV irradiation. The trend remains increasing in systematic way after 1000 h until 2000 h for BPC of VOP. The tensile strength slightly decreases from 2000 h to 3000 h for BP and BPC of VOP and WOP.

According to the mechanism of photo degradation, the increment from 0 h to 250 h UV irradiation shows radical initiation of the soft segment. At 250 h UV irradiation, the value of tensile strength of BP and BPC of VOP and WOP was higher than unexposed (0 h) due to the crosslinking of the soft segment radical. It can be prove by FTIR spectra at 1202 cm^{-1} becomes more intense after UV irradiation for 250 h for BPC which can be assigned to crosslinking of the soft segment.

Meanwhile, the decrement of the tensile strength at 500 h UV irradiation due to the propagation and increment of tensile strength at 750 h due to the crosslinking of the soft segment radical at the initial stage in both samples of VOP and WOP thin films. It can be prove by increasing in the quantity of hydroxyl groups that can be observed at 3340 cm^{-1} , which is also broadened by the NH groups. Tensile strength of BP and BPC thin films are significantly stable at more than 750 h to 3000 h UV exposure. The chain scission of the soft segment radical happened beyond 3000 h of UV irradiation shows the decreased of intensity at 1700 cm^{-1} peaks were due to the oxidation reaction of C = O in (NH-CO-O) bond.

From this study, the results for BP and BPC of WOP show tensilized films of WOP10 tend to have higher tensile strength of 7.67 MPa at 250 h UV irradiation. This is the maximum tensile strength before failure which indicated from chain scission. WOP_{7.5} has lowest tensile strength at 500 h with 2.36 MPa. Meanwhile, results for BP and BPC of VOP show tensilized films of VOP_{2.5} tend to have higher tensile strength at 2000 h UV irradiation while 3000 h for sample VOP₅ has lowest tensile strength of 7.82 MPa and 4.67 MPa respectively.

The addition of TiO₂ to BP of VOP and WOP reduce the tensile strength as compared to pure BP is due to agglomeration between BP and TiO₂, so that it could be the starting point for stress concentration and initiation of cracks, and this will reduce the strength and reflect on the strain of the samples. A further drop in tensile strength was noticed when the samples were radiated with UV irradiation.

Mechanical properties of BP and BPC can be strongly influenced by photo-oxidation upon UV weathering exposure. The ductility disappears and the strength and strain at rupture strongly decrease at 500 h and stable until 3000 h UV exposure. The tensile strength of VOP and WOP shows the significant change of UV irradiation. From the data, BP and BPC of VOP give better tensile strength as compared to BP and BPC of WOP.

Figure 7 and Fig. 8 show elongation at break against UV irradiation time for BP and BPC of VOP and WOP respectively. For both BP and BPC, the higher elongations at break were at 250 h UV irradiation. This is similar as tensile strength where at 250 is the higher tensile value. While at 500 h UV irradiation, the elongations at break were decreased. This is due to the mechanical properties of BP and BPC of VOP and WOP where the tensile strength decreased, the elongation at break also decreased. The highest percentage elongation at break for unexposed and UV exposure for VOP is VOP_{7.5} which is 21% and 26% respectively. Meanwhile the highest percentage elongations at break for unexposed and UV exposure for WOP is WOP_{2.5} which is 64% and 69% respectively.

This study is similar with [5], where they found that both properties, tensile strength and maximum elongation showed a significant decrease with UV irradiation time, a typical behaviour of a polymer that undergo chain scission reactions during weathering. Comparing the various types of samples, it is clear that those containing the UV stabilizer are more stable, confirming the protecting action of this additive in the amorphous region for BP and BPC of VOP and WOP.

A more reduction in mechanical properties reflects by the unhomogeneity of TiO₂ fillers in BP of VOP and WOP which will effect on the mechanical properties. The reductions in tensile strengths and elongation at break are attributed to the distribution of particles TiO₂ between the chain of BP, since these particles gives more spacing between the fold chains, and the stretch mobility of main chain will be effected. This will reduce the interaction of the applied stress with these specimens, when these chains where stressed, the aggregation of some of these particles in local area will decrease the strength of polymer, because these particles will acts as a weakness points. These weakness points will accumulate a stress around these areas and the crack will propagate causes a failure in the polymers [6].

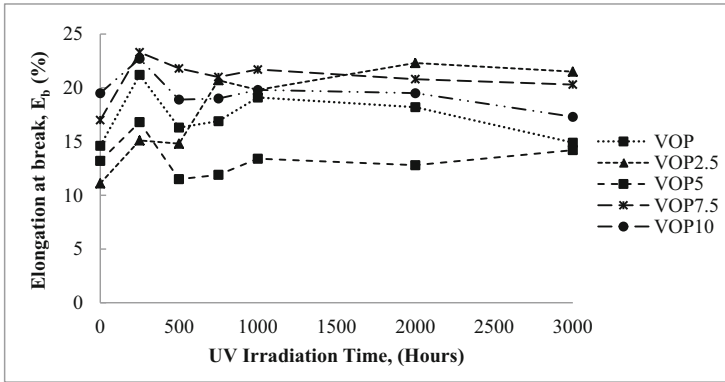


Fig. 7. Graph of elongation at break (%) against UV irradiation time (Hours) for BP and BPC of VOP.

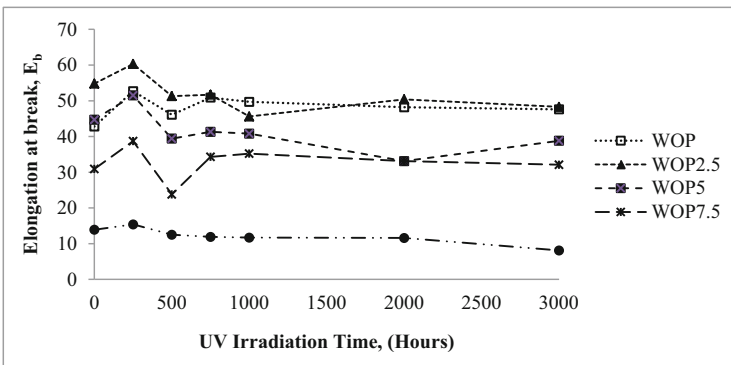


Fig. 8. Graph of elongation at break (%) against UV irradiation time (Hours) for BP and BPC of WOP.

3.2 Carbonyl Index (CI) of BP and BPC Upon UV Irradiation

The CI values for UV irradiated biopolymer were calculated as the ratio of area of peak centered at carbonyl (C = O) region and area of the peak centered at alkene (CH) group (CI = A1702/A2916).

The tensile strength and CI values for BP and BPC of VOP and WOP as refer in the Fig. 9 (a), (b), (c), (d), and (e) and Fig. 10 (a), (b), (c), (d), and (e) show that the increased in the carbonyl index will decreased the mechanical strengths. The increasing of CI of BP and BPC of VOP and WOP thin films after UV irradiation is the indication that oxidation occurred during the exposure. The growth of CI relates to the level of oxygen which has been incorporated into the polymer and wherever a carbonyl group is introduced the polymer undergoes a chain scission such as molecular weight reduction and a change in physical and chemical properties. The increased in CI formation for polymer after UV irradiation is proportional to the number of chain scissions that occur in the polymer thin films associated with the scission of the (-NHCO-O-).

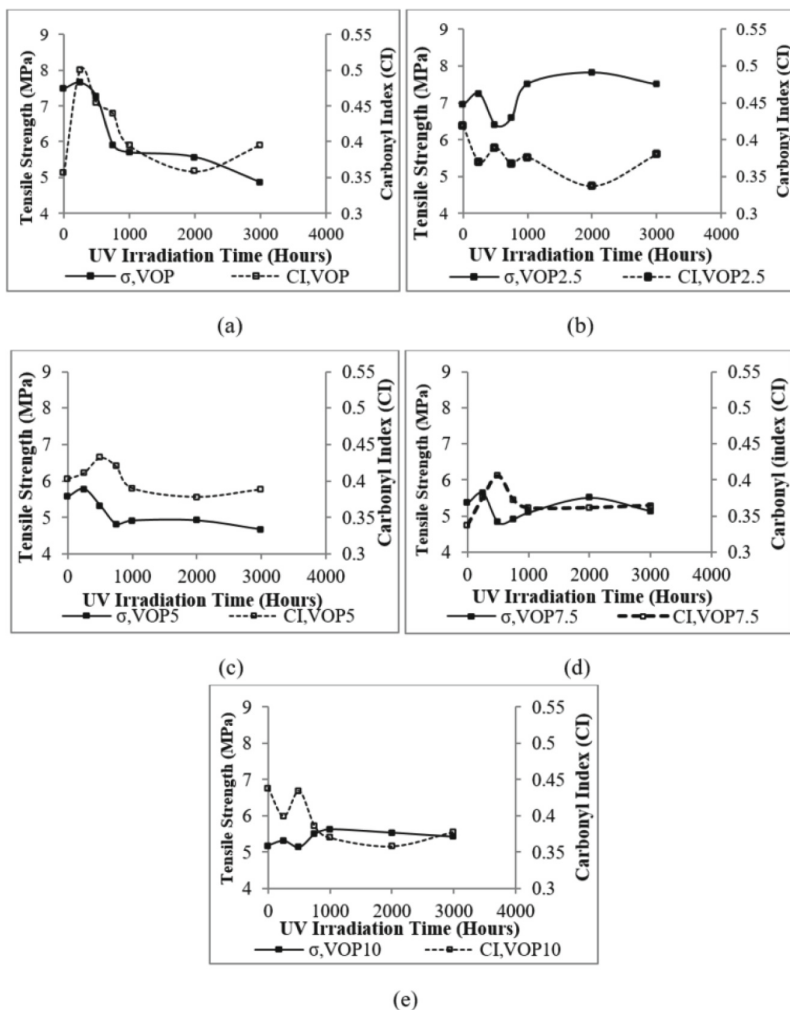


Fig. 9. Carbonyl index (CI) for non-UV irradiated and UV irradiated BP and BPC of (a) VOP (b) VOP_{2.5} (c) VOP₅ (d) VOP_{7.5} and (e) VOP₁₀ at 250, 500, 750, 1000, 2000, and 3000 h.

The increase in the absorption intensity of these bands indicated that the formed products were resulted from an extensive chain scission due to UV exposure, followed by an oxidation of formed fragments due to the presence of oxygen during irradiation of peak positions are changed, which indicates that the photo-degraded products are not the same during the degradation process. It has been reported that the rate of photooxidative degradation of poly (Florstyrene) [18] caused by polychromic irradiation ($\lambda \geq 290$ nm) at 55 °C in presence of oxygen is reduced in copolymers containing aromatic systems. In the same way, introducing aromatic rings into aliphatic compounds increases their stability. It was assumed that the aromatic moiety, which is very radiation resistant, accommodates the extra energy remaining unaffected.

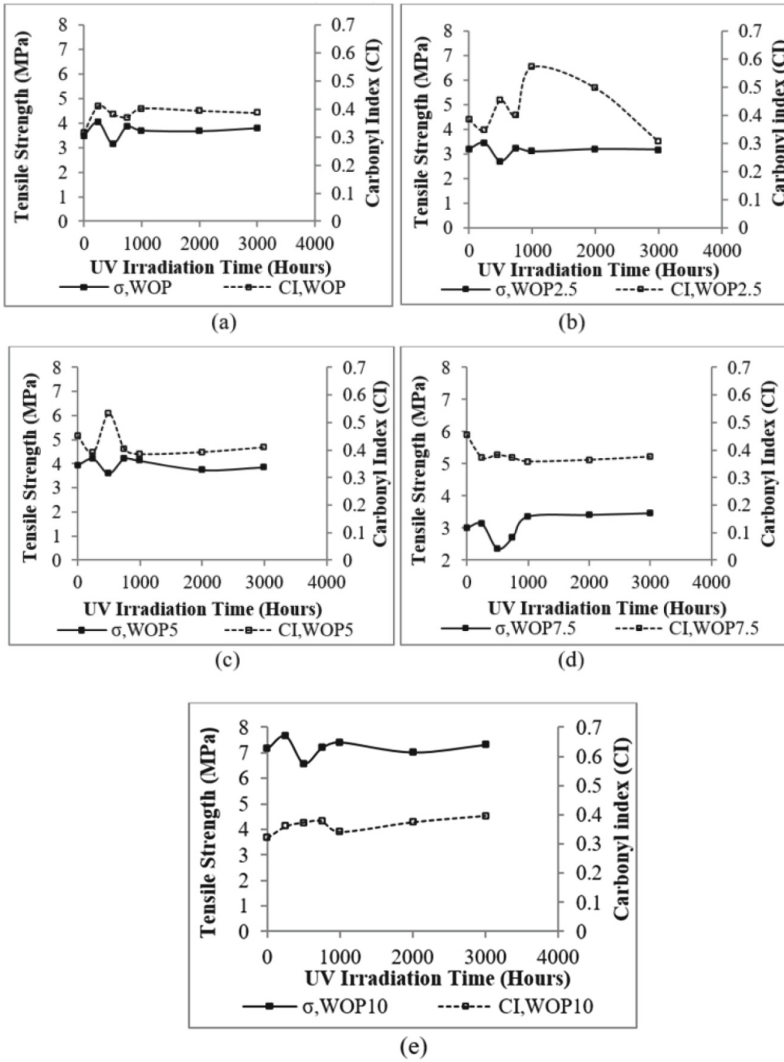


Fig. 10. Carbonyl index (CI) for non-UV irradiated and UV irradiated BP and BPC of (a) WOP (b) WOP_{2.5} (c) WOP₅ (d) WOP_{7.5} and (e) WOP₁₀ at 250, 500, 750, 1000, 2000, and 3000 h.

The carbonyl index (CI) of BP of VOP and WOP_{2.5} revealed higher values at 250 h and 1000 h which is 0.5 and 0.57, respectively. The CI value decreases with the increased of UV irradiation. The lowest CI is WOP_{2.5} and WOP_{2.5} at 2000 h and 3000 h, which both of sample is the same of approximately is 0.3.

4 Conclusion

The calculated Carbonyl Index (CI) was indicated that the above mechanism is true. Meanwhile, the calculated Hydroxyl Index (HI) also shown that BPC were evidently decreased its HI of UV irradiated thin films with more crosslinking of CH₂ which attributed to stabilization of the thin films chain radical. The mechanical properties for tensile test of polymer thin films upon UV irradiation for photo degradation were significantly reduce its reduction strength. As compared to WOP, VOP gives better result for tensile strength and elongation at break. The results obtained support the photo degradation mechanism of BP and BPC after UV irradiation proposed in the literature for aromatic polymer from renewable resources, involving the scission of the (-NHCO-O) group and oxidation of the central methylene group with quinone (yellow) formation as a chromophoric reaction product. The highest percentage filler loading in the BPC give small changes in colour which is suitable and important in coating application sectors. The yellowing decreases with the increasing percentage ratio of TiO₂ loading on VOP and WOP thin films. For future study on the stability of polymer composites properties, the bio polymer thin film doped with TiO₂ irradiated upon UV light to enhance the degradability and stability of polymer composites applied in coating industry.

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