EVALUATION OF HIGH DENSITY POLYETHYLENE COMPOSITE FILLED WITH BAGASSE AFTER ACCELERATED WEATHERING FOLLOWED BY BIODEGRADATION

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Wood-plastic composites (WPC) have many applications as structural and non-structural material. As their outdoor application becomes more widespread, their resistance against weathering, particularly ultraviolet light and biodegradation becomes of more concern. In the present study, natural fiber composites (NFPC) made of bagasse and high density polyethylene, with and without pigments, were prepared by extrusion and subjected to accelerated weathering for 1440 h; then weathered and un-weathered samples were exposed to fungal and termite resistance tests. The chemical and surface qualities of samples were studied by ATR-FTIR spectroscopy, colorimetry, contact angle, and roughness tests before and after weathering. Using bagasse as filler does reduce the discoloration of weathered samples. Adding pigments may reduce the effect of weathering on lignin degradation, although it favors polymer oxidation, but it increases the weight loss caused by fungi. Despite the high resistance of samples against biological attack, weathering triggers attack by termites and fungi on the surface and causes surface quality loss.

Keywords: Wood plastic composite; Bagasse; Pigment; Photodegradation; Discoloration; FTIR spectroscopy; Biodegradation

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INTRODUCTION

Wood-plastic composites (WPCs) are gaining interest as environmentally friendly materials, since renewable and biodegradable lignocellulose fibers are used as reinforcing filler (Bledzki and Gassan 1999; Eichhorn *et al.* 2001). Many non-wooden cellulose-based fibers, like those obtained from residual annual crops such as sugar cane bagasse, one of the most abundant agro fiber resources, offer advantages as additives in plastics: low density, low cost, non-abrasive nature to the processing equipment, possibility of high filling levels, availability of wide variety of fibers throughout the world, and generation of agricultural economy (Sanadi *et al.* 1994). Moreover, in case of exposure to light, compared to wooden fibers they contain less lignin, which is most susceptible to photo-degradation.

The use of biodegradable polymers for the production of these natural fiber plastic composites (NFPCs) can even lead to totally recyclable materials. However, for structural and non-structural purposes this might cause problems in service life. Along with all the advantages of NFPCs, they also experience color change after exposure to the outdoor environment, and they can also suffer mechanical and biological degradation (Morrell *et al.* 2006, Manning and Ascherl 2007). WPCs lighten in color and a white chalky layer appears on their surface after weathering (Stark 2006). Chemical reactions and changes in wood and wood plastic composites happen during these phenomena (Stark and Matuana 2004, Fabiyi *et al.* 2006). This is not the only effect of outdoor applications. WPCs, which were thought as impervious to biological attack, show signs of fungal growth during their service life and remain clearly susceptible to biodegradation (Schauwecker *et al.* 2006) with a dependency on many factors such as the fiber species (Fabiyi *et al.* 2009). Laboratory observations show that various decay fungi cause substantial weight loss of NFPCs under certain conditions (Wan *et al.* 2009; Bras *et al.* 2010). This biodegradation can be made more severe by other factors, such as weathering, which will be the focus of this study.

So far, a number of research articles on sugarcane bagasse-based composites as value-added products have been published worldwide, showing potentiality and feasibility of using bagasse fiber in composite products (Collier *et al.* 1997). Bagasse can be introduced in PE as a binding material to improve the mechanical properties and dimensional stability of the product (Raj and Kokta 1991). The research conducted on composites made from bagasse enables the selection and best combination of bagasse origin, size, and molding pressure (Sousa *et al.* 2004). However, the photostability of such products has not been studied yet; as bagasse contains less lignin than wood fibers, it should exhibit less photodegradation. Besides, using various pigmentations in WPCs reduces lightening caused by weathering (Du *et al.* 2010, Zhang *et al.* 2010), although pigment use usually triggers the oxidation of polymers. In this study we will try to clarify the combined effect of weathering and biological attack on several chemical and physical properties, including color, of bagasse/HDPE composites, with a special focus on the consequences of pigment addition to the flour.

EXPERIMENTAL

Materials

Sugar cane (*Saccharum officinarum L.*) bagasse was collected at a wood plastic factory DEZCHOOB in Iran in powder shape (20 mesh). High density polyethylene (HDPE) with density 0.951 g/cm^3 and melt flew index (180/6) 0.45 ± 0.05 g/ 10 mm (ASTM 1238) was supplied from Iranian Petro-chime Co (WE6040). Bagasse powder was oven-dried at 100 ± 3 ºC for 24 h before processing. Master batch brown pigments (Grade 7110, Ilam Fara Shimi Co.) were used. Table 1 summarizes the formulations used. The biodegradability in PE films only has a pronounced effect if it contains 30% or more of cellulose-based fibers (Behjat *et al.* 2009). Therefore, we used 50% w/w fibers to ensure a visible contribution of the fiber addition to the durability of the product. The mixtures were rotated using a Valtorta-Brescia pre-mixer device with 300 rpm and 115 °C for 5 min before processing to remove moisture. After pre-mixing, the samples were passed through a conical twin screw extruder (Conical screw 2 m, output capacity 80 to 100 kg/hr, QC Future Plastic Machinery CO., LTD) with an I-shaped die. Extrusion temperature was kept under 180 °C to avoid thermal degradation of the wood flour. Samples were immediately cooled in a water pool. After air drying, all samples were sanded to remove the polymer-rich surface. This action, which results in more fiber exposed to the surface, is performed by most factories, adding sanding plates with different patterns to their line, to provide a wood-like character to their product.

Table 1. Combinations of Materials Used in the Manufacture of Bagasse/Plastic Composite Boards Based on Total Dry Weight of Mixture (w/w)

Samples Code	Bagasse fiber	HDPE	Maleic anhydride polypropylene (MAPP) (POLYBOND W30)	Stearic acid (lubricant)	Granule Pigment
A0	49%	49%	2%	1%	0%
Ap	47.5%	47.5%	2%	1%	3%

Methods

Accelerated artificial UV weathering tests were conducted in a QUV weather-ometer according to EN4892-2, as recommended in test methods for characterization of WPCs (CEN/TS 15534-1), for 1440 hrs in Critt Bois Epinal, France. The cycle was closed and followed a vaporization cycle of 18 min for water spray, 102 min exposure to sec interval between two sprays, and relative humidity of 65 ± 5 %. Temperature of the Black Stallion was 60 ± 3 ° C. All properties of the composites were measured/observed before and after weathering.

Contact angle

Contact angle (θ) tests were conducted with a Thermo Cahn DCA (Dynamic Contact Angle, Model No: DCA 300) as a quantitative measure of the wetting of samples' surfaces; it was measured by fitting a mathematical expression to the shape of the drop and by calculating the slope of the tangent to the drop at the liquid-solid-vapor (LSV) interface line (according to ASTM D7334).

Roughness

Roughness represents the quality of the surface and the degree of fiber removal from the surface. It was determined by measuring the height, width, and shape of the peaks and valleys produced. The three parameters of surface roughness, average roughness (R_a), root means square roughness (R_q), and average distance between highest peak and lowest valley (R_z) were measured with a Surftest SJ-201 Series 178-Portable Surface Roughness Tester (Mitutoyo).

Color change

Color change was measured using a Datacolor spectrocolorimeter based on the CIELab color system. It measures the lightness of the sample (L^*) and color coordinates: redness (a^*) in the green-red axis and yellowness (b^*) in the blue-yellow axis. Color indexes were measured on more than 20 replicates for each group, following different stages of the experience: right after samples were extruded and sanded, after QUV exposure, and after fungal resistance test. Color change is expressed through differences of color indexes,

$$\delta L = L^* - L^*_i \quad ; \quad \delta a = a^* - a^*_i \quad ; \quad \delta b = b^* - b^*_i \qquad (1)$$

where *i* indicates the initial value (before weathering and fungal attack). From these algebraic increments, the so-called “total color change” ΔE, a positive quantity corresponding to a distance in the (L^*, a^*, b^*) space, can be computed:

$$\Delta E = \sqrt{\delta L^2 + \delta a^2 + \delta b^2} \quad (2)$$

Chemical analysis

For chemical analysis a Vertex 80 Spectrum with attenuated total reflectance (ATR) acquired was used. The Fourier transform infrared spectroscopy (FTIR) scans were obtained at a resolution of 4 cm^{-1} from 4000 to 600 cm^{-1}. For quantifying data, the net peak absorbance was obtained after baseline correction. The peak intensities of absorption bands (I) at 1030, 1715, and 2915 cm^{-1} were used to calculate both wood index WI= (I_{1030}/I_{2918}) x 100 and carbonyl index CI = (I_{1715}/I_{2918}) x 100 (Stark and Matuana 2004). Peak intensity was normalized using the peak at 2912 cm^{-1}, which corresponds to alkane C-H stretching vibrations of methylene groups ($-CH_2$). Hydroxyl index (HI) was calculated as the ratio of the area (absorbance) of the band at 3500 to 3080 cm^{-1} and the antisymmetric C–H stretching band of methylene groups (2917 to 1912 cm^{-1}). In addition, lignin index (LI) was taken as the ratio of the area (absorbance) of the band at 1512 to 1508 cm^{-1} and the antisymmetric C–H stretching band of methylene groups (2917 to 1912 cm^{-1}) (Gupta 2006).

Biodegradability

Biodegradation toward Basidiomycete fungi was evaluated using an agar-plate test method. Samples of dimensions 25*12*9 mm were dried at 103 °C, weighed, sterilized, and exposed to cubic decay fungi (*Coniophora puteana*, strain BAM Ebw. 15). For control, 30*10*5 mm (L,R,T) pine sapwood (*Pinus sylvestris*) samples were used. One control and two samples were placed in each of the malt-agar plates (40 g malt/L, 20 g agar/L). After 12 weeks at 22°C, 65% RH, samples were removed, cleaned, and dried at 103 °C. The surface was qualitatively assessed, and the weight loss (WL), moisture content at the end of fungal test (MC), as well as color (L^*, a^*, b^*) were measured.

Termite resistance

Resistance towards termites (*Reticulitermes santonensis*) was also evaluated according to the guidelines of EN117. Samples (25*12*9 mm) were exposed to 250 termite workers for 8 weeks (27 °C, 70% RH). Pine sapwood samples (50*25*15 mm, L,R,T) were used as controls. At the end of the test, the survival rate of termites, the weight loss of the samples, and their moisture content at the end of the tests were calculated

RESULTS AND DISCUSSION

Effect of Weathering on Chemical Properties

Figure 1 shows the FTIR spectra obtained from weathered and un-weathered samples. The peaks related to fibers tended to vanish as a result of their degradation by weathering. This result was confirmed by the decrease of WI, LI, and HI. Compared to non-pigmented samples (controls), WI and HI decreased more in pigmented samples (Fig. 2), while LI and CI decreased to a lesser extent. This can be due to the higher removal of fibers from the surface because of the weaker bonds in pigmented samples or degradation of pigment itself. However, in this batch, a lower decrease in LI through weathering suggests less lignin degradation, which is confirmed by a peak at 1512 cm^{-1} (Fig. 1). Change in crystallinity of HDPE before and after weathering can be obtained from studying the doublet peaks at 730 cm^{-1} and 720 cm^{-1} (Stark and Matuana 2004; Muasher and Sain 2006). The peaks at 1474 cm^{-1} and 730 cm^{-1} correspond to the polyethylene crystalline content, and the peaks at 1464 cm^{-1} and 720 cm^{-1} represent the amorphous content. Figure 1 shows that the two peaks corresponding to the polymer crystalline content increase markedly after weathering, for pigmented samples only. These two sets of observation suggest that the presence of pigment possibly triggers the oxidation and chain scission of the polyethylene, causing production of shorter chains that then recrystallize, leading to a more brittle material as often mentioned for WPC weathering (Klyosov 2007).

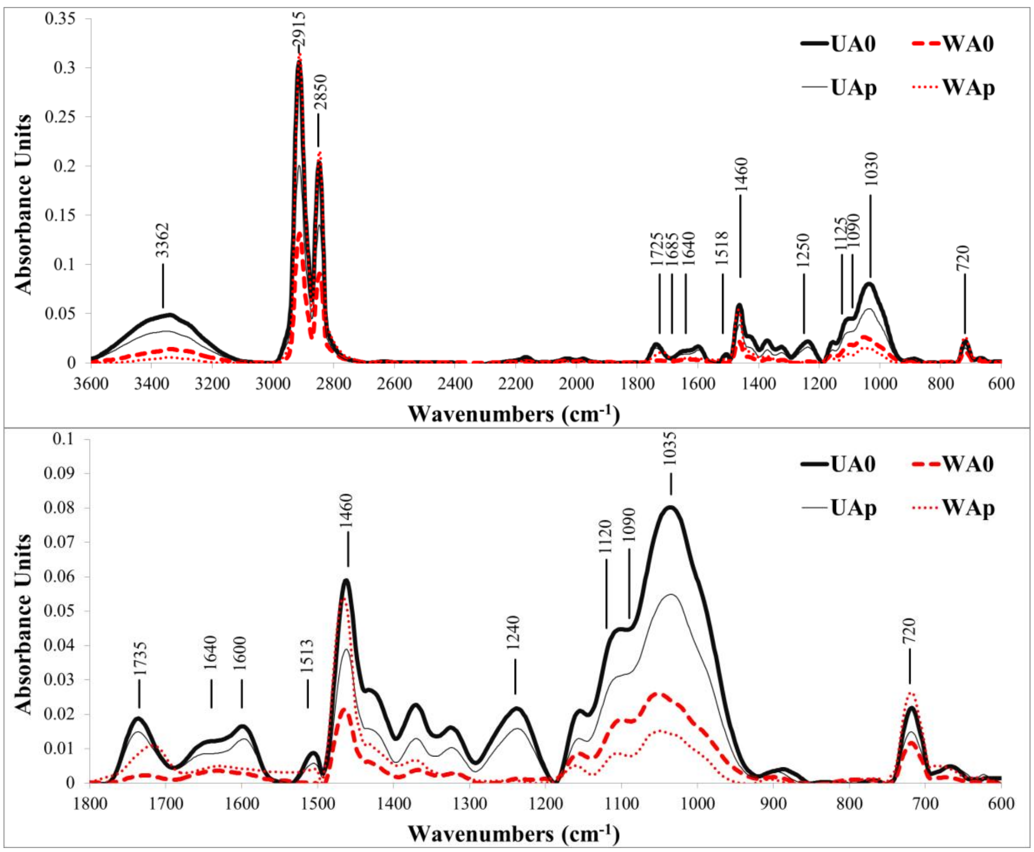

Fig. 1. ATR-FTIR spectra of control (A0) and pigmented sample (Ap) before (UA0. UAp) and after weathering (WA0. WAp)

Carbonyl groups are a good indicator of the oxidation, because they increase drastically with the oxidation in the surface upon xenon-arc and UVA weathering of HDPE and PP-based WPC (Stark and Matuana 2007; Fabiyi *et al.* 2008). In Table 2, which shows the values of all parameters measured, CI decreased for both control and pigmented samples, although an increase would have been expected as the result of the oxidation. This decrease, which already had been observed after long-time exposure by

Fabiyi (2007), could be explained by the fibers being washed away from the surface. The comparatively lower CI decrease of Ap compared to A0 could have indicated a higher amount of carbonyl groups in the surface due to oxidation. On the other hand, after weathering the removal of fibers, the decrease in WI and HI was higher in the case of Ap. Although more fibers were removed, more carbonyl groups still have been produced and remained on the surface of Ap, which can be explained by higher polyethylene oxidation. Nevertheless, the degradation of lignin was less in Ap, because of the presence of the pigment.

Table 2. Results of Different Tests

Characteristics			nb	A0		Ap	
				U	W	U	W
Chemical properties	wood index	WI	1	26.20	18.31	27.37	4.57
	lignin index	LI	1	0.00265	0.00007	0.00258	0.00126
	hydroxyl index	HI	1	0.701	0.406	0.705	0.053
	carbonyl index	CI	1	3.55	1.44	4.57	3.47
Wettability	contact angle (°)	θ	3	$87.6^{\pm1.34}$	$62.5^{\pm2.65}$	$77.0^{\pm0.94}$	$90.2^{\pm1.06}$
Roughness (mm)	average	Ra	3	$3.99^{\pm0.34}$	$4.88^{\pm0.56}$	$4.09^{\pm0.09}$	$4.58^{\pm0.21}$
	root mean square	Rq	3	$5.86^{\pm0.93}$	$6.11^{\pm0.75}$	$5.24^{\pm0.53}$	$6.26^{\pm0.35}$
	Dist. Between peaks	Rz	3	$37.6^{\pm1.02}$	$31.2^{\pm0.84}$	$28.2^{\pm0.46}$	$33.9^{\pm093}$
Color	lightness	L*	20	$55.56^{\pm1.02}$	$68.79^{\pm0.84}$	$48.79^{\pm1.40}$	$57.56^{\pm0.85}$
	'redness'	a*	20	$5.98^{\pm0.29}$	$3.08^{\pm0.31}$	$5.74^{\pm0.92}$	$2.07^{\pm0.10}$
	'yellowness'	b*	20	$10.32^{\pm0.57}$	$5.55^{\pm0.48}$	$5.60^{\pm0.91}$	$2.60^{\pm0.26}$
Color after fungal resistance test	lightness	L*	12	$46.68^{\pm3.59}$	$52.29^{\pm4.24}$	$42.07^{\pm2.65}$	$49.90^{\pm3.77}$
	'redness'	a*	12	$5.29^{\pm1.27}$	$4.93^{\pm0.45}$	$5.57^{\pm0.71}$	$3.57^{\pm0.44}$
	'yellowness'	b*	12	$14.75^{\pm3.42}$	$15.41^{\pm1.54}$	$10.96^{\pm1.75}$	$12.67^{\pm2.03}$
Fungal resistance	weight loss (%)	WL	12	$0.19^{\pm0.16}$	$0.26^{\pm0.10}$	$0.63^{\pm0.30}$	$0.64^{\pm0.09}$
	m.c. (%)	MC	12	$7.38^{\pm1.21}$	$7.37^{\pm1.62}$	$9.14^{\pm1.77}$	$10.81^{\pm0.45}$
Termite's resistance	weight loss (%)	WL	3	$0.00^{\pm0.00}$	$0.45^{\pm0.34}$	$0.00^{\pm0.00}$	$0.40^{\pm0.07}$
	m.c. (%)	MC	3	$1.52^{\pm0.15}$	$1.50^{\pm0.17}$	$1.40^{\pm0.17}$	$1.59^{\pm0.15}$

A0 = control; Ap = pigmented; U = un-weathered; W = weathered; (±) = standard deviation
nb: number of repetitions; m.c.: moisture absorbed by samples at the end of test

Effect of Weathering on Color

Figure 2 shows results for control and pigmented samples the variations induced by weathering (W), fungal attack on un-weathered samples (UF), and fungal attack on weathered samples (WF). After weathering (W), the surface became lighter (δL>0), less red (δa<0), and less yellow (δb<0). However, for pigmented samples the variations of L^* and b^* were less pronounced and, as a result, ΔE was lower. The fungal attack both on un-weathered (UF) and weathered samples (WF) provoked the appearance of dark spots and a yellowish color that resulted in the observed L^* decrease and b^* increase. For weathered samples (WF), the darkening was more pronounced and accompanied by an increase of the red component. This difference can be explained by the removal of the chalky white surface present on the weathered specimens, which was easily removed by fungi. A similar effect was observed after termite attack, except that termites had no effect on un-weathered samples. Even without any treatment, simply using bagasse as filler without any additives resulted in a low total color change (ΔE = 14.5). In previous studies on WPC, with similar processing conditions and weathering procedure, such ΔE levels were not attained without using photostabilizer and many additives (Fabiyi *et al.* 2008).

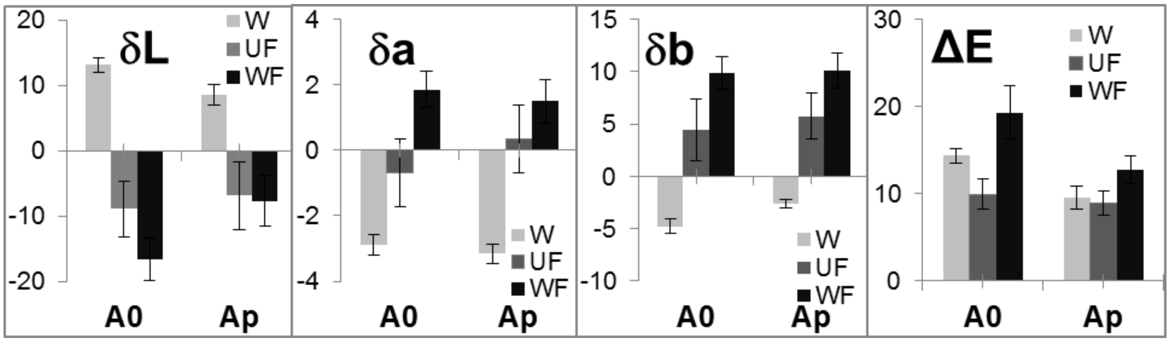

Fig. 2. Color changes induced by weathering and/or fungal attack. U: un-weathered; W: weathered; F: after fugal attack

Effect of Weathering on the Visual Appearance of the Surface

Figure 3 shows the state of the surface of the control samples at the different stages of degradation. Weathering alone induced small cracks, possibly due to the swelling and shrinkage of the fibers and weakening of the bonds between fibers and plastic. Fungi removed the fibers (mainly cellulose) accessible on the surface, even for un-weathered samples. However, on the samples exposed to termites, there were no sign of fiber feeding on the surface unless it has been weathered. This can be due to the fact that the termites were not able to detect the cellulose of the fiber (not accessible) on un-weathered surface and if they could, it would not be easy to remove them. Cracks and loose bonding between fibers and polyethylene facilitate the action of termites.

Effect of Weathering on Wettability and Roughness

Results in Table 2 show that adding pigment to the flour resulted in a higher wettability of the surface (lower θ). After exposure to weathering, the wettability of the control sample increased considerably, as also observed on WPC (Stark and Matuana 2007), while it decreased for the pigmented samples. These facts can be linked to chemical variations: for control samples, LI dropped markedly after weathering, suggesting that the degraded fibers on the surface had some of their lignin removed, while the small cracks mentioned previously (Fig. 3) allowed the accessibility to more fibers. Pigmented samples exhibited the highest WI and HI change after weathering, meaning more fibers were removed from the surface and there was a higher ratio of remaining polymer, leading to the observed decrease in wettability. Because of cracks and fiber removal, R_a and R_q, quantifying the depths of voids created on the surface, increase. After fungal or termite attack, the surface quality was so obviously damaged that there was no point in measuring roughness.

Effect of Weathering and Biodegradation

Weight loss from fungal attack shown in Table 2 can be also considered as a surface property, as only a thin layer of surface was affected. For both un-weathered and weathered samples, WL was indeed very small, far within the threshold required to consider the material as very durable. The pigmented material was slightly more vulnerable to fungi than the reference. For termite attack, as shown in Table 2, the weathered samples were more susceptible to fiber removal. This can be less in WAp compared to WA0, as less fiber remained on the surface than in W0, due to weathering. However, the termite survival rate was zero. Un-weathered samples remained untouched after the termite test, while fibers were removed from the surface of weathered samples. However, in both cases WL was negligible (visual rating = 0 for all NFPCs meaning no attack visible by the eye) and at the end of the test not even one termite survived with the NFPCs, while for controls the survival rate was above 50% and a visual rating of 4 (strong attack) was obtained; pine sapwood virulence controls showed 13.6 % weight loss indicating good conditions of termite activity.

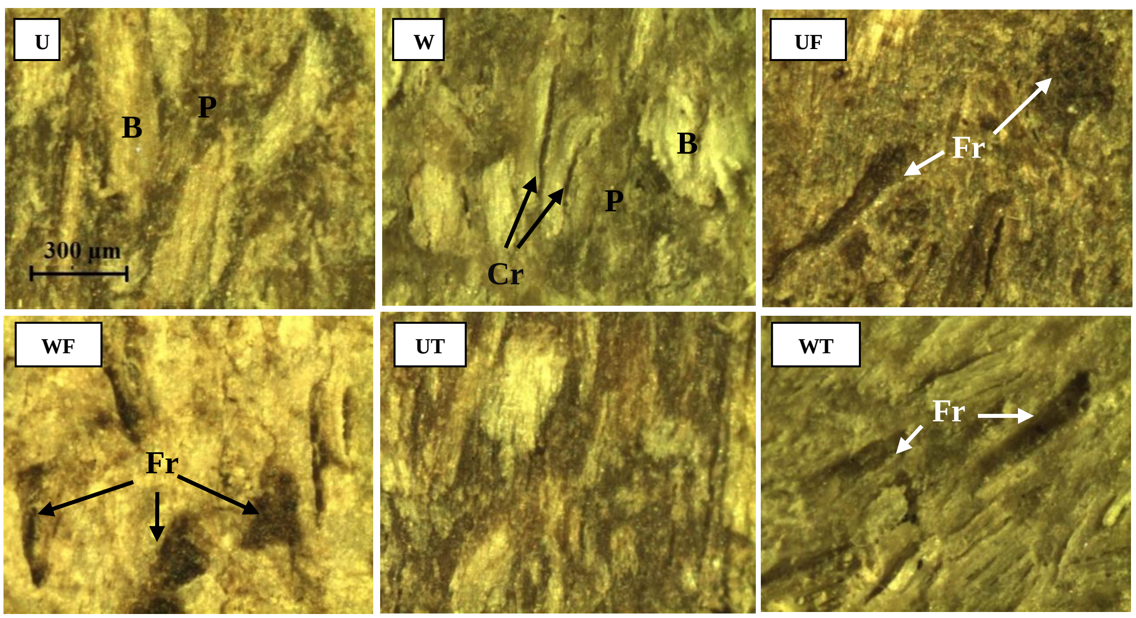

Fig. 3. Surface of control (non-pigmented) sample at different stages of degradation: U: un-weathered; W: weathered; F: after fugal attack ; T: after termite attack. B = bagasse fiber; P = Plastic; Cr = cracks; and Fr = fiber removal.

CONCLUSIONS

1. Using agricultural bio-products such as bagasse as filler resulted in a low level of color change, even without any additive as usually the case for WPC.
2. Presence of pigment can reduce the lightness and total color change due to less lignin degradation besides causing an increase in polyethylene oxidation. However, the total color change was less in comparison to non-pigmented samples.
3. Despite high resistance of NFPC against biological attack, weathering can increase the water absorption due to surface cracks and lignin degradation. Therefore, it becomes easier for termites to use the fibers at the surface.
4. Generally samples in this study can be classified as highly resistant for exterior use above ground.

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