

Enhancement of Recycled WPC with Epoxy Nanocomposite Coats

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THE RECYCLED plastic and wood waste were recycled to produce new useful product, wood plastic composite (WPC), that having characteristics close to commercial wood. WPC was performed by compounding and forming different recycled polyethylene/wood flour (PE/WF) weight ratios as 70/30, 60/40, 50/50 and 40/60 using twin screw extruder. Based on the mechanical properties measured via dynamic mechanical analysis (DMA), an optimized ratio was found to be (50/50) and was chemically treated with chromic acid solution and investigated by FTIR that approved successful functionalization after treatment. The treated WPC was then coated with different epoxy nanocomposites including reactive rubber nanoparticles (RRNP), montmorillonite nanoclay (MMT) and carbon nanotubes (CNTs). The coated WPC was fully tested using mechanical characterizations (modulus and scratch), water absorption (dist. and sea water), and thermal gravimetric analysis (TGA). The results showed that WPC coated with epoxy/CNT, epoxy/MMT and epoxy/RRNP has an enhanced final product with promising mechanical and physical properties compared to blank or uncoated WPC specimens. Also WPC coated with all epoxy nanocomposites has got enhanced modulus, more scratch resistance, lower water absorption and developed thermal stability, compared to the un-filled epoxy coat. Of those, the epoxy-CNT nanocomposite is an optimized protective coat for WPC even after immersion in water with different conditions.

Keywords: Coating, Nanocomposites, WPC, Modulus.

Introduction

The term wooden polymer composites "WPCs" relates to composites product that contains plant/natural fiber and thermoset or thermoplastic. Thermosets are plastics that, once cured, cannot be melted by repeating such as epoxies and phenolic. Thermoplastics are polymers that can be repeatedly melted such as polypropylene (PP), polyethylene (PE) and polyvinyl chloride (PVC) [1,2]. The hydrophobic nature of thermoplastic matrix and the hydrophilic nature of the fibrous filler can cause some incompatibility between both components resulting in undesirable properties. So it is usually to use coupling agent such as maliated PP and maliated PE to make enhanced attachment with stronger fiber-matrix interface [3,4]. Plastic and agricultural wastes that have a main environmental concern can be used in the preparation of advanced WPCs. These new composites have a variety of applications ranging from component

and product prototyping to outdoors, indoors, decking, construction and automotive applications [5,6]. WPCs already have a significant market share and the trend is increasing, the current reliance on plastics, especially those derived from fossil sources, means that this demand is likely to continue increasing especially as developing economies continue to grow.

Other advantages for WPCs are environmental impact, ease of maintenance, improved durability, acceptable relative strength and stiffness and fewer prices [7,8]. Different types for preparation, treatment and coating of WPC have been studied; the type and technique depend on the final application and environment of composite. Previous studies have been reported on surface treatments making the WPC surface more adhere with the coating layer. Gramlich et al. (2006) performed different treatments on WPC to improve its adhesive of its surface with epoxy,

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the treatments were chromic acid, flame, water, flame then water and water then flame[9]. The chromic acid and flame treatments resulted in considerable improvement in joint strengths. Oporto *et al.* (2007) evaluated various surface treatment techniques including mechanical methods. Thermodynamic and spectroscopic techniques were employed for surface characterization, it was postulated that the WPCs relatively low surface energy was likely due to the surface migration of lubricant used in the extrusion process[10]. Gupta and Laborie (2007) used chromic acid, flame, oxygen plasma and benzophenone/ultraviolet irradiation surface treatment techniques for improving the adhesion; they observed that the chromic acid and flame treated substrates had the highest and lowest joint strengths, respectively[11]. Laborie and Gupta (2008) used a proprietary oxyfluorination surface treatment to evaluate adhesion of an acrylic coating to WPC substrates, the WPC formulations were altered by introducing maliated-PP coupling agent into the compounds. The indicators for changes in surface chemistry were due to increase in carbonyl and hydroxyl absorption bands on IR spectra [12]. Wolkenhauer *et al.* (2008) applied plasma treated-WPC substrates and measured adhesion of paints and adhesives to substrates that resulted in an increase in polar component of surface energy and also surface roughness of substrates[13]. Oporto *et al.* (2009) used "forced atmospheric air plasma" to treat WPC surface, the analysis indicated that the acid-base component of the surface energy for WPC after treatment correlates with an increase in wettability. The results of shear tests on bonded joints for treated substrates showed a 50% increase compared to blank samples [14]. Dimitriou *et al.* (2016) have studied three effective surface pretreatment methods for WPCs; hydrogen peroxide, hot air and flame. A further treatment using halogen heat lamp failed to show an improvement in adhesion at any of the speeds studied [15]. Yáñez-Pacios and Martín-Martínez (2017) used atmospheric pressure rotating plasma jet of several WPCs made with different polymers which is considered as an environmentally friendly alternative for increasing their hydrophilicity and adhesion[16].

After treatment specimens coated with epoxy which modified with different nanomaterials such as carbon nanotubes (CNTs) [17].

In this work, different ratios of WPCs based on recycled PE and wood flour were prepared. The surface of WPC has subjected to surface modification using chromic acid solution and then coated with different epoxy-CNT/MMT/RRNP nanocomposites. The obtained coated WPCs have been fully characterized using microstructural, thermal, physical and mechanical techniques.

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Experimental

Materials and chemicals

Recycled PE and wood flour (local source) were used for the preparation of WPC, maleic anhydride PE (Sigma Aldrich) was used as coupling agent. Potassium dichromate and sulphuric acid (Sigma Aldrich) were used for preparation of chromic acid solution for treatment of WPC. The epoxy coat is Bisphenol-A Epotuf U.S Composites. The nanofillers used for filling epoxy are carboxylic acid treated multiwall carbon nanotubes (MWCNT-COOH) from Cheap Tubes, Cloisite 30B montmorillonite nanoclay (MMT) from Southern Clay Products and styrene butadiene reactive rubber nanoparticles (RRNP) that were synthesized as reported by Ahmed *et al.* (2015) and Solyman *et al.* (2017) [18,].

Formulation of WPC

Wood flour was firstly dried at 80°C for 2 hr. and then mixed with recycled PE using mechanical mixer. Blank PE and different ratios of PE/wood flour as 70/30, 60/40, 50/50 and 40/60 weight % were compounded using MD-30 Bausano Twin screw extruder containing five zones with gradual temperature range starting from 170 to 180°C. 3.7% is a constant concentration of the maleic anhydride PE coupling agent in all patches.

Treatment of WPC

The WPC surface was subjected to treatment with chromic acid solution following ASTM D-2093. Samples were immersed at room temperature for 30 min. in a solution prepared by adding 3.75 g potassium dichromate to 6 and 40.8 ml distilled H₂O and conc. H₂SO₄, respectively.

After immersion, the treated samples were washed and dried at 37°C for 2 hr.

Coating of WPC

The treated sample was coated using epoxy and epoxy-MWCNT, epoxy-MMT and epoxy-RRNP nanocomposites as 1, 6 and 7% (an optimized percent) respectively, according to optimized concentrations of epoxy-MWCNT/MMT/RRNP nanocomposites as listed in Ahmed *et al.* (2015). Each nanofiller was dispersed in the epoxy base with sonication for 10 min., then the hardener was added and mixed well (base: hardener is 2:1) and applied on the treated WPC surface with a brush. Finally, the coated nanocomposites were held for seven days at room temperature for curing.

Characterization

Fourier transform infrared spectroscopy

ATR-FTIR Alpha Bruker Platinum Fourier transform infrared spectroscopy was used to investigate the chemical treatment on the surface of WPC.

Dynamic mechanical analysis

The storage modulus (E') of WPCs was measured using bending mode in Triton Technology dynamic mechanical analysis (DMA) following ASTM D-4065. Samples with 25, 10 and 3 mm length, width and thickness were heated by 5°C/min to 100°C at 1Hz frequency.

Water gain

The stability of the WPCs against sea and distilled water absorption was calculated using Equation 1 according to ASTM D-570 where W_{wet} is the weight after immersion and W_{original} is initial weight, Samples were immersed in distilled and sea water at room and boiling temperature.

$$\text{WPG}(\%) = \frac{W_{\text{wet}} - W_{\text{original}}}{W_{\text{original}}} \times 100 \quad \text{Equation 1}$$

Nanoscratch

Nanoscratch test was performed for coated WPCs using Nano Test Vantage machine. Each sample was tested against 3 scratches using the Pyramidal indenter. The scratch load is 200 mN that was applied by load 10 mN/sec, the scratch length is 500 μm . The resulted scratch depth was drawn against distance.

Thermal gravimetric analysis

Thermal gravimetric analysis (TGA) was performed for all epoxy nanocomposites to show the effect of addition of the different nanofillers to epoxy coat. This analysis was done using TGA 55 TA Instrument, all samples were heated from room temperature to 600°C with a heating rate of 5°/min. under N_2 gas.

Results and Discussion*Optimization of WPC*

The storage modulus was measured for all WPCs by DMA to investigate an optimized ratio that achieved the highest value as in Table 1. It is clear that storage modulus increases with increasing wood flour till the 50/50 composite that achieved the best mechanical properties (1.46 GPa). Increasing more wood flour leads to decrease the modulus as in 40/60 WPC. In this ratio, the coupling agent cannot combine the polyethylene with wood fiber together due to the more ratio of wood flour leading to poor mechanical

properties. So, the 50/50 WPC has been selected as an optimized composite.

Fourier transform infrared spectroscopy for chromic acid treatment

The FTIR spectra for 50/50 WPC treated with chromic acid at 30, 60 and 120 min. are shown in Fig. 1. All samples include peaks at 2900, 1460 and 1023 cm^{-1} that respectively characteristic to C-H stretching, C-H bending and C-O-C stretching. For both aliphatic C-H stretching and bending (Fig. 2), the corresponding peaks have increased intensities with increasing the immersion time in chromic acid solution till 60 min, due to the effect of etching resulting in more aliphatic groups. However, more immersion time with 120 min. lead to lower intensities of both peaks due to lower C-H groups, compared to 30 and 60 min., but still more than the untreated sample. For the ether peak (Fig.3), it became broader with increasing the immersion time regarding the formation of inter/intra molecular hydrogen bonding [20]. Consequently, the more functionalized WPC sample treated with chromic acid for 60 min. has been selected for further coating.

Dynamic Mechanical Analyzer

The effect of nanofillers on the mechanical properties of epoxy coated WPC was studied by measuring of modulus using dynamic mechanical analyzer (DMA), also, the effect of water immersion (distilled and sea water at room and boiling temperature) on the modulus of epoxy or epoxy-MWCNT or epoxy-MMT or epoxy-RRNP coated 50/50 WPC, are indicated in Table 2. All samples were immersed in sea and distilled water at boiling and room temperature to investigate the mechanical stability for epoxy nanocomposite coat after water diffusion. Before immersion, it is clear that modulus of coated WPC was enhanced; the highest modulus was achieved by epoxy-CNT coat followed by epoxy-MMT coated due to the strong modulus for nanofillers. Epoxy-RRNP coat achieved lower modulus compared to epoxy-CNT and epoxy-MMT, but still higher than epoxy (neat) coated sample, due to the elasticity nature for RRNP. Even after water immersion at the indicated different conditions; coating WPC with different epoxy nanocomposites (epoxy-CNT/MMT/RRNP) lead to increase the modulus of whole samples.

Since WPC voids and the lumens wood flour were filled with nanofillers, this prevented the water diffusion

TABLE 1. Dynamic mechanical analysis (DMA) for blank recycled PE and different WPC composites.

Sample (PE/Wood flour)	Blank PE	70/30	60/40	50/50	40/60
Storage modulus (GPa)	0.22	0.79	0.91	1.46	1.32

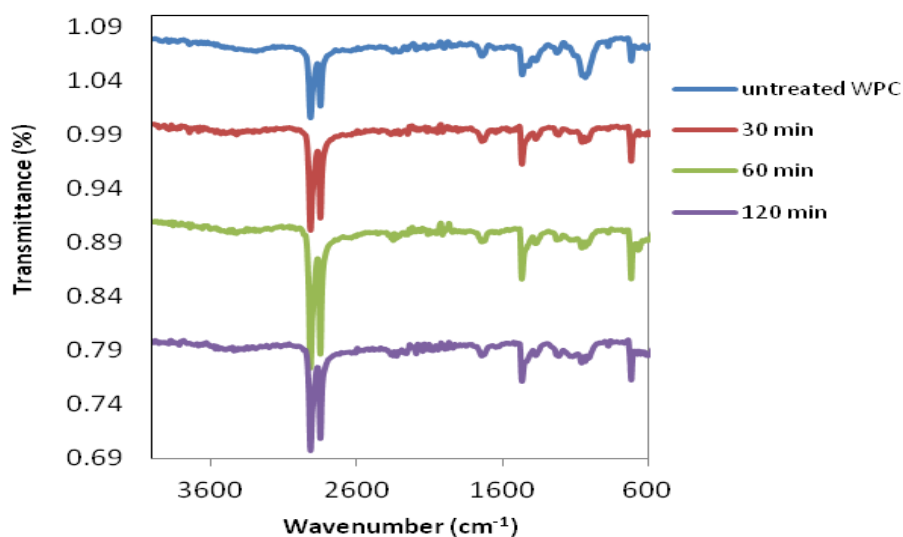


Fig. 1. FTIR for untreated and chromic acid-treated WPC at different times of immersion (30, 60,120) min.

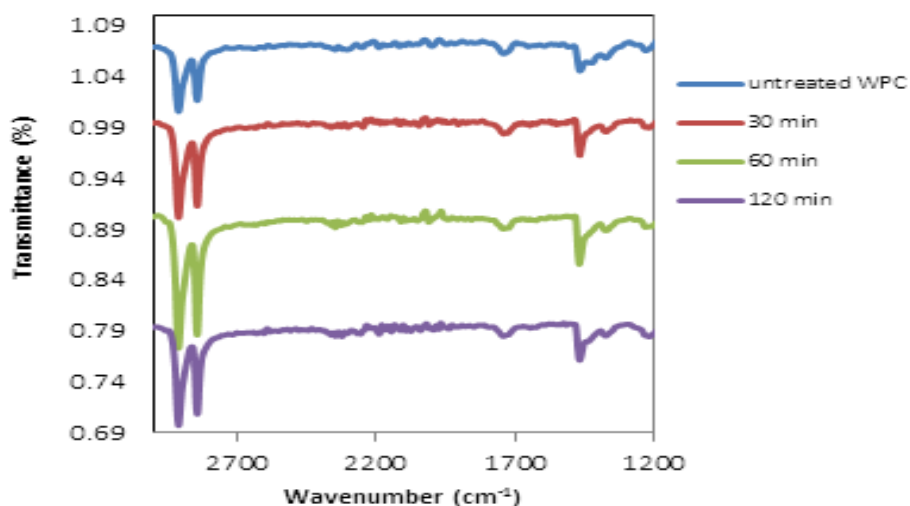


Fig. 2. Aliphatic stretching and bending peaks for untreated and chromic acid-treated WPC.

inside WPC by the capillary action, compared to control samples (without nanofillers). It can be concluded that epoxy nanocomposite coats succeeded in enhancement of the mechanical property of WPC in addition to protection of its surface from water penetration.

Thermogravimetric analysis

Thermogravimetric analysis TGA is a method that is based on the measurement of weight change in relation to temperature. The standard TGA method employs a constant heating rate and usually an inert environment of nitrogen or argon; or this may employ

a “high resolution” heating method that is based on adjusting the heating rate in accordance with the rate of weight loss. This method, which results in a very slow heating rate at the temperature at which the conversion of solid mass into gaseous degradation products reaches a maximum, has significantly increased sensitivity compared to the standard (constant heating rate) methodology. In addition to gaining resolution by adjusting the heating method, the sample’s surrounding atmosphere may be changed to cause degradation of a particular component. The thermal degradation profiles of blank epoxy, epoxy-CNTs, epoxy-MMT and epoxy-

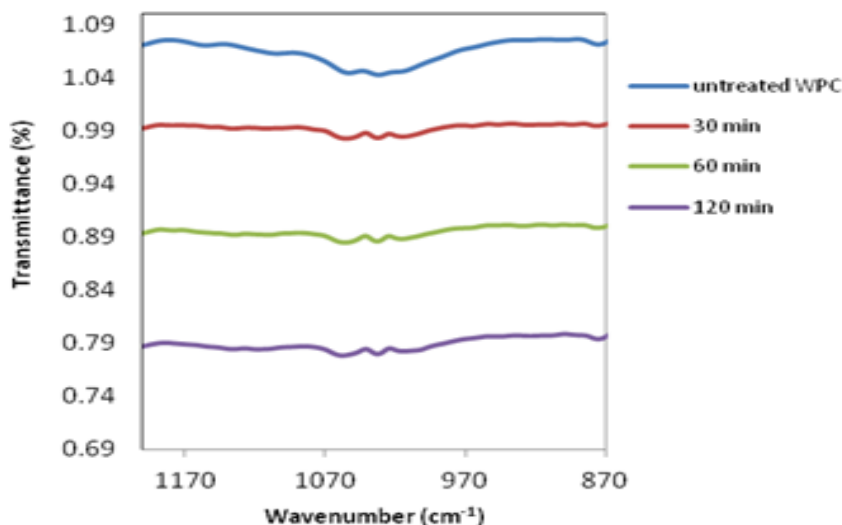


Fig. 3. Ether peak at (1023) cm-1 for untreated and chromic acid-treated WPC.

TABLE 2. Modulus values (GPa) for epoxy and epoxy-CNT/MMT/RRNP coated 50/50 WPC before and after different condition water immersion.

Blank WPC		1.46	
Coated WPC before water immersion	Epoxy		1.70
	Epoxy-CNT		2.22
	Epoxy-MMT		1.72
	Epoxy-RRNP		1.49
Coated WPC after immersion at boiling point	Epoxy	Dist. water	1.62
		Sea water	1.71
	Epoxy-CNT	Dist. water	1.83
		Sea water	1.84
	Epoxy-MMT	Dist. water	3.91
		Sea water	1.84
	Epoxy-RRNP	Dist. water	1.62
		Sea water	2.08
Coated WPC after immersion at room temp.	Epoxy	Dist. water	1.73
		Sea water	1.84
	Epoxy-CNT	Dist. water	1.79
		Sea water	1.73
	Epoxy-MMT	Dist. water	1.52
		Sea water	1.84
	Epoxy-RRNP	Dist. water	1.52
		Sea water	3.37

RRNP by TGA reveals that most the degradation events occur between 300°C and 425°C as shown in Fig. 4. Three steps in the thermal decomposition could be observed.

The first step degradation occurred between 70°C

to 200°C while the second step degradation occurred above 200°C for all samples. The first step weight loss was associated with the evaporation of absorbed moisture from the samples.

There are three different types of water, such as free

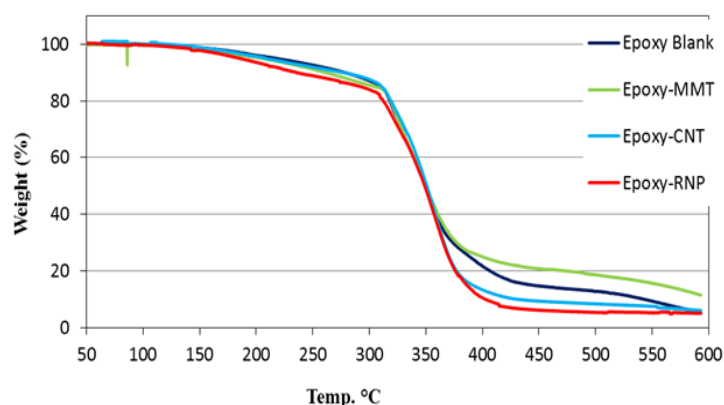


Fig. 4. Thermogravimetric analysis for blank epoxy and epoxy-MWCNT/MMT/RRNP Nanocomposites.

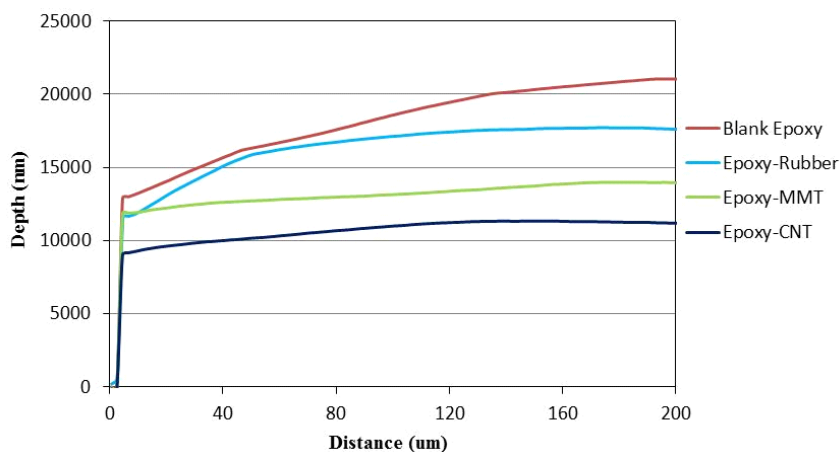


Fig. 5. Nanoscratch for WPC coated with blank epoxy and epoxy-MWCNT/MMT/RRNP nanocomposites.

water, loosely bonded water and chemically bonded water, in wood. As a consequence, the loss of water, as recorded by the thermo gravimetric curve, is the result of the overlapping of three different processes in which the three types of water are lost. The polyolefin (PE) degrade over a 150°C - 200°C temperature range. Second-step degradation between 300°C - 450°C. In this second stage, hemicellulose, cellulose and lignin degrade between 200°C -500°C most of the degradation events occur between 300°C and 450°. The final step, between 450°C and 600°C at this step it is very clear that MMT has the lower weight loss percent this due to the fact that MMT layers could prevent the passage of volatile decomposed product throughout the composite. Thus the addition of MMT gives the best thermal stability degradation of all nanocomposites.

Nanoscratch

The interaction between the treated WPC surface and different epoxy coats was examined by

nanoscratching as in Fig. 5. The values of deformation depth, created by the scratching indenter, are reduced with all epoxy nanocomposite coats. The deformation degree is as epoxy-CNT < epoxy-MMT < epoxy-RRNP < epoxy within all scratch distance; the lower deformation depth (~11000 nm for epoxy-CNT), the stiffer interface. The ability of epoxy-CNT to reinforce and enhance the surface profile of WPC is clear here regarding minimum deformation depth reached, compared to the un-filled epoxy coat that deformed with largest depth (~21000 nm). Although epoxy-RRNP coat has the highest depth (~17000 nm) among all nanocomposites, its deformation depth still lower than epoxy. This manner resulted from the ductile effect of rubber particles. As a whole, a surface interaction has successfully created between the treated WPC and all epoxy nanocomposites leading to stiffer surface, which has been detected by lower scratching depths.

The interaction between the treated surface of WPC

TABLE 3. WPC samples before and after water immersion at different conditions.

WPC with Blank Epoxy		
Conditions		%WPG
Room temp. (24h)	Dist. Water	1.67
	Sea water	1.13
Boiling temp. (2h)	Dist. Water	2.42
	Sea water	2.26
WPC with Epoxy-CNT		
(24h).Room temp	Dist. Water	0.42
	Sea water	0.38
Room temp. (2h)	Dist. Water	1.2
	Sea water	1.4
WPC with Epoxy-MMT		
Room temp. (24h)	Dist. Water	0.39
	Sea water	0.42
Boiling temp. (2h)	Dist. Water	1.94
	Sea water	1.83
WPC with Epoxy-RRNP		
Room temp. (24h)	Dist. Water	0.68
	Sea water	0.58
Room temp. (2h)	Dist. Water	1.83
	Sea water	1.61

and coat was examined by scratching. Figure 5 shows the obtained deformed depth versus scratch distance for WPC coated with blank epoxy and epoxy-MWCNT/MMT/RRNP nanocomposites. It is clear that the epoxy-MWCNT, epoxy-MMT and epoxy-RRNP show less deformation depth than blank epoxy. This is due to more chemical interaction between the treated WPC surface and epoxy nanocomposite.

The less deformation depth the stiffer interface, so epoxy-MWCNT has stiffer interface than epoxy-MMT then epoxy-RRNP blank sample has the less interface stiffness.

Water gain

Water uptake behavior is important to investigate the durability of the WPC exposed to the environmental conditions. The results of water percentage gain (WPG) for all composites after immersion in sea and distilled water at boiling and room temperature are indicated in Table 3. It is clear that values of WPG are lower after immersion at room temperature, compared to boiling water with both sea and distilled water. Furthermore, the least WPG was achieved by epoxy-CNT and epoxy-MMT coats, compared to epoxy-RRNP coat which still

lower than blank epoxy. Even after water immersion at different conditions; coating WPC with different epoxy nanocomposites (epoxy-MWCNT, epoxy-MMT and epoxy-RRNP) lead to minimize the WPG of whole samples. It can be concluded that epoxy nanocomposite coats succeeded in protection of WPC surface against water attack, this notification assures the improvement in modulus values for all nanocomposite coats discussed before.

Conclusions

Different percentages of recycled PE-wood flour WPC have been formulated and optimized, an 50/50 composite achieved best mechanical properties due to the effect of coupling agent. An optimized WPC has been functionalized by chromic acid treatment, FTIR spectra indicated that treatment for 60 min. succeeded in treatment of WPC surface to be able to interact with epoxy-MWCNT/MMT/RRNP nanocomposite coats. The different epoxy nanocomposites have improved the mechanical properties of WPC, epoxy-CNT achieved the highest modulus followed by epoxy-MMT and epoxy-RRNP, due to the elastic manner

for RRNP. In addition, all nanocomposites recorded lower water absorption values regarding surface protection and chemical interaction between WPC surface and nanocomposite coat. Even after water immersion, all epoxy nanocomposite coats recorded enhanced modulus values compared to neat epoxy-coated WPC. Nanoscratch test indicated the strong interaction between treated WPC and nanocomposite coats through the obtained deformation depth which is a minimum with epoxy-CNT, this result from the more stiff coating layer.

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تحسين الخشب البلاستيكي باستخدام دهانات الايبوكسي النانومترية

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تم فحص وتجميع عدد من المؤلفات العلمية المنشورة حول الخشب البلاستيكي (WPC)

Wood plastic composite

وأيضاً حول المعالجات السطحية باستخدام دهانات مختلفة. تم تحضير نسب مختلفة من متراكبات البلاستيك الخشبي صديق البيئة باستخدام جهاز البائق الحراري وتعيين أفضل نسبة والتي حققت أعلى خواص ميكانيكية. وتم ذلك بعمل عينات بنسب خلط مختلفة من مادتي البولي إيثيلين ونشارة الخشب باستخدام البائق الحراري وشملت (80/20) (70/30) (60/40) (50/50) (40/60) % وزناً وبسبب طبيعة البلاستيك الكاره للماء (hydrophobic) والخشب المحب للماء (hydrophilic) فمن الضروري إضافة عامل اقتران (coupling agent) لتشكيل الخشب / البوليمر المركب الثابت عن طريق الترابط الكيميائي.

ولقد اتضح من نتائج قياس القوة الميكانيكية الديناميكية (DMA) أن أفضل نسبة هي (50/50%) (البولي إيثيلين / نشارة الخشب) وتم تحضير وتوصيف مركبات مختلفة نانومترية الحجم واستخدامها كإضافات محسنة ومقوية لمادة الإيبوكسي والتي تستخدم كطلاء لحماية المتراكبات المحضرة.

تم معالجة أفضل نسبة من الخشب البلاستيكي كيميائياً باستخدام حمض الكروميك وذلك طبقاً للمواصفة ASTM D2093. وكذلك طلاء أفضل نسبة من الخشب البلاستيكي بمادة الإيبوكسي الغير معالج. وكذلك طلاء أفضل نسبة من الخشب البلاستيكي بمادة الإيبوكسي المعالج باستخدام مواد نانومترية بنسب مختلفة من مواد Carbon nanotubes (CNTs)، Montmorillonite (MMT) و Reactive rubber nanoparticles (RRNP).

تم غمر عينات الخشب البلاستيكي (أفضل نسبة) في الماء وتم إيجاد نسبة الماء الممتص (عن طريق وزن العينات قبل الغمر وبعد الغمر ومن ثم إيجاد الفرق بينهما) عند ظروف بيئية مختلفة للعينات الغير معالجة والمعالجة على السواء تم قياس الخواص الفيزيائية والميكانيكية للعينات كل على حده لمعرفة مدى فاعلية عملية المعالجة وكذلك الطلاء وتأثير كل منهما على الخواص الفيزيائية والميكانيكية. بالنسبة لعملية المعالجة بحمض الكروميك تبين باستخدام التحليل الطيفي بالأشعة تحت الحمراء أن هذه المعالجة قامت بتحسين الخصائص الميكانيكية مقارنة مع العينات الغير معالجة وبالنسبة إلى التحليل الحراري الوزني (TGA) Thermogravimetric analysis

وجد أن إضافة هذه المواد النانومترية قد حسن من الخواص الحرارية. وبدراسة الخصائص الميكانيكية باستخدام جهاز التحليل الميكانيكي (DMA) Dynamic mechanical analyzer

وجد أن الجسيمات النانومترية المضافة تجعل الإيبوكسي أكثر قوة وصلابة ومقاومة للضغط. أما بدراسة الخصائص الميكانيكية بمقياس النانو باستخدام Nanoindentation technique وجد أن اختراق الإبرة لسطح العينة المغطاة بالإيبوكسي المخلط بجسيمات نانومترية أقل منه بالنسبة للإيبوكسي الخام.