



Biological properties of nutshell and microcrystalline cellulose (MCC) filled high density polyethylene composites

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ABSTRACT

Purpose: The composites based on natural fibre and wood can be destroyed by fungi attack, even the lignocellulosic materials were used as filler in hydrophobic thermoplastic matrix. The aim of this study was to investigate the effect of microcrystalline cellulose (MCC) and nutshell fibre content on decay resistance of natural fibre composites.

Design/methodology/approach: Half of the composite samples were immersed in water for 60 days and then incubated by fungi to investigate the leaching effect on biological resistance. Furthermore, water absorption rate and thickness swelling of samples were determined during water immersing. For this purpose, total nine thermoplastic composites filled different MCC rate (5%, 10% and 15%) and nutshell content (30%) were produced. Decay test were conducted by using a white rot fungus–*Trametes versicolor*, and a brown rot fungus–*Tyromyces palustris*, according to EN 113 standard.

Findings: Based on findings from this study, weight loss, water absorption rate and thickness swelling correlated with lignocellulosic content in composites. Samples exhibited less than 1% weight loss in decay test and excellent biological resistance against testing fungi.

Research limitations/implications: Weight loss (%) and moisture content (%) values of MCC-nutshell HDPE composites after *Tyromyces palustris* and *Trametes versicolor* attack were found under 3% and 20%, respectively. Low weight loss values obtained in the study are supposed to be related with the low moisture content.

Originality/value: There is not a study dealing with the decay resistance of WPCs produced by a combination of nutshell and MCC fibres. In addition, there is not a substantial study on the effects of MCC/plastic ratio for decay performance of WPCs contained nutshell after long-term leaching test.

Keywords: Microcrystalline cellulose; Nutshell; Composite; Decay; Water absorption; Thickness swelling

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PROPERTIES

1. Introduction

Lignocellulosic fibres obtained from cotton, coconut, flax, hemp, jute, kenaf, sisal, wood flour/fibres, tea mill waste, recycled fibre based products such as newspaper, and agricultural residues like hazelnut shells, rice husks, banana fibre and sugarcane bagasse have been used due to the growing concern for environmental protection [1-4].

Agricultural wastes have several advantages such as low price, biodegradability, easy-accessibility, profitability for farmers, and causing low erosion to process equipment compared to glass fibre, talc, and calcium carbonate [4,5]. Various agricultural wastes were added in polymer matrix made by polyethylene, polystyrene, polyvinyl chloride, polypropylene, and polyethylene terephthalate [1,6-7]. Among agricultural wastes, nutshell can be used with thermoplastic matrices to obtain composite materials [8]. The world nutshell production has been approximately 497,000 tons per year for the last five year. Turkey which is the biggest exporter of nutshell is supplied of around of 70-75% of the total hazelnut production in world [4,9]. MCC has generated much attention in industrial fields because of renewability, biodegradability, high surface area and stiffness, and availability from different sources [10-11]. The combination of MCC with the matrix encounters is important to produce better cellulose based composites because of the issue of compatibility between hydrophilic cellulose and hydrophobic polymers [12,13].

Wood fiber filled plastic composites have significant advantages such as increased mechanical properties, decreased thermal expansion, easy availability, and low costs [14]. Despite these advantages, the addition of wood fiber into polymer matrix decrease dimensional stability and resistance against biological attacks of composites [15,16]. Wood polymer composites (WPCs) in above ground applications could also be susceptible to decay by fungi. Fungi colonisation on WPC deck boards in Florida and Hawaii was observed. Therefore, biological performance of WPCs in field and laboratory tests has become a major interest in nowadays since the demand for WPCs has increased [17].

This study aims to investigate the effect of nutshell and various loadings of MCC fibres on some physical properties and decay resistance of WPCs. Leaching test for 60 days in distilled water was conducted on the samples in order to evaluate any loss in effectiveness in biological resistance.

2. Description of the approach, work methodology, materials for research, assumptions, experiments etc.

The high-density polyethylene (HDPE) with a density of 0.96 g/cm^3 and melt flow index of 11 g/10 min at 190°C /2.16 kg was purchased from Petkim Petrochemical Company in Izmir, Turkey. Microcrystalline cellulose (MCC) with average volume diameter~ 40 μm and polyethylene graft maleic anhydride (PE-g-MA) as a coupling agent with a density of 1.48 g/cm^3 and melting point of 52.8°C were supplied by Merck (Germany). Nutshells were obtained from the East Black-Sea region in Trabzon, Turkey. The granulated nutshells were screened into three size fractions ranging from 0.5 to 2 mm. The nutshell fibres of 0.5-mm screen were used for MCC-nutshell production.

2.1. Preparation of MCC and nutshell filled composites

Processing parameters are listed in Table 1. All materials were dried in an oven at 80°C until they reached below 1% moisture content. The oven-dried nutshell fibres (30 wt.%), HDPE, PE-g-MA (4 wt.%), and MCC (5, 10, and 15 wt.%) as stated rates were mixed for 3 min before composite production. Firstly, all MCC and nutshell compounding was conducted using a single-screw extruder (L/D 30, TeknomatikCo., Turkey). The temperature was set to 180°C and the screw speed at 50 rpm. The extrudates were cooled using a water bath and then pelletized. All pellets were granulated by a lab scale grinder. Then the pelletized materials were dried in a conventional oven at 105°C for 24 h for injection moulding. All compounded materials were completed using an injection moulding machine (HDX-88, Ningbo Haida Plastic Machinery Co. Ltd., China). The granulated pellets were pressed using mould dimensions of 20 cm by 20 cm by 0.5 cm under 102 kg/cm^2 at 180 to 200°C from feed to die zone with an injection speed of 80 mm/s.

2.2. Physical properties

Water absorption rate (WA) and thickness swelling (TS) of composites were determined according EN 317 (1993) [18]. The MCC-nutshell HDPE composites were conditioned at 20°C and 65% relative air humidity prior to immersion in distilled water. The samples were immersed in distilled water at 23°C for 60 days. Water was changed

with the fresh water for every week. Five replicate samples were tested for each group. The thickness swelling of each sample was calculated based on its dry thickness while the water absorption of each sample expressed as water weight absorbed was calculated based on its initial dry weight. After water immersing test, the WPCs were exposed to decay test. Those samples were labelled as leached WPCs in the study.

Table 1.
Parameters of MCC and nutshell filled polymer composites

Samples	Composition*			
	PE	PE-g-MA	MCC	Nutshell
Neat HDPE	100	-	-	-
HDPE/MCC-0	96	4	-	-
HDPE/MCC-5	91	4	5	-
HDPE/MCC-10	86	4	10	-
HDPE/MCC-15	81	4	15	-
HDPE/MCC-0/N-30	66	4	-	30
HDPE/MCC-5/N-30	61	4	5	30
HDPE/MCC-10/N-30	56	4	10	30
HDPE/MCC-15/N-30	51	4	15	30

*Values are percentage by weight (wt.%); HDPE: high density polyethylene; MCC: microcrystalline cellulose; N: nutshell; and PE-g-MA: polyethylene graft maleic anhydride

2.3. Decay test

Decay test was carried out according to principles of EN 113 (1997) with some modifications on sample dimensions, kolle flasks and total test period [19]. Plastic sterile petri dishes with the diameter of 9 cm were used instead of kolle flasks. Five replicates for each group were exposed to fungal attack. Beech (*Fagus orientalis* L.) and Scots pine (*Pinus sylvestris* L.) wood samples with the same dimensions as WPCs had were exposed to fungal attack as reference specimens for validity of decay test. A brown-rot fungus *Tyromyces palustris* and a white rot fungus *Trametes versicolor* were inoculated in the petri dishes contain %4.8 sterile malt extract agar solution. After incubation period of inoculated petri dishes, two WPCs were placed on the growing mycelium in each petri dish. Wood samples were placed separately. The petri dishes were then incubated at 20°C and 65% RH for 40 days. After the test all WPCs and wood samples were cleaned from the surface mycelium, and weighted for moisture content calculations. Then, they dried at a temperature of

103±2°C, weighed, and the weight loss was calculated on the basis of oven dry weight before and after fungal attack for 40 days.

Differences among treatment groups were analyzed using one-way analysis of variance (ANOVA) test using with SPSS 18.0 program. The significance ($P<0.05$) was compared with Duncan homogeneity groups. Means with the same letter for each property showed no significant difference at the 5% significance level by Duncan's homogeneity groups.

3. Description of achieved results of own researches

3.1. Water absorption and thickness swelling of samples

The water absorption (WA) and thickness swelling (TS) of MCC-nutshell HDPE composites after water immersion of 60 days are given in Table 2. The WA values varied between 0.17% to 0.62 for MCC/HDPE composites, and 1.13% and 1.36% for nutshell fiber filled HDPE/MCC/N composites. The WA values of HDPE/MCC/N composites were found higher than those of HDPE/MCC composites. The WA of the HDPE/MCC composites decreased with increasing MCC content. The highest WA value was obtained from the addition of 30 wt.% nutshell fiber without MCC. The WA value of the HDPE/MCC composites that consists of 30% nutshell was higher than that of HDPE/MCC composites. It is known that the WA of natural fiber filled thermoplastic composites is influenced by structure of natural fiber. Because natural fiber which contains hydroxyl groups is susceptible to water, it interacted with water molecules by hydrogen bonding [2]. In addition, a linear increase was seen in the WA values of HDPE/MCC/N composites when the rate of MCC increased. There were no significant differences in WA values of MCC filled HDPE composites except for HDPE/MCC-5.

As shown in Table 2, the greatest increase rate was obtained by using HDPE/MCC-0/N-30. TS values of MCC/HDPE composites ranged from 0.60% to 1.32% while that of nutshell fiber filled MCC/HDPE composites varied between 0.92% and 1.54%. The TS of the samples increased until 5% MCC rate, and then a small decrease was observed in HDPE/MCC composites. Similar to WA rate, the TS values of the HDPE/MCC/N composites increased slightly with the increase of MCC. The TS values of HDPE/MCC/N composites were found higher than those of HDPE/MCC composites.

Table 2.

Water absorption (%) and thickness swelling (%) of MCC-nutshell HDPE composites after water immersion of 60 days (%)

MCC-nutshell HDPE	Water absorption (%)	Thickness swelling (%)
Neat HDPE	0.05e*(0.06)**	0.34d(0.49)
HDPE/MCC-0	0.11e(0.10)	0.76bcd(0.88)
HDPE/MCC-5	0.62d(0.13)	0.87bcd(0.85)
HDPE/MCC-10	0.20e(0.13)	0.60cd(0.42)
HDPE/MCC-15	0.17e(0.09)	1.32abc(1.42)
HDPE/MCC-0/N-30	1.66a(0.42)	2.00a(1.02)
HDPE/MCC-5/N-30	1.13c(0.08)	0.92bcd(0.33)
HDPE/MCC-10/N-30	1.16bc(0.23)	1.50abc(0.78)
HDPE/MCC-15/N-30	1.36b(0.30)	1.54ab(0.52)

*: Groups with same letters in column indicate that there is no statistical difference ($p < 0.001$) between the samples according to Duncan's multiply range test.

** : The values in the parentheses are standard deviations.

Table 3.

Weight loss (%) and moisture content (%) of MCC-nutshell HDPE composites after *Trametes versicolor* attack

MCC-nutshell HDPE	Leached Groups		Unleached Groups	
	Moisture Content (%)	Weight Loss (%)	Moisture Content (%)	Weight Loss (%)
Neat HDPE	0.06e* (0.07)**	0.00d(0.09)	0.04d(0.13)	0.03cd(0.07)
HDPE/MCC-0	0.27de(0.19)	0.24abc(0.13)	0.08d(0.09)	-0.07d(0.19)
HDPE/MCC-5	0.76c(0.27)	0.17bcd(0.13)	0.69c(0.18)	0.14abc(0.01)
HDPE/MCC-10	0.77c(0.15)	0.43a(0.22)	0.42c(0.23)	0.29a(0.06)
HDPE/MCC-15	0.58cd(0.06)	0.13cd(0.01)	0.45c(0.16)	0.17abc(0.07)
HDPE/MCC-0/N-30	1.64b(0.42)	0.36ab(0.10)	1.40b(0.27)	0.22ab(0.08)
HDPE/MCC-5/N-30	1.62b(0.19)	0.31abc(0.14)	1.49b (0.21)	0.14abc(0.16)
HDPE/MCC-10/N-30	1.52b(0.41)	0.18bcd(0.16)	1.41b(0.28)	0.08bcd(0.05)
HDPE/MCC-15/N-30	2.13a(0.53)	0.31abc(0.15)	1.87a(0.45)	0.26a(0.07)

*: Groups with same letters in column indicate that there is no statistical difference ($p < 0.001$) between the samples according to Duncan's multiply range test.

** : The values in the parentheses are standard deviations.

The water absorption and thickness swelling determines the field of application of wood plastic composites exposed to surrounding environment. Kord and Hosseinihashemi stated that the fungal decay had an adverse effect on the dimensional stability of bagasse fibre (BF) based polypropylene composites containing the weight ratio of 60BF/40PP [20].

3.2. Decay resistance of samples

Weight loss and moisture content (%) of samples along with Duncan homogeneity groups are shown in Tables 3 and 4. Weight loss was found to be 23.20%

for Scots pine wood after *Tyromyces palustris* attack and 35.52% for beech wood after *Trametes versicolor* attack. These results verified that the decay test was valid.

After *Trametes versicolor* attack, the leached samples exhibited weight loss and moisture content in the range 0.0-0.43% and 0.06-2.13%, respectively. Weight loss and moisture content were found to be 0.0-0.29% and 0.04-1.87%, respectively, for unleached samples. In the case of *Tyromyces palustris* attack, leached samples demonstrated 0.03-0.29% weight loss and 0.09-2.19% moisture content, and unleached samples demonstrated 0.03-0.3% weight loss and 0.09-2.02% moisture content.

Table 4.
Weight loss (%) and moisture content (%) of MCC-nutshell HDPE composites after *Tyromyces palustris* attack

MCC-nutshell HDPE	Leached Groups		Unleached Groups	
	Moisture Content (%)	Weight Loss (%)	Moisture Content (%)	Weight Loss (%)
Neat HDPE	0.09d*(0.22)**	0.03b(0.06)	0.09e(0.16)	0.03a(0.07)
HDPE/MCC-0	0.25d(0.08)	0.21ab(0.24)	0.13e(0.23)	0.19a(0.34)
HDPE/MCC-5	0.98c(0.16)	0.23ab(0.13)	0.59d(0.20)	0.16a(0.12)
HDPE/MCC-10	0.78c(0.19)	0.28a(0.11)	0.44de(0.35)	0.24a(0.36)
HDPE/MCC-15	0.69c(0.17)	0.28a(0.12)	0.51d(0.18)	0.03a(0.06)
HDPE/MCC-0/N-30	1.62b(0.17)	0.25ab(0.09)	1.47bc(0.14)	0.19a(0.14)
HDPE/MCC-5/N-30	1.69b(0.42)	0.29a(0.29)	1.25c(0.42)	0.25a(0.21)
HDPE/MCC-10/N-30	2.08a(0.35)	0.28a(0.08)	2.02a(0.36)	0.30a(0.14)
HDPE/MCC-15/N-30	2.19a(0.43)	0.24ab(0.03)	1.67ab(0.31)	0.18a(0.24)

*: Groups with same letters in column indicate that there is no statistical difference ($p < 0.001$) between the samples according to Duncan's multiply range test.

** : The values in the parentheses are standard deviations.

A relatively low (<0.5%) weight losses were found in HDPE composites. According to EN 113 test, the average weight loss of treated samples should be less than 3% of their initial dry mass for a good protection against decay fungi. HDPE composites had reduced weight losses compared to solid wood samples, met the EN 113 criteria, and had an excellent protection against decay fungi. In general, high moisture level (>30%) is needed by fungi to attack wood. Low weight losses observed in HDPE composites during fungal decay process is related with the low moisture content of the composites [17].

MCC addition to HDPE matrix increased the weight loss and moisture content compared to these of neat HDPE for all groups. In general, the HDPE composites produced with both MCC and nutshell showed greater moisture content and weight loss than HDPE composites with MCC. Nutshell, one of the lignocellulose based materials, could encourage fungi grow or fungi colonization on composites. MCC concentration seemed to have a statistical effect on moisture content and weight loss by *Trametes versicolor* attack. 10% MCC concentration in HDPE composites showed the greatest weight loss both for leached and unleached composites after *Trametes versicolor* attack. In the case of *Tyromyces palustris* attack, MCC concentration did not have a statistical effect on weight loss values but it did on moisture content values. The presence of plastic in WPC prevents moisture uptake, but fungal attack has emerged under high moisture conditions due to fungi degrade wood compounds [21,22]. Some researchers observed the effects of wood in HDPE composites on fungal susceptibility are enriched by soil test. When high

wood content (<50%wt. wood loading) was used in composites, the fungal attack were increased significantly because of hydrolytic degradation process compared to low wood content (>50%wt. wood loading) [23,24]. More wood component is available for the fungus to feed in wood based composites containing 50% wood flour [25]. The weight loss was determined as 18% in polyethylene based wood plastic composites with 50-70 % wood (w/w) after *Trametes versicolor* attack for 16 weeks [26]. Some researchers also confirmed a linear interaction between the fungal attacks and high wood content in composites [27,29]. It is well known that fungal decay affected negatively the physical properties of wood plastic composites containing more than 50% wood content [30,31]. Laks et al. found that the usage of more than 50% weight ratio of wood particle caused degradation by white-rot and brown rot fungi in wood plastic composites [32]. Similar to findings in this study, Ibach et al. declared that 30/70% (aspen fibre/PP) composites had less than 0.25% weight loss exposed to *Gloeophyllum trabeum*. When these composites were exposed to *Coriolus versicolor*, the weight loss did not occur because the fungi could not get access to the fibre into composite with high ratio plastic [33].

In the study, leaching test for 60 days in distilled water increased weight loss and moisture content of HDPE composites, but this method did not cause high weight loss on HDPE composites due to the slow rate of water absorption of composites. Water immersion did not cause high moisture levels that are generally needed by microorganisms.

4. Conclusions

WPCs can be susceptible to fungal decay due to their wood or lignocellulosic material component. Samples in outdoor exposure were found to develop moisture in the outer surface sections of composites which were sufficient to initiate and support fungal decay and growth. Biological performance of WPCs in field and laboratory tests has become a major interest in nowadays. Long-term leaching exposure or some pre-treatments (aging, weathering, water soaking etc.) are more reliable for such end-use performance evaluations. Because WPCs do not get wet easily due to their hydrophobic characters, it is desirable to investigate the effect of long-term leaching periods or pre-treatments prior to decay test.

Fungi need minimum 20% moisture in samples for decay process. In this study, weight loss (%) and moisture content (%) values of MCC-nutshell HDPE composites after *Tyromyces palustris* and *Trametes versicolor* attack were found under 3% and 20%, respectively. Low weight loss values obtained in the study are supposed to be related with the low moisture content. Therefore, long term aging or weathering tests are recommended to increase moisture content in WPCs before decay test as well as to increase weight losses, and to evaluate long term durability of composites in outdoors. For better understanding the role of nutshell and MCC loading level on decay resistance of WPCs, more than 30% nutshell and MCC loading rate in WPCs is needed.

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