

Manufacturing Biocomposites Using Black Pine Bark and Oak Bark

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Bark as a biowaste has a huge availability throughout the world and has had limited use in industrial applications. Black pine bark and oak bark were considered in this work. The aim was to manufacture a new biocomposite with different combination of black pine bark, oak bark, polypropylene, polyethylene, and a coupling agent, and to determine some physical and mechanical properties of the manufactured biocomposites. Density, thickness swelling, water absorption, tension strength, modulus of rupture and modulus of elasticity in bending and tension of the biocomposites were determined. According to the results, thickness swelling and water absorption properties were improved up to 80% when compared with wood-plastic composites (WPC) produced with wood flour. Although the new biocomposites displayed lower mechanical performance in comparison of biocomposites made with wood flour, the observed results were satisfactory. Based on the results of this study, black pine bark and oak bark can be used as filler materials in WPCs production. Hereby, these bark materials can be the raw material for value-added products. Bark use in biocomposite production also can contribute to reduced requirements of wood material and petroleum products.

Keywords: Biocomposites; Bark flour; Black pine bark; Oak bark

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INTRODUCTION

Wood-plastic composites (WPCs) are typically manufactured using mixtures with various proportions of thermoplastic polymers and small wood particles. Generally, in the manufacturing of WPC products, the wood and thermoplastics are compounded above the melting temperature of the thermoplastic polymers and then processed. Wood-plastic composite products can be manufactured in a variety of colors, shapes, sizes, and surface textures. Common applications of WPCs include windows, door frames, interior panels in cars, railings, fences, landscaping timbers, cladding and siding, park benches, moldings, and furniture (Taylor *et al.* 2016).

To use a WPC product in place of another material, generally it should achieve greater performance, reduced price, or reduced environmental impact. On the other hand, there could be some disadvantages to consider when choosing WPCs, especially when replacing responsibly sourced renewable materials. Schwarzkopf and Burnard (2016) stated that including any non-renewable materials in the product can significantly increase its environmental impact. However, the case for WPCs could be different when the competing product is made entirely of non-renewable polymers. This is because the

contribution of wood materials of many WPCs can approach 50% of the product volume, and therefore such substitution can reduce the resource pressure on non-renewable materials

The environmental impact of WPCs is directly related with additive ratio of renewable to non-renewable materials in the product. WPCs have lower environmental impacts than unfilled plastics but higher than solid wood or most other wood composites. Use of sustainably harvested and recovered wood products in long-life products sequesters atmospheric carbon and can produce a positive environmental impact (Hill *et al.* 2015).

Polypropylene (PP), polyethylene (PE), and polyvinylchloride (PVC) are the most commonly used thermoplastic materials in the manufacturing of WPCs. Wood flour, wood fiber, and bio wastes are the most commonly used filling materials for WPCs.

Bark is the outer part of a tree, which acts to protect the tree stem from biotic and abiotic factors. Bark generally has a much less fibrous structure in comparison to the woody parts of a tree, and its proportion of fibers is lower than that of woods. Its morphology and chemical composition are different from wood as well (Harkin and Rowe 1971)

Bark, as a lignocellulosic residue, is often used as a thermal energy resource. However, a more productive utilization of this material would be as an alternative raw material for wood-plastic composites (Safdari *et al.* 2011; Avci 2012). There is a huge potential for wood bark but insufficient research has been done on the subject related to WPCs.

Utilization of the bark has negative effects on mechanical properties of the biocomposites according to studies in literature, but it also has some significant advantages. Bark has positive effects on water absorption and thickness swelling properties of the biocomposites. Bouafif *et al.* (2009) reported that composites made with bark particles exhibit lower water absorption compared to those made with wood particles. They stated that differences in chemical composition between bark and wood are the reason for the differences in water uptake. According to Najafi *et al.* (2008), adding MAPP had no effect on the water diffusion coefficients and K_{SR} values of composites made with higher biowaste contents. Therefore, it can be said that at higher bark contents, the compatibilizer improves the water resistance by limiting maximum water absorption. Thus, it can be inferred from the literature that bark can be an alternative raw material in composite production to improve hygroscopic properties of the products and also reduce usage content of plastic components. In addition, use of bark in biocomposite production could reduce the requirement of wood material and petroleum products such as polypropylene, polyethylene, *etc.* Beside economical contribution, it would provide protection of forest assets.

There are many factors that affect the mechanical and physical properties of composites and the amount and type of lignocellulosics are a salient factor (Bledzki *et al.* 1998). In WPCs, increasing the wood fiber loadings initially resulted in an increase in some of the mechanical properties (Bouafif *et al.* 2009; Basiji *et al.* 2010). However, with further increase of the weight percentage of the fillers to WPCs, an optimum threshold is reached, and there is no value in increasing the content of wood fibers (Lu *et al.* 2015). Thus, it seems that bark-plastic composites could meet the usual performance requirements if the uses of bark flour have an optimized content in blending with the wood flour. The bark flour can play an important role in the manufacture of thermoplastic composites and may be one of the most efficient uses of the bark.

Avci (2012) investigated the effects of wood type, plastic type, and a few combinations of these factors on physical, mechanical, and technological properties of

WPCs. As a result of the study, it was concluded that wood type has no significant effect on physical and mechanical properties of WPCs and it was recommended to use biowaste as a possible replacement of wood material.

Safdari *et al.* (2011) reported that both poplar bark flour and wood flour significantly increased mechanical properties in comparison with neat polypropylene. However, composites made with bark flour (BF) exhibited lower mechanical strength compared to those made with wood flour (WF). They reached this conclusion because of the differences in chemical composition between bark and wood; fines and low slenderness ratio of BF; poor dispersion of BF; and also the lower intrinsic fiber strength of bark fibers compared to wood fibers.

Kord (2011) investigated the effects of bark content on the mechanical properties of wood-plastic composites. According to the results of the study, the flexural strength, flexural modulus, and impact strength of the polypropylene-wood flour composites decreased with an increase in the bark flour loading. Conversely, the mechanical properties of the samples improved with the increasing coupling agent content.

Ulgur *et al.* (2013) studied wood-plastic composites produced with red pine (*Pinus brutia*) bark and cable plastic waste. The aim of the study was to create a new material with outstanding features and a wide range of applications, using recycled products to save forest resources. Three different mixture ratios were used. According to the results of the study, products between 60-mesh and 80-mesh were suitable for application.

Avci *et al.* (2014) studied biocomposites manufactured with hazelnut husks. Certain physical and mechanical properties were measured to determine the performance properties of the biocomposites. The results of the study showed that the performance properties of the biocomposites manufactured from hazelnut husks could be comparable to neat PP composites. From these results, it can be concluded that hazelnut husks could be used as an alternative sustainable raw material for the manufacturing of biocomposites.

Mathias *et al.* (2015) investigated upcycling of sunflower stems as natural fibers for biocomposite applications. They analyzed physical and chemical properties. Also, socio-economic impacts evaluated. According to results, the authors concluded that it constitutes a promising raw material for a variety of applications due to their mechanical and thermal properties, as well as to their environmental impacts.

The total acreage of Turkey contains approximately 27.6% of forest area. The total forest area is 21,700,000 ha, with 5,150,000 ha of the total amount consisting of oak trees (*Quercus* sp.), and 4,690,000 ha consisting of black pine (*Pinus nigra*) (Forestry and Water Ministry of Turkey 2012). Black pine is one of the most commonly grown species of coniferous trees, and oak is the most commonly grown species of deciduous trees in Turkey. The residues of these species have a noticeable amount of potential waste.

The aim of this study is to develop wood-plastic composites with black pine bark and oak bark that have a higher economic value compared with their use as a thermal energy source.

EXPERIMENTAL

Materials

In this study, polyethylene (PE) and polypropylene (PP) were used as the thermoplastic matrix, the bark wastes of black pine (*Pinus nigra*) and oak (*Quercus* sp.) trees were used as filling materials, maleic anhydride grafted polypropylene (MAPP) and

polyethylene (MAPE) were used as coupling agents. Both types of tree bark waste were supplied from the Black Sea region of Turkey. The density of the PP was 0.905 g/cm³, while the density of the PE was 0.919 g/cm³. The melting point was approximately 150 to 180 °C, and the melt flow index (MFI) was 47 g/10 min. The PP and PE were supplied from Petkim Petrokimya Inc., Izmir, Turkey.

Methods

Biocomposite manufacturing

The first step of the manufacturing process was drying. Both bark materials were oven-dried at a temperature of 90 °C until the moisture content had decreased to 20%. Following drying, the bark wastes were milled using a laboratory-type Wiley grinder (Wiley, Swedesboro, NJ, USA). The flour obtained was between 40- and 60-mesh. The bark flour was then dried at a temperature of 80 °C using a laboratory-type oven (Binder, Tuttlingen, Germany) until its moisture content decreased to less than 2%.

The manufacturing occurred in two steps, which consisted of extruding and injection molding of the biocomposites. The bark flour, PP, PE, MAPE, and MAPP were compounded in a laboratory-type, twin-screw extruder (Aysa, Istanbul, Turkey) at a temperature of 190 °C. The bark flour proportions of the mixtures were 10%, 30%, and 50%, while the PP and PE proportions of the mixtures were 90%, 70%, and 50%, respectively. The MAPP and MAPE loading levels were 5% for all of the composite groups. The extruded materials were cooled and granulated as pellets. The pellets were then molded using a laboratory-type injection molding machine (TSP, Istanbul, Turkey) at a temperature of 195 °C.

Testing procedure

To determine the physical properties of the biocomposites developed in this study, density, water absorption (after 1, 3, 7, and 30 days of water soaking), and thickness swelling were measured. The tension strength, elongation at break, modulus of elasticity in bending (MoE), and modulus of rupture (MoR) were also measured to evaluate the mechanical properties. The test standards used in the study are listed in Table 1.

Table 1. Tests and Standards Used

Test	Standard
Density	ASTM D792 (2013)
Thickness Swelling	ASTM D570-98 (2010); ISO 62 (2008)
Water Absorption	ASTM D570-98 (2010); ISO 62 (2008)
Modulus of Rupture	ASTM D790 (2007); ISO 178 (2010)
Modulus of Elasticity in Bending	ASTM D790 (2007); ISO 178 (2010)
Tension Strength	ASTM D638 (2007), ISO 527-1 (2012)
Modulus of Elasticity in Tension	ASTM D638 (2007), ISO 527-1 (2012)

RESULTS AND DISCUSSION

Tree bark is widely available in Turkey as a renewable natural resource. Biocomposite materials that consisted of different loading levels of bark flour were manufactured. Physical and mechanical tests were performed to determine the end-use performance. The performance properties of the biocomposites were determined based on

the bark type, plastic type, and loading level. In total, 12 bark and plastic combinations were investigated. Results of physical and mechanical performances were given following.

Physical Performance

The average density values of the biocomposite (BC) groups are listed in Table 2.

Table 2. Density Values of the Composites

Groups	Oak Bark	Black Pine Bark
Polypropylene Density (g/cm ³)		
BC ₁₀	0.928	0.918
BC ₃₀	0.989	0.957
BC ₅₀	1.010	0.988
Polyethylene Density (g/cm ³)		
BC ₁₀	0.929	0.936
BC ₃₀	0.988	0.979
BC ₅₀	1.065	1.016

The results indicated that the density values of the composites were affected by the tree bark waste content. As shown in Table 2, there was not a substantial difference between the density values of the PP for the BC₁₀ groups. The lowest density value was obtained in BC₁₀ of the black pine bark and PP group, while the highest density value was obtained in BC₅₀ of the oak bark and PE. The density values of the composites increased as the bark flour loading increased from 10% to 50%.

The thickness swelling values of the composite groups after one day (24 h), three days (72 h), seven days (168 h), and thirty days (720 h) of water immersion are given in Table 3.

Table 3. Thickness Swelling Values of the Biocomposites

		Thickness Swelling (%)			
Groups		1 Day	3 Days	7 Days	30 Days
Oak Bark	Polypropylene				
	BC ₁₀	0.112	0.179	0.229	0.408
	BC ₃₀	0.150	0.200	0.251	0.429
	BC ₅₀	0.302	0.502	0.680	0.930
	Polyethylene				
	BC ₁₀	0.136	0.215	0.237	0.288
BC ₃₀	0.194	0.280	0.384	0.464	
BC ₅₀	0.225	0.303	0.427	0.488	
Black Pine Bark	Polypropylene				
	BC ₁₀	0.106	0.168	0.263	0.426
	BC ₃₀	0.123	0.230	0.348	0.527
	BC ₅₀	0.263	0.436	0.481	0.565
	Polyethylene				
	BC ₁₀	0.132	0.200	0.258	0.315
BC ₃₀	0.147	0.226	0.384	0.441	
BC ₅₀	0.332	0.417	0.529	0.614	

As shown in the table, there was a linear proportion between increased thickness swelling values and increased water immersion time for all of the composite groups. According to the table, there were some differences between the thickness swelling

performances of the composite groups. The BC₁₀ of black pine bark and PP had the lowest 24-h (0.106%) and 72-h (0.168%) thickness swelling values. The lowest swelling values at 168 h were obtained from the BC₁₀ of oak bark and PP (0.229%). The lowest swelling values at 720 h were obtained from the BC₁₀ of oak bark and PE (0.288%). In contrast, the highest thickness swelling values at the end of 24 h were obtained from the BC₅₀ of black pine bark and PE (0.332%). At the end of 72, 168, and 720 h, the highest thickness swelling values were obtained from BC₅₀ of oak bark and PP (0.502%, 0.680%, and 0.930% respectively). When compared the results in the literature, it is seen that thickness swelling values of composites manufactured with bark flour has quite lower than wood flour composites. It is thought that, this is cause because of some of hydrophobic components in chemical composition of the bark materials.

There was a linear interaction between the thickness swelling and water absorption values of the biocomposites. The results of water absorption after 24, 72, 168, and 720 h of water immersion are given in Table 4. The results indicated that the bark flour had some minor effects on the water absorption properties of the biocomposites.

Table 4. Water Absorption Values of the Biocomposites

		Water Absorption (%)			
Groups		1 Day	3 Days	7 Days	30 Days
Oak Bark	Polypropylene				
	BC ₁₀	0.119	0.119	0.178	0.268
	BC ₃₀	0.140	0.167	0.277	0.470
	BC ₅₀	0.162	0.271	0.510	0.752
	Polyethylene				
	BC ₁₀	0.000	0.090	0.090	0.120
	BC ₃₀	0.028	0.114	0.143	0.313
BC ₅₀	0.156	0.312	0.338	0.520	
Black Pine Bark	Polypropylene				
	BC ₁₀	0.000	0.030	0.030	0.060
	BC ₃₀	0.029	0.116	0.231	0.376
	BC ₅₀	0.055	0.166	0.334	0.473
	Polyethylene				
	BC ₁₀	0.030	0.151	0.211	0.301
	BC ₃₀	0.057	0.171	0.256	0.341
BC ₅₀	0.081	0.190	0.299	0.489	

As shown in Table 4, the lowest water absorption values for all of the time schedules were obtained in the BC₁₀ composite groups, while the highest water absorption values were obtained in the biocomposites made from 50% bark flour. The highest value was obtained in the BC₅₀ composite of oak bark+PP after 720 h. It should be noted, however, that the highest value was less than 1%. Therefore, it can be concluded that this value was negligible when compared with the water absorption properties of solid wood.

The results indicated that the water absorption values of the composites increased with an increase in the water immersion time from 24 to 720 h. It can also be concluded that the water absorption values of combinations increased as the bark flour loading increased from 10% to 50%. Thus, it is clear that the level of bark flour loading has negative effect on the water absorption characteristics of the composites.

Mechanical Performance

The results of the MoR and MoE in bending tests are given in Table 5 and Table 6 respectively.

Table 5. Results of MoR in Bending

Modulus of Rupture (MoR) in Bending (N/mm ²)			
Groups		Oak Bark	Black Pine Bark
Polypropylene	BC10	52.453	58.598
	BC30	52.562	57.868
	BC50	56.454	52.244
Polyethylene	BC10	36.415	40.469
	BC30	36.649	41.703
	BC50	38.879	39.602

Table 5 shows that the lowest values for the MoR were obtained from the BC groups consisting of oak bark and PE (36 to 39 N/mm²), while the highest values were obtained from black pine bark and PP BC groups (52 to 58 N/mm²). In general, all combinations of oak bark+PE composites were yielded approximately 31% lower performance than oak bark+ PP composites. The situations of black pine bark combinations were similar. Black pine bark+PP combinations yielded higher performance (a 23% to 33% increase) than black pine bark+PE combinations. In addition, there was observed a linear proportion of increasing in performance of oak bark combinations made with both PP and PE according to bark content ratio, but the situation for the black pine bark combinations were realized opposite. It was observed that overall the performance decreased in minor values as the bark content ratio increased.

Table 6. Results of MoE in Bending

Modulus of Elasticity (MoE) in Bending (N/mm ²)			
Groups		Oak Bark	Black Pine Bark
Polypropylene	BC10	2651.537	2213.910
	BC30	2763.748	2345.685
	BC50	2795.685	2739.149
Polyethylene	BC10	1798.558	1839.020
	BC30	2396.132	2758.203
	BC50	2615.171	2883.223

According to Table 6, the lowest values for MoE in bending were obtained from BC₁₀ group of oak bark+PE combination, while the highest values were achieved from the BC₅₀ group of black pine bark+PE combination. It can be inferred from the table that increasing of bark content in composites positively affected the MoE values in bending. In general, all BC groups of oak bark+PP were performed better than black pine bark+PP groups between 2% and 17%. However, all BC groups of black pine bark+PE yielded higher performance (2% to 15% higher) when compared to oak bark+PE combinations.

The tension strength, MoE in tension, and percentage of total elongation at maximum force (percentage strain at maximum load) results of the biocomposites are given in Tables 7 and 8, respectively.

As shown in Table 7, the tensile strength values of the BC₁₀ groups were slightly higher than those of the other BC groups except for the black pine bark+PP groups. The

highest value was obtained from the BC₁₀ of oak bark+PP, while the lowest value was achieved from BC₃₀ of black pine bark+PE. The tensile strength values of all oak bark groups decreased as the bark loading level increased from 10% to 50%. On the other hand, the tension values in black pine bark groups were shown scattered distribution. In addition, for both oak bark and black pine bark, all PP groups were yielded higher performance than PE groups.

Table 7. Results for Tensile Strength

	Groups	Tensile Strength (N/mm ²)
Oak Bark	Polypropylene	
	BC ₁₀	23.219
	BC ₃₀	18.852
	BC ₅₀	15.763
	Polyethylene	
	BC ₁₀	17.679
	BC ₃₀	15.891
Black Pine Bark	Polypropylene	
	BC ₁₀	16.916
	BC ₃₀	19.712
	BC ₅₀	17.145
	Polyethylene	
	BC ₁₀	16.012
	BC ₃₀	14.092
	BC ₅₀	15.529

Table 8. Results of MoE in Tension, and Percentage of Total Elongation at Maximum Force

	Groups	Modulus of Elasticity (MoE) in Tension (N/mm ²)	Percentage of Total Elongation at Max Force (Percentage Strain at Max Load)
Oak Bark	Polypropylene		
	BC ₁₀	1656.883	4.368
	BC ₃₀	1621.337	3.316
	BC ₅₀	1646.593	2.455
	Polyethylene		
	BC ₁₀	1408.582	7.151
	BC ₃₀	1485.304	4.960
Black Pine Bark	Polypropylene		
	BC ₁₀	1300.326	3.655
	BC ₃₀	1841.614	2.658
	BC ₅₀	2027.766	1.591
	Polyethylene		
	BC ₁₀	1296.567	6.806
	BC ₃₀	1307.586	5.599
	BC ₅₀	1560.788	4.385

Table 8 shows that generally the MoE in tension was positively affected by increasing of bark flour proportion in the manufactured composites. The BC₅₀ groups were given the highest values for all combinations while the lowest values obtained from BC₁₀

combinations except oak bark+PP. In general, PP combinations yielded higher performance than PE combinations for both of the bark types.

To investigate results of total elongation, it is seen that the proportion of bark flour in the composites had a significant effect on elongation properties. The total elongation values decreased by increasing proportion of bark flour in the manufactured composites. PE combinations were given highest values than PP combinations for both of bark type.

Table 9. Results of MoR in Bending, MoE in Bending, and Tension Strength of WPCs which Manufactured with Various Type of Wood Flour (Avci 2012)

Samples		Polypropylene			Polyethylene		
		Beech	Poplar	Black Pine	Beech	Poplar	Black Pine
Groups	Content of Wood Flour						
Modulus of Rapture (MoR) in Bending (N/mm ²)	0%	61.66			37.36		
	10%	63.88	62.08	62.62	42.74	43.13	41.59
	30%	60.71	59.51	57.18	34.73	35.27	33.06
	50%	59.1	54.89	52.80	32.12	30.72	30.61
Modulus of Elasticity (MoE) in bending (N/mm ²)	0%	2011.56			1625.99		
	10%	2584.81	2601.42	2699.66	1722.95	1825.58	1934.59
	30%	3603.93	3538.78	3503.57	2183.94	2088.36	2026.45
	50%	4248.8	4710.63	4266.57	2608.5	2835.72	2780.47
Tension Strength	0%	29.81			21.24		
	10%	30.11	29.27	29.31	22.51	21.41	20.64
	30%	29.47	28.53	27.47	22.04	20.86	19.57
	50%	28.94	28.39	26.82	21.82	20.09	18.97

Table 9 shows the results of the study carried out by Avci (2012). The study investigated some mechanical properties of WPCs which are manufactured with various type of wood flour. A comparison was done between mechanical properties of this study and Table 9 because of they have similar materials and methodology. According to the table, all combinations of wood flour were given similar results. It can be inferred from table that wood type has no significant effect on mechanical properties.

When compared the average results of black pine wood flour and black pine bark flour combined with PP, it is seen that there is a decrease by 2% in MoR values and there is an increasing by 16% in PE combinations. The comparison results of MoE, it is seen that there is a decrease by 30% in PP combinations while an increased realized by 11% in PE combinations. To investigate tension strength values, it is seen that there is a significant decrease in both PP and PE combinations by 36% and 23% respectively. In general, it can be said that a decrease realized on mechanical properties of composites manufactured by bark flour.

CONCLUSIONS

The results of this study are in general agreement with previous literature regarding the subject of biocomposites in WPCs. According to the results, the following conclusions were obtained.

1. The loading level of both tree bark types linearly affected the density values of the biocomposites; however, there was no significant effect of bark type. Density generally increased with increasing bark loading.
2. The loading of bark flour affected the thickness swelling and water absorption values of the resulting composites. Thus, it can be inferred that there was a linear interaction between the bark flour loading level and both thickness swelling and water absorption value. Thickness swelling generally increased with bark loading and with the time of exposure to water.
3. The thickness swelling and water absorption values decreased up to 80% when compared with composites manufactured with wood flour. Thus, it can be recommended to use the composites manufactured with bark flour for constructions that require higher water resistance.
4. The modulus properties can be improved by the increasing of bark flour proportion.
5. Generally, the composites made with PP resulted in higher values of MoR than those made with PE. This factor can be considered in the design process of a product.
6. It can be understood from the results that there is a linear proportionality between the loading level and MoE of the materials.
7. Based on the results, all manufactured composite groups showed an irregular behavior with respect to tensile strength. Thus, further studies are requested to find out more details about this subject.
8. It can be concluded that black pine bark and oak bark can be used as alternative raw materials as filler in WPC production. Therefore, using these bark materials as biowaste can be recycled to value-added products.
9. Overall, mechanical properties of the WPCs made of bark flour yielded lower performance when compared with WPCs made of wood flour.
10. The use of bark residues in biocomposite production could reduce the requirement of wood material and petroleum products such as polypropylene, polyethylene, *etc.* In this manner, besides a positive economical contribution, these materials would save forest assets.

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