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Evaluation of the physical, mechanical properties and formaldehyde emission of particleboard manufactured from waste stone pine (*Pinus pinea* L.) cones

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ABSTRACT

The objective of this study was to investigate some physical/mechanical properties and formaldehyde emission of particleboard containing particles of waste stone pine cone at various usage ratios using urea–formaldehyde resin. Some physical (thickness swelling, water absorption), mechanical (modulus of elasticity, modulus of rupture, internal bond strength) properties and formaldehyde emission of particleboards were evaluated. The addition of cone particle improved water resistance of the panels and greatly reduced their formaldehyde emissions. However, flexural properties and internal bond strength decreased with increasing cone particle content in the panel. The cone of the stone pine can be considered as an alternative to wood material in the manufacture of particleboard used in indoor environment due to lower thickness swelling, water absorption and formaldehyde emission.

1. Introduction

The raw material demand of the forest products industry increases annually. Recently, approximately 70% of raw material requirement for the Turkish particleboard industry is supplied from Canada, Russia, and Ukraine because of decreasing availability of raw material in Turkey (Ayrilmis et al., submitted for publication). There is still an outgoing research interest to find alternative sources of raw materials for composite manufacture (Guntekin et al., 2008). Therefore, alternative raw materials such as agricultural residues will play an important role in the forest products industry (Nemli et al., 2009; Bektas et al., 2005).

Many researchers have investigated properties of agro-based particleboards made from wide variety of agricultural residues: hazelnut husk (Guler et al., 2009), eggplant stalks (Guntekin and Karakus, 2008), peanut hull (Guler et al., 2008), wheat straws and corn pith (Wang and Sun, 2002), cob and maize husk (Sampathrajan et al., 1992), waste of tea leaves (Yalinkilic et al., 1998), sunflower stalks (Bektas et al., 2005), kiwi pruning (Nemli et al., 2003), grass clipping (Nemli et al., 2009), pine needle litter (Nemli and Aydin, 2007; Nemli et al., 2008) and pepper stalks (Guntekin et al., 2008).

The total area covered by stone pine woodlands is 380,000 ha (75% in Spain, 9% in Portugal, 9% in Turkey, 5% in Italy, and lower percentages in Greece, Lebanon and France) (Moussouris and Regato, 1999). The stone pine forests of Turkey cover 54,000 ha and total cone production of the stone pine is approximately 3500 tons in 2006 according to Forestry Statistics of Turkish General Directorate of Forestry (Ayrilmis et al., submitted for publication). The pine cone, a renewable resource, is not used effectively. Large quantities of cones are produced annually throughout the world, especially in pine plantations grown for the pulp and paper industry.

Pinus pinea cones are the most significant value among the pinus species because the pine nut (pinyon), which is the edible seed of the pine, is a widely used and highly appreciated food source. They are collected, dried to facilitate seed release, and generally discarded or burned in the stove in winter. They don't require any additional cost for collecting and drying. For this reason, waste cone is a significant potential for wood based panel industry in Mediterranean countries having stone pine forests. New uses for pine cones could provide additional income for forest landowners (Ayrilmis et al., submitted for publication). The pine cones contain large quantities of glucose, probably derived from cellulose, and smaller quantities of mannose, galactose, and xylose, probably derived from hemicellulose (Petterson, 1984). They also contain significant amounts of lignin and ethanol/toluene extractives (Gonultas, 2008).

Formaldehyde is considered a dangerous substance, and its concentration in indoor environments is restricted in many countries because of its reactivity, toxicity, and pungent odor (Wang et al.,

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2008; Nemli and Ozturk, 2006). Formaldehyde-based resins are used extensively as the adhesives in manufacturing various household products such as particleboard, plywood, and fiberboard. With the rising economic standards, concerns about human health and the environment have been raised due to the increasing demand for wood based panels (Kim, 2009). Nemli and Colakoglu (2005) found that the addition of mimosa bark particle greatly reduced formaldehyde emission of particleboards.

Ayrilmis et al. (submitted for publication) investigated the use of pine cone flour in MDF production. They found that depending on the addition amount of cone flour in MDF panels, formaldehyde emission values ranged from 2.6% to 55.3% lower than produced only industrial wood fibers. Using of pine cone in production of particleboard not only can decrease wood demand but also reduce formaldehyde emission. Up to date, there is no information on using of the stone pine cone as an alternative to wood material in manufacture of particleboard. The objective of this study was to investigate some physical/mechanical properties and formaldehyde emission of particleboard panels containing cone particles of stone pine at various usage ratios using urea-formaldehyde resin. Our secondary objective was to find a solution increasing raw material requirement for the particleboard industry in Turkey.

2. Methods

Wood particles (a 50:50 blend) consisting of pine (*Pinus nigra Arnold var. pallasiana*) and beech (*Fagus orientalis* Lipsky) species were obtained from a commercial particleboard plant in Gebze, Turkey. The fresh cones were collected from stone pine (*Pinus pinea* L.) trees in the Fatih Forest District in Belgrade Forest, Istanbul, Turkey. The cones were placed on a plastic cover under the sun. After drying, the cones opened up and the nuts fell out. The pine cones without nuts were soaked in hot water for four hours at 80 °C. This treatment was believed to partially remove gum on the surface of the cones and improve the grinding process and bonding properties of cone particles. After this treatment, the wet pine cones were dried in an oven up to 20–25% moisture content (based on the oven-dry cone weight) at 60 °C. The pine cones were coarsely chipped and then were classified using a horizontal screen shaker. The particles that remained between 3–1.5 mm sieves and between 1.5–0.8 mm sieves were utilized in the core and middle sections of the panels, respectively. The particles used in particleboard production were dried at 100–110 °C in an oven to reach target moisture content (3%).

The panels were designed to consist of 35% chips at the face layer and 65% at the core layer and the target density of the panels was 0.65 g/cm^3. The boards were pressed under 2.6 N/mm^2 pressure, at 150 °C, for 7 min. The experimental design is shown in Table 1. Two experimental particleboards having dimensions of 55 × 55 × 1 cm were produced for each design and then the produced particleboards were conditioned at 20 ± 2 °C and 65 ± 5% relative humidity to the moisture content of about 12%. Edges of the boards were then trimmed to the final dimension of 50 × 50 × 1 cm. Urea-formaldehyde (UF) resin at 10% adhesive level was used for the core and outer layers based on oven-dry weight. One-percent ammonium chloride (NH_4CL) was added to resin as a hardener. The chips were placed in a drum blender and sprayed with urea formaldehyde and ammonium chloride for 5 min to obtain a homogenized mixture. This study did not include the addition of any external wax or water-repellent chemicals to the wood and cone particles. Besides, the cone particle ratios based on the oven-dry weight of particles, other process options, such as particle properties, resin type (UF), percentage of UF resin, and press parameters, were unchanged in all experimental panels.

Table 1
Experimental design.

Board type	Raw material	
	Pine cone (%)	Wood (%)
A	0	100
B	10	90
C	20	80
D	30	70
E	40	60
F	50	50

Some physical; water absorption (WA) and thickness swelling (TS) (EN 317, 1993) and mechanical; modulus of rupture (MOR) (EN 310, 1993), modulus of elasticity (MOE) (EN 310, 1993) and internal bond (IB) (EN 319, 1993) strength properties were determined for the produced particleboards. Average of 10 and 20 measurements was reported for mechanical and physical properties, respectively. Twenty samples were randomly taken from each type of the panel for formaldehyde emission determination using the perforator method based on the EN 120-1 (1994) standard. The obtained data were statistically analyzed by using the analysis of variance (ANOVA) and Duncan's mean separation tests.

3. Results and discussion

The results of ANOVA and Duncan's mean separation tests for WA and TS of particleboards made using the mixtures of pine cone and wood chips for 24 h water immersion times are shown in Table 2. With the increasing of the pine cone usage in the particleboard panels, the TS and WA values decreased from 19.2% to 13.9% and 70.4% to 36.4%, respectively. These results may be attributed to the presence of more amounts of extractives in the pine cone. Gonultas (2008) found that stone pine cone contained higher extractives than its wood. The positive effect of extractives on the resistance to the water has been mentioned by several researchers (Pasillias and Voulgaridis, 1999; Nemli et al., 2004a,b; Nemli and Aydin, 2007). Similarly, Nemli and Colakoglu (2005) and Nemli et al. (2008) found that mimosa bark and pine litter usage significantly decreased TS of particleboards.

Statistical analysis found some significant differences ($p < 0.01$) between some group means for TS and WA values. Significant differences between groups were determined individually for these tests by Duncan's multiple-comparison tests. The results of Duncan's multiple range tests are shown in Table 2 by letters. The average TS value of panel type C did not show any significant difference with panel type D but significant differences were found with other panel types. The average TS and WA values of particleboard with cone particles decreased from 5.0% to 27.5% and 11.6% to 48.2% as compared to values of the panels made from 100% wood particles, respectively (Fig. 1).

TS value of the particleboard containing 50% pine cone particles was found to comply with particleboard maximum property requirement of 14% for based on the EN 312 Type P3 (2005) for use in non load-bearing applications in humid conditions. The other particleboard types did not meet maximum TS requirement of the EN 312 Standard. These high values may be related to the fact that no wax or other hydrophobic substance was used during particleboard manufacture. Heat-treatment, adding water-repellent chemicals such as paraffin, use of phenolic resins, coating of the particleboard surfaces and acetylating of particles can improve the water repellency of the panels (Nemli et al., 2005; Ayrilmis et al., submitted for publication; Guntekin et al., 2008; Rowell and Norimoto, 1988).

Table 3 shows the results of mechanical properties for produced particleboard. The highest MOR (16.07 N/mm^2) and MOE (2068.6 N/mm^2) values were measured for particleboard produced using industrial wood particles. On the other hand, the lowest MOR

Table 2
Thickness swelling (TS) and water absorption (WA) test results of ANOVA and Duncan's mean separation tests of particleboards produced from pine cone and wood particles.

Physical properties	Board type	Soaking time (hour)	Mean (%)^{a}	Standard deviation	Standard error	X_{Min}^{b}	X_{Max}^{c}	p^{d}
Thickness swelling (TS)	A	24	19.18^{p}	0.92	0.21	17.61	20.78	•
	B	24	18.21^{s}	0.94	0.21	16.66	19.50	•
	C	24	17.30^{u}	1.12	0.25	15.47	19.15	•
	D	24	16.80^{u}	1.07	0.24	14.86	18.76	•
	E	24	14.75^{v}	0.95	0.21	13.39	16.30	•
	F	24	13.91^{y}	0.82	0.18	12.25	15.24	•
Water absorption (WA)	A	24	70.36^{p}	1.03	0.23	68.71	72.30	•
	B	24	62.17^{s}	1.10	0.25	60.16	64.51	•
	C	24	56.00^{u}	1.27	0.28	53.61	58.54	•
	D	24	53.22^{v}	1.37	0.31	51.16	55.31	•
	E	24	42.15^{y}	1.39	0.31	40.33	44.46	•
	F	24	36.42^{z}	1.04	0.23	34.46	38.04	•

^{a} Mean values are the average of 20 specimens.
^{b} Minimum value.
^{c} Maximum value.
^{d} Significance level of 0.01 (for ANOVA).
^{p,s,u,v,y,z} Values having the same letter are not significantly different (Duncan test).

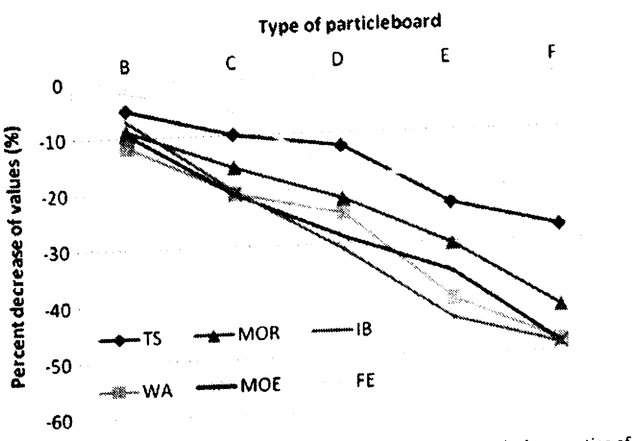

Fig. 1. Percent decreases in average values of physical and mechanical properties of the panel types.

(9.34 N/mm^2) and MOE (1064.6 N/mm^2) values were obtained for F type panels including 50% pine cone particles. The results indicated that the increasing of the pine cone content in the mixture significantly decreased the MOR and MOE values of the particleboards. All panel types showed statistically significant differences ($p < 0.01$) in their MOR and MOE properties from each other.

Depending on the amount of cone particles in the panels, average MOR values ranged from 8.7% to 41.9% lower than the average of the panel type A (Fig. 1). MOE values showed similar trends to results of the MOR. The average MOE values of the particleboard with cone particles decreased from 9.2% to 48.5% as compared to values of the panels made from 100% wood particles (Fig. 1). Addition of the cone particles into particleboard decreased the MOE values more than the MOR.

Panels A, B, C and D satisfied the minimum MOR requirements for general purpose use and interior fitments including furniture manufacture stated in the EN Standard (EN 312, 2005). Average MOE values of the panel types A and B met the minimum

Table 3
The mechanical properties of particleboards made from pine cone and wood particles and the test results of ANOVA and Duncan's mean separation tests.

Mechanical properties	Board type	Mean^{a}	Standard deviation	Standard error	X_{Min}^{b}	X_{Max}^{c}	p^{d}
MOR (N/mm^2)	A	16.07^{p}	0.78	0.248	14.51	16.87	•
	B	14.66^{s}	0.81	0.257	13.25	15.80	•
	C	13.58^{u}	0.62	0.195	12.46	14.41	•
	D	12.57^{v}	0.46	0.145	11.68	13.39	•
	E	11.21^{y}	0.85	0.268	9.68	12.49	•
	F	9.34^{z}	0.65	0.206	8.55	10.48	•
MOE (N/mm^2)	A	2068.6^{p}	83.32	26.35	1938.6	2194.1	•
	B	1879.3^{s}	62.83	19.87	1786.2	1960.5	•
	C	1644.2^{u}	53.43	16.89	1538.1	1729.2	•
	D	1474.4^{v}	66.92	21.16	1386.6	1567.5	•
	E	1337.9^{y}	91.72	29.00	1207.5	1466.7	•
	F	1064.6^{z}	83.35	26.36	931.7	1245.3	•
IB (N/mm^2)	A	0.568^{p}	0.015	0.005	0.542	0.593	•
	B	0.528^{ps}	0.029	0.009	0.488	0.569	•
	C	0.455^{u}	0.023	0.007	0.416	0.487	•
	D	0.392^{v}	0.020	0.006	0.359	0.423	•
	E	0.320^{y}	0.023	0.007	0.280	0.347	•
	F	0.292^{z}	0.020	0.006	0.257	0.323	•

^{a} Mean values are the average of 10 specimens.
^{b} Minimum value.
^{c} Maximum value.
^{d} Significance level.
• Significant at 0.01 for ANOVA.
^{p,s,u,v,y,z} Values having the same letter are not significantly different (Duncan test).

requirement for interior fitments including furniture manufacture stated in the EN 312 Standard.

IB values of the experimental panels ranged from 0.29 to 0.57 N/mm^2. The highest IB value was observed for A type panel while the lowest was observed for F type panel. IB values decreased with the increasing of pine cone particle content in the panels. The pine cone particle usage decreased IB values of the panels (6.9% to 48.6%) as compared to values of the panels made from 100% wood particles. All panel types showed statistically significant differences ($p < 0.01$) in IB values from each other. All of the produced panels met the IB requirement for general purpose end-use while A, B and C type particleboards met the minimum requirement for interior fitments including furniture manufacture stated in the EN 312 Standard.

Stone pine cone contains lower holocellulose (hemicelluloses and cellulose) content and higher extractives than its wood. Amount of holocellulose and lignin in the stone pine cone has been found as 67.6% and 37.2% based on the weight of extracted wood, respectively (Gonultas, 2008). Stone pine cone contains significant amounts of ethanol/toluene extractives (29.2%) as compared to contents of such substances, 5.1% for sapwood and 22.6% for heartwood, in the stone pine wood. Decreasing of mechanical properties of the particleboard panels with increasing pine cone particle can be attributed to higher contents of extractives in the cone than the wood. Extractives can have adverse effects on the setting of adhesives, thereby towering the particle–particle bond strength and may cause blows and severely reduce the internal bond strength (Moslemi, 1974). In addition, both ethanol- and water-soluble extractives have a significant effect on the UF resin gel time which plays major roles in determining adhesive bond-quality. The negative effect of the extractives on the mechanical properties of particleboard has been mentioned by several researchers (Nemli and Colakoglu, 2005; Nemli and Aydin, 2007; Nemli et al., 2004a,b).

Similarly, lower mechanical properties have been reported for the particleboards made using agricultural residues (Ayrilmis et al., submitted for publication; Nemli et al., 2008; Nemli et al., 2009; Bektas et al., 2005). Coating of the particleboard surfaces and use of phenolic resins can improve mechanical properties of the panels (Nemli, 2003; Nemli et al., 2005; Chow et al., 1996; Lee and Kim, 1985).

The addition of pine cone particle into particleboard significantly decreased formaldehyde emission value. Type F panels had the lowest formaldehyde emission value with 1.99 mg/100 g (19.76% of type A panel), followed by type E panels with 2.04 mg/100 g (17.74%), type D panels with 2.05 mg/100 g (17.34%), type C panels with 2.33 mg/100 g (6.05%), type B panels with 2.43 mg/100 g (2.02%), and type A panel with 2.48 mg/100 g, respectively (Fig. 2). Depending on the addition amount of cone flour in the panels, formaldehyde emission values ranged from 2.02% to 19.76% lower than panel type A (Fig. 2).

Decrease in the formaldehyde emission of the panels containing the cone particles was attributed to high amount of phenolic extractives of the stone pine cones. Nemli and Colakoglu (2005) found that formaldehyde emission of particleboards containing 50% mimosa bark particles decreased from 1.48 mg/100 g to 0.66 mg/100 g. They reported that decrease in formaldehyde emission values in may be due to the high amounts of polyphenolic extractives in bark, especially tannin. Total phenol and condensed tannin contents of the stone pine cone was found as 28.6 mg/g and 14.5 mg/g while there was no hydrolyzable tannins such as gallo and ellagtannin were not determined in the cone (Gonultas, 2008). The condensed tannin, due to its phenolic nature, can react with formaldehyde in the adhesive, even at normal temperatures. This results in lower formaldehyde emission from the panels containing the cone particles. Decreasing of formaldehyde emission

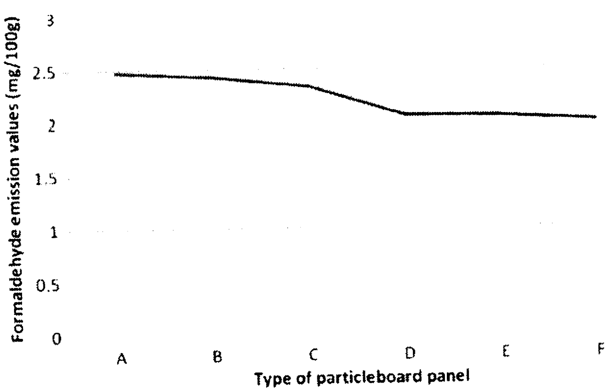

Fig. 2. The variations in the formaldehyde emission values depending on the type of particleboard.

from particleboard containing the cone particles is considerable for furniture materials used in the indoor environment.

4. Conclusions

This study investigated the feasibility of using pine cone in the manufacture of three-layer particleboard. The results show that it is possible to produce particleboards using mixture of pine cone and wood particles while using urea-formaldehyde as an adhesive. The addition of cone particle improved water resistance of panels and greatly reduced their formaldehyde emission. However, the mechanical properties decreased with the increasing of the cone particle content in the panels. Coating of the particleboard surfaces and use of phenolic resins can improve the mechanical properties of the panels. We recommend that the cone of the stone pine can be considered as an alternative to wood material in the manufacture of particleboard used in indoor environment due to lower thickness swelling, water absorption and formaldehyde emission.

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