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Properties of fiberboards produced from kermes oak (*Quercus coccifera* L.) and brutian pine (*Pinus brutia* Ten.) woods

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Abstract: In this study the utilization of kermes oak (*Quercus coccifera* L.) wood, which could be used as an industrial raw material for the production of fiberboard, was investigated. In the experimental design, boards were produced from mixtures of kermes oak and brutian pine (*Pinus brutia* Ten.) fibers in ratios of 0:100, 25:75, 50:50, 75:25, and 100:0. The chemical and thermal characteristics of the fibers used were determined, and the effects of these characteristics on the physical and mechanical properties of the produced fiberboards were revealed. The main chemical component analysis indicated that kermes oak fibers contained higher quantities of extractives, α-cellulose, and hemicelluloses and a lower quantity of lignin compared to the brutian pine fibers. Analyses of the main chemical components were supported by Fourier transform infrared spectroscopic analyses and gas chromatographic determination of monosaccharides. The pH determination showed that kermes oak fibers were more acidic than the brutian pine fibers. Thermogravimetric analysis indicated that kermes oak fibers had higher thermal stability than brutian pine fibers. An increase in kermes oak in the fiber mixture raised water absorption and thickness swelling (TS) but reduced the modulus of elasticity (MOE), modulus of rupture (MOR), and internal bond (IB) strength of the produced boards. The boards produced with 25% kermes oak fibers met the requirements of standard TS-EN 622-5 in terms of TS, MOE, MOR, and IB strength for load-bearing applications in dry conditions.

Key words: Brutian pine, chemical composition, kermes oak, physical and mechanical properties, thermal stability

1. Introduction

Medium fiberboard density (MDF) is one of the fastest increasing composite panel products on the market among forest products (Ayrılmış, 2007, 2008; Çöpür et al., 2008). The physical and mechanical properties of MDF depend primarily on the characteristics of the raw materials (wood, binders, and other additives) and parameters of production (Akbulut et al., 2004). Wood fiber properties including fiber structure and strength, anatomical and chemical properties of fiber, and fiber composition (percentages of whole and broken fibers and fines) are considered to be basic characteristics influencing fiberboard properties because they occupy a large proportion of the total volume of panels (Maloney, 1977; Suchsland and Woodson, 1991; Groom et al., 1999).

In Turkey the General Directorate of Forestry (OGM) produced 20–20.5 million m^3 of wood from forest areas; 16.6 million m^3 of this yield is used annually as industrial raw material, but 21–22 million m^3 of wood is consumed yearly by industry. Particle and fiberboard production used 6.9 million m^3 of the 16.6 million m^3 of industrial wood produced by the OGM, and further wood requirements are met by importation (OGM, 2016).

Maquis plants cover an area of 4.23 million ha and constitute a significant portion of the Mediterranean forestlands of Turkey (Evrendilek and Doygun, 2000). Kermes oak (*Quercus coccifera* L.) is an evergreen hardwood species that is usually in brush form, especially in the Mediterranean flora (Akman, 1995; Kaya and Aladağ, 2009).

Oak species occupy fourth place, representing 8% of the Turkish forest areas after pine (63%), beech (14%), and fir (9%) species (OGM, 2016). Fibers generated from low-quality oak, beech, and pine are used either alone or in combination for MDF production in Turkey (Akbulut et al., 2000). Although it has very high potential due to its biomass, the use of kermes oak as a raw material has not been observed in any field of the industry. Kermes oak wood, which occurs in high quantities in the large area occupied by maquis plants (4.23 million ha), could help to overcome the shortage of raw material for wood industry products, such as MDF.

Some studies in the literature proposed that the wood of evergreen maquis species in the Mediterranean flora could be used as a raw material in the production of

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composite panels (Lacroix, 1973a, 1973b; Tsoumis et al., 1988; Barboutis and Philippou, 2007; Lykidis et al., 2014; Güler and Yaşar, 2018).

Other *Quercus* species were investigated as raw material for fiberboard production (Chow, 1979; Kartal and Green, 2003; Akgül et al., 2007, 2010, 2013; Baharoğlu et al., 2013); therefore, kermes oak may have potential as a raw material for fiberboard production.

In our previous work, we investigated kermes oak wood as a raw material for particleboard production after mixing with brutian pine (*Pinus brutia* Ten.) wood and determined that kermes oak particles added up to 50% will produce particleboard that meets the requirements for general-purpose particleboard used in dry conditions according to standard TS-EN 312 (Güler and Yaşar, 2018).

In this study, the wood of kermes oak, which is a Mediterranean evergreen hardwood species growing in Turkey, was studied as a possible fiberboard raw material. Since brutian pine (*Pinus brutia* Ten.) is one of the most important sources of raw material used in the production of fiberboard in Turkey (Özdemir and Uçar, 2016), boards were produced by mixing kermes oak and brutian pine fibers at different ratios (0:100, 25:75, 50:50, 75:25, and 100:0). The chemical and thermal characteristics of the species used were determined, and their effects on the physical and mechanical properties of the produced fiberboards were investigated.

2. Materials and methods

2.1. Materials

The trunk materials from kermes oak between 5 and 10 cm in diameter were collected from Söbü, Isparta province, in Turkey in the first week of October 2016. After debarking, the trunks were chipped with a hammer mill. Before refining, particles of the trunks were steamed at 125 °C for 10 min and then converted to fibers using a stone mechanical refiner. The kermes oak fibers obtained were laid out and air-dried for 30 days. The brutian pine fibers were obtained from AGT A.S. (Antalya, Turkey). Properties of the kermes oak and brutian pine fibers used are shown in Table 1.

The adhesive (urea formaldehyde (UF) resin) and hardener (ammonium chloride) were from ORMA A.S. (Isparta, Turkey). Properties of the UF resin are shown in Table 2.

2.2. Methods

2.2.1. Chemical analyses

The pH values of the kermes oak and brutian pine fibers were determined according to Johns and Niazi (1980). To determine the extractive content of samples, the fibers were subjected to extraction with cyclohexane:ethanol (2:1, v/v) in a Soxhlet apparatus for 6 h followed by ethanol. The extractive content was expressed as the percentage of oven-dried material. Holocellulose was obtained from the samples extracted with cyclohexane and ethanol according to the method of Wise and Karl (1962), and α-cellulose and hemicellulose contents were determined from holocellulose according to ASTM D 1103 (1980). The α-cellulose and hemicellulose contents were calculated as the percentage of oven-dried material. The acid hydrolysis method developed by Dill et al. (1984) was applied with slight modification. The fiber samples extracted with cyclohexane and ethanol were first hydrolyzed in 20 mL of 72% H_2SO_4 at 30 °C for 2 h. After dilution with distilled water to 360 mL, the samples were hydrolyzed in a JP-Selecta autoclave at 120 °C for 30 min (Yaşar et al., 2010). After filtering, Klason lignin was dried at 105 ± 2 °C and calculated as the percentage of oven-dried material. The monosaccharides obtained in the acid hydrolysate were analyzed in a PerkinElmer Autosystem XL gas chromatograph according to the method developed by Cao et al. (1997). Fourier transform infrared (FTIR) spectra of the pellets obtained by pressing 10 mg of fiber samples with 1000 mg of KBr and recorded by PerkinElmer BX FTIR spectrometer at room temperature in the range of 4000 to 400 cm^{-1}. Thermogravimetric analysis (TGA) of 5 mg of fiber samples was performed in a PerkinElmer SII Diamond thermal analyzer in the temperature range

Table 1. Fiber characteristics of kermes oak and brutian pine.

Fiber characteristics*	Kermes oak		Brutian pine	
	Fiber length (mm)	Fiber diameter (μm)	Fiber length (mm)	Fiber diameter (μm)
Minimum	1.20	7.63	3.56	5.59
Maximum	10.12	123.41	16.57	55.19
Mean	4.12	51.95	7.44	27.77
Standard Deviation	2.24	30.80	3.25	10.70

*Obtained using Nikon SMZ745T microscope.

Table 2. Properties of urea formaldehyde (UF) resin used.

Properties	UF resin
Solid content (%)	65 ± 1
Density (g/cm^3)	1.27–1.29
pH (25 °C)	7.5–8.5
Viscosity (cps, 25 °C)	150–200
Gel time (s, 100 °C)	25–30
Storage time (day, at 25 °C)	60
Flowing time (s, 25 °C)	20–30
Free formaldehyde (max.) (%)	0.19

of 25 °C to 800 °C and a heating rate of 10 °C/min under nitrogen.

2.2.2. Fiberboard production and physical and mechanical tests

Kermes oak and brutian pine fibers were dried to a 3% moisture content at 102 ± 5 °C before board production. The experimental design of the fiberboards is presented in Table 3. Fibers were weighed to achieve the 0.75 g/cm^3 target density of the boards. The boards were prepared by spraying the adhesive on the fibers in a drum blender with an air-atomized metered system. Based on the oven-dried fiber weight, 10% UF resin was applied as adhesive and 1% NH_4Cl at a concentration of 35% was used as a hardener. After adhesion the fibers were formed into a mat on a metal plate using a 31 × 35 cm wooden box. The manually formed mats were pressed for 8 min at 150 ± 5 °C under 2.5–3 N/mm^2 pressure to achieve a board thickness of 1.2 cm. The produced fiberboards were stored in a climate room at 20 °C and 65% humidity for 30 days for conditioning.

The modulus of elasticity (MOE), the modulus of rupture (MOR), internal bond strength (IB), thickness swelling (TS), and water absorption (WA) of the fiberboards were determined according to standards TS-EN 310 (1999), TS-EN 317 (1999), and TS-EN 319 (1999).

2.2.3. Statistical analysis

Statistical analyses were performed using Minitab 16 software. First, analysis of variance (ANOVA) was applied for the physical and mechanical tests. When a significant difference was found between the mean values of the samples in the ANOVA test, Duncan's test was applied to determine the different groups.

3. Results and discussion

3.1. Main chemical component composition and pH values

The main chemical component composition of the kermes oak fibers was compatible with typical hardwood species, whereas composition of the brutian pine fibers was in agreement with softwood species, as reported in the literature (Fengel and Wegener, 1984) (Table 4). The findings for the main chemical component composition of kermes oak fibers were supported by the findings of Yaşar and Kılınç (2018) and Güler and Yaşar (2018), who reported the main chemical component composition of kermes oak wood. The findings in brutian pine fibers for main chemical component composition were compatible with the findings of Kılıç et al. (2010), who published the main chemical component composition of brutian pine. The extractive α-cellulose and hemicellulose contents of kermes oak fibers were higher than those of brutian pine fibers, while the lignin content was lower in kermes oak fibers than in brutian pine fibers. The pH value of brutian pine fibers was consistent with the findings of Taş and Sevinçli (2015), who reported a pH value of 4.83 for brutian pine wood. The pH value of kermes oak fibers was very low. Similarly, woods of oak species are known to be very acidic (Balaban and Uçar, 2001).

Table 3. Experimental design of fiberboards.

Board type	Kermes oak (%)	Brutian pine (%)
A	0	100
B	25	75
C	50	50
D	75	25
E	100	0

3.2. Monosaccharide composition

Gas chromatographic analyses showed that the monosaccharides in kermes oak fibers were rhamnose, arabinose, xylose, mannose, galactose, and glucose; they were arabinose, xylose, mannose, galactose, and glucose in brutian pine fibers (Figure 1).

Table 4. Main chemical components and pH values of kermes oak and brutian pine fibers.

	Kermes oak (%)	Brutian pine (%)
Extractives	5.26 (0.28)[1]	4.48 (0.15)
Lignin	22.25 (0.27)	27.34 (0.41)
α-Cellulose	51.95 (0.55)	48.24 (0.45)
Hemicelluloses	25.29 (0.43)	23.85 (0.49)
pH	3.84 (0.03)	4.94 (0.02)

[1] Standard deviation.

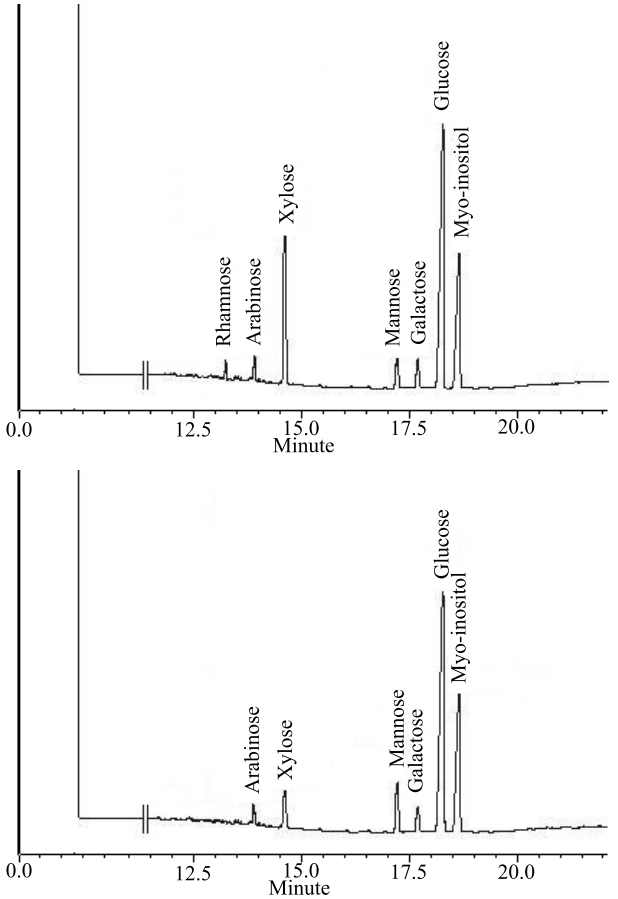

Figure 1. GC chromatograms of kermes oak (a) and brutian pine (b) fibers.

The monosaccharide composition of kermes oak was consistent with observations by Yaşar and Kılınç (2018) and Yaşar and Güler (2018), while the composition of brutian pine was in agreement with findings by Yaşar (2014) and Yaşar and Güler (2018). Glucose is available not only in the cellulose but also in the mannan of hardwoods and softwoods. The reported mannose:glucose ratio in the mannan of hardwoods was 1.5–2:1, while it was 3:1 in the mannan of softwoods (Fengel and Wegener, 1984). Therefore, the maximum glucose content in the mannan of kermes oak fibers will be 1.03%, and the maximum in the mannan of brutian pine fibers is 3.57%. The rest of the glucose content, which is related to cellulose, is 49.38% in kermes oak fibers and 42.98% in brutian pine fibers. The hemicelluloses arise from rhamnose, arabinose, xylose, mannose, galactose, and the glucose in the mannan of kermes oak fibers with a content of 26.61%. In brutian pine fibers hemicelluloses arise from arabinose, xylose, mannose, galactose, and the glucose of the mannan with a content of 25.98% (Table 5). The findings here indicate that the content of cellulose and hemicellulose in kermes oak fibers is higher than the content in brutian pine fibers. The monosaccharide compositions of kermes oak and brutian pine fibers are compatible with findings from α-cellulose and hemicellulose analyses in the study.

Table 5. Monosaccharide composition of kermes oak and brutian pine fibers.

Monosaccharide	Kermes oak (%)	Brutian pine (%)
Glucose	50.41 (0.02)[1]	46.55 (0.04)
Mannose	2.05 (0.03)	10.70 (0.05)
Xylose	21.27 (0.02)	7.85 (0.03)
Galactose	1.04 (0.03)	2.15 (0.03)
Arabinose	0.86 (0.02)	1.71 (0.01)
Rhamnose	0.36 (0.02)	-

[1] Standard deviation.

3.3. Fourier transform infrared spectra

The FTIR spectra of kermes oak and brutian pine fibers are shown in Figure 2. For evaluating cellulose, hemicellulose, and lignin in the samples, the bands at 3460, 1750, 1512, 1384, and 902 cm^{-1} were used. The band at 2942 cm^{-1} was established as an internal standard (Sinha and Rout, 2009; Mahato et al., 2014). The absorbance values of the abovementioned bands were divided into the absorbance value of the band at 2942 cm^{-1}, and the FTIR spectra of kermes oak and brutian pine fibers were compared (Table 6). The band at 2942 cm^{-1} is assigned to C-H stretching in methyl and methylene groups (Sinha and Rout, 2009; Mahato et al., 2014). The band at 3460 cm^{-1} is attributed to H-bonded H-O stretching (Luna et al., 2012). The fact that the absorbance value was higher in the kermes oak samples indicates that kermes oak fibers contained a higher content of –OH groups than the brutian pine fibers. The band at 1750 cm^{-1} represents the C-O stretching of the carboxyl and acetyl groups of hemicellulose in the samples (Luna et al., 2012). The absorbance value of this band was higher in kermes oak fibers than in the brutian pine fibers. This result reveals that the hemicellulose content in kermes oak fibers was higher than in brutian pine fibers. The band at 1512 cm^{-1} was related to the aromatic skeleton vibration in the lignin (Li et al., 2010; Luna et al., 2012). The fact that this band had a lower absorbance value in kermes oak fibers than in brutian pine fibers shows that kermes oak contained lower lignin content than brutian pine. The band at 1384 cm^{-1} represents the C-H deformation in cellulose and hemicellulose (Li et al., 2010). The absorbance value at this band was higher in kermes oak fibers than in brutian pine fibers. This shows that the polysaccharide content is higher in kermes oak than in brutian pine. The band at 902 cm^{-1} is related to the

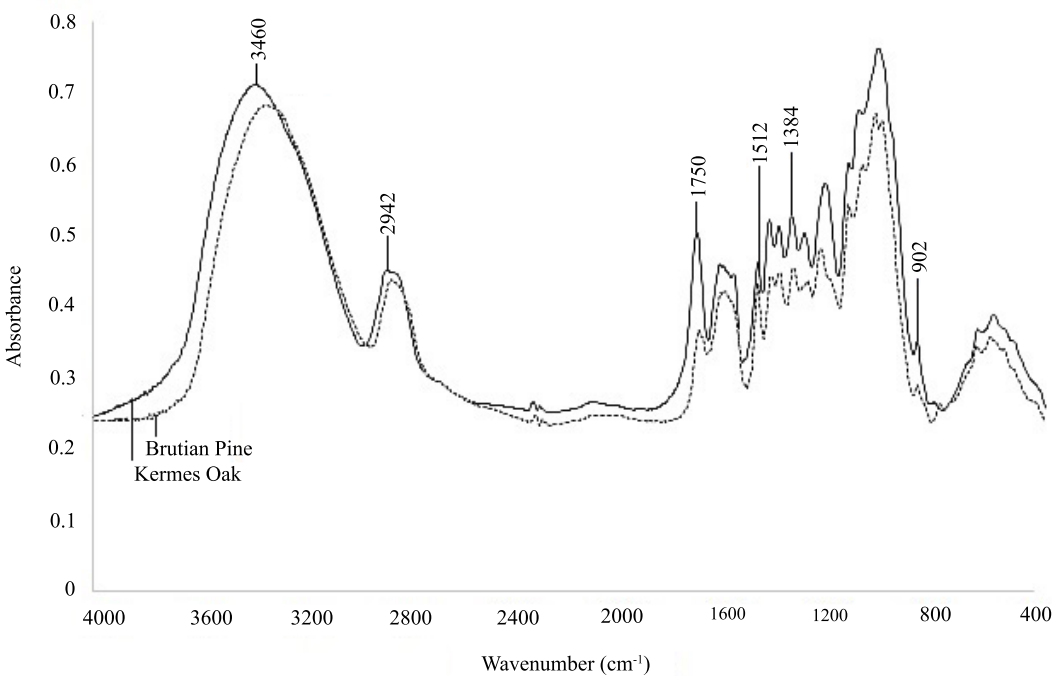

Figure 2. FTIR spectra of kermes oak and brutian pine fibers.

Table 6. Absorbance intensity ratio of infrared spectra of kermes oak and brutian pine fibers.

A_v/A_{2942}	Kermes oak	Brutian pine
A_{3460}/A_{2942}	1.588	1.553
A_{2942}/A_{2942}	1.000	1.000
A_{1750}/A_{2942}	1.087	0.851
A_{1512}/A_{2942}	1.003	1.013
A_{1384}/A_{2942}	1.154	1.048
A_{902}/A_{2942}	0.763	0.684

C-H deformation in the glucose ring in cellulose (Luna et al., 2012). At this band, the absorbance value of kermes oak fibers was higher than the value in brutian pine fibers, and kermes oak contained a higher cellulose content than brutian pine. The FTIR results are consistent with the findings from the α-cellulose, hemicellulose, and lignin analyses in the study. These current FTIR findings were also supported by findings in kermes oak and brutian pine particles from Güler and Yaşar (2018).

3.4. Thermogravimetric analysis and differential thermogravimetric analysis (DTG) thermograms

TGA and DTG analysis thermograms of kermes oak and brutian pine fibers are shown in Figure 3.

Water and some extractives were removed until temperatures of 105 °C in the brutian pine sample and 148 °C in the kermes oak sample were attained. Actual decomposition, which is the thermal degradation of hemicellulose and the rest of the extractives, lignin, and cellulose, occurred at temperatures from 105 °C to 397 °C in brutian pine and from 148 °C to 464 °C in kermes oak. Maximum degradation occurred at 271 °C in brutian pine and at 317 °C in kermes oak. Similar curves of the thermal decomposition of lignocellulosic fibers have been reported in earlier studies (Shebani et al., 2008; Jumhuri et al., 2014; Tanobe et al., 2014; Yaşar and İçel, 2016; Güler and Yaşar, 2018). TGA showed that kermes oak fibers had higher thermal stability than brutian pine fibers. This indicates that increasing the proportion of kermes oak fibers could lead to an increase in the thermal stability of produced boards.

The TGA findings from kermes oak and brutian pine fibers are compatible with the findings in kermes oak and brutian pine particles by Güler and Yaşar (2018).

3.5. The physical properties of fiberboards

The physical properties of boards produced from kermes oak and brutian pine fibers are presented in Table 7. There was a statistical difference between the values of physical properties according to ANOVA ($P < 0.001$). The different groups were identified according to Duncan's test and are shown by different letters in each column of the table. Previous studies showed that extractive compounds hinder the entrance of water into the wood (Gönültaş, 2008; Nemli et al., 2008; Büyüksarı et al., 2010; Nasser, 2012). In particular, wax and oil compounds act as a thin

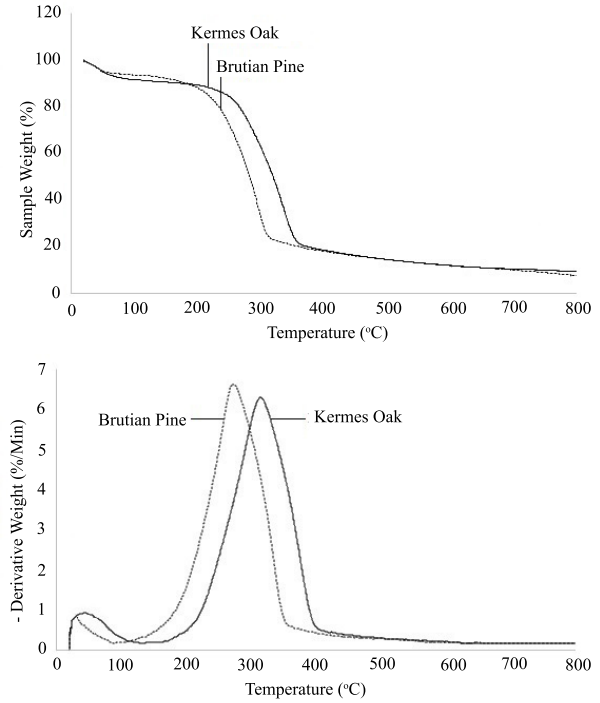

Figure 3. TGA (a) and DTG (b) thermograms of kermes oak and brutian pine fibers.

film and prevent water from penetrating the wood (Ashori and Nourbakhsh, 2008, 2010; Pirayesh et al., 2012, 2013). Polysaccharides in lignocellulosic material (cellulose and hemicellulose) are rich in –OH content in their molecular structures. Therefore, they are hydrophilic and promote water entrance (Gwon et al., 2010). Lignin resists water ingress into the wood, which makes it hydrophobic (Nourbakhsh et al., 2011). Compared to brutian pine fibers, kermes oak fibers had higher extractive and holocellulose (cellulose and hemicellulose) contents but lower lignin content. Generally, kermes oak fibers contained higher hydrophilic compounds content but lower hydrophobic compounds content. For this reason, an increase in the proportion of kermes oak fibers may raise the WA and TS of the produced boards.

According to the standard TS-EN 622-5 (2011), fiberboards should have a maximum TS (24 h) value of 15% for load-bearing applications in dry conditions. The A- and B-type boards of this study met the requirement of TS (24 h) value for load-bearing applications in dry conditions. The TS (24 h) values of A- and B-type boards were under 15%, with values of 13.48% and 14.66%, respectively.

3.6. The mechanical properties of fiberboards

The mechanical properties of the boards produced from kermes oak and brutian pine fibers are shown in Table 8. Statistical differences were found between the values of mechanical properties according to ANOVA (MOE and MOR, $P < 0.05$; IB, $P < 0.01$). The different groups identified by Duncan's test are given with different letters in each column of the table. Previous studies reported that extractive contents had a negative effect on adhesion between the wood fibers and particles, resulting in decreased mechanical properties of the produced boards (Ayrılmış et al., 2009; Büyüksarı et al., 2010). The source of polar –OH groups in wood comprises, in particular, holocellulose (cellulose and hemicellulose) and lignin. Polar –OH groups are responsible for hydrogen bonds with polar adhesive polymers (Ayrılmış et al., 2009; Ayrılmış and Winandy, 2009). Therefore, increased holocellulose content would contribute to an improvement in the mechanical properties of the produced composite boards (Yaşar and İçel, 2016). Moreover, lignin is a natural adhesive; therefore, it would positively contribute to adhesion between the fibers and it has a positive effect on the mechanical properties of the produced boards (Joseleau et al., 2004). The curing of UF resin is dependent on wood acidity. When the pH value of wood is lower than 4, precuring of resin occurs before hot pressing, resulting in negatively affected mechanical properties in the produced boards (Akyüz et al., 2010; Baharoğlu et al., 2013). A general evaluation of chemical composition

Table 7. Physical properties of boards produced from kermes oak and brutian pine fibers.

Board type	WA - 2 h	WA - 24 h	TS - 2 h	TS - 24 h
A	32.04 (2.47)[1] a[2]	46.71 (3.11) a	7.52 (1.65) a	13.48 (1.82) a
B	34.07 (3.36) a, b	49.37 (3.90) a, b	8.36 (1.58) a	14.66 (1.76) a
C	36.88 (4.88) b, c	52.04 (4.09) b	9.73 (1.67) b	16.41 (2.07) b
D	39.24 (4.43) c, d	56.41 (4.21) c	11.55 (2.89) c	18.59 (2.15) c
E	41.82 (4.80) d	59.18 (3.97) c	12.75 (1.78) d	19.92 (1.49) d

[1] Standard deviation, [2] Groups defined by different letters in each column according to Duncan's test for WA (2 and 24 h) and TS (2 and 24 h); $P < 0.001$.

Table 8. Mechanical properties of boards produced from kermes oak and brutian pine fibers.

Board type	MOR (N/mm^2)	MOE (N/mm^2)	IB (N/mm^2)
A	29.77 (1.97)[1] a[2]	2982 (142) a	0.68 (0.12) a
B	28.23 (1.89) a	2902 (136) a, b	0.67 (0.01) a
C	27.83 (1.76) a, b	2867 (131) a, b, c	0.66 (0.11) a
D	26.34 (1.61) a, b	2742 (127) b, c	0.63 (0.11) a, b
E	24.07 (1.59) b	2662 (118) c	0.59 (0.13) b

[1] Standard deviation, [2] Groups defined by different letters in each column according to Duncan's test for MOE and MOR ($P < 0.05$) and IB ($P < 0.01$).

and pH reveals that increasing the proportion of kermes oak fibers could lead to poor mechanical properties in the produced boards.

Based on standard TS-EN 622-5 (2011), fiberboards should have a minimum MOE value of 2800 N/mm^2, a minimum MOR value of 27 N/mm^2, and a minimum IB strength value of 0.65 N/mm^2 for load-bearing applications in dry conditions. The A-, B-, and C-type boards of the study satisfied standard TS-EN 622-5 (2011) in terms of MOE, MOR, and IB strength with values of 2982, 2902, and 2867 N/mm^2 for MOE; 29.77, 28.23, and 27.83 N/mm^2 for MOR; and 0.68, 0.67, and 0.66 N/mm^2 for IB strength, respectively.

Consequently, TS (24), MOE, MOR, and IB strength should be considered together to qualify fiberboards for load-bearing applications in dry conditions according to standard TS-EN 622-5 (2011). In this case, only boards containing 25% kermes oak fibers meet the requirements for load-bearing applications in dry conditions according to standard TS-EN 622-5 (2011).

3.7. Conclusions

In this study the chemical and thermal properties of kermes oak, which is a potential raw material for the wood industry, were determined, and their effects on the physical and mechanical properties of the produced fiberboards were evaluated. Kermes oak fibers were mixed with brutian pine fibers in proportions of 0%, 25%, 50%, 75%, and 100% and used in board production. Results of TGA showed that increasing the proportion of kermes oak fibers could raise the thermal stability of the produced boards. Compared to brutian pine fibers, a higher content of hydrophilic compounds and lower content of hydrophobic compounds were determined in kermes oak fibers. This led to a decrease in the water resistance of the produced boards when a higher proportion of kermes oak fibers was utilized. Although a higher holocellulose content, which improved mechanical properties, was determined in kermes oak fibers, the lower lignin content, higher extractive content, and much lower pH value of kermes oak fibers resulted in poor mechanical properties in the produced boards. Only boards produced with 25% kermes oak fibers satisfied standard TS-EN 622-5 (2011) in terms of TS, MOE, MOR, and IB strength for load-bearing applications in dry conditions. The utilization of kermes oak fibers at more than 25% in the production of boards led to weak physical and mechanical properties.

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