ABSTRACT

Black walnut wood (Juglans nigra L.) was treated with fresh cow manure for several days and then some physical and mechanical properties of manure-treated black walnut wood were evaluated. The results showed that swelling percent and several mechanical properties (compressive strength parallel to grain, static bending strength and Janka hardness) of wood were clearly decreased after the manure treatment. However, shrinkage percent and impact bending strength were not significantly affected by manure treatment.

KEY WORDS: black walnut wood, physical properties, Juglans nigra L., mechanical properties, manure

INTRODUCTION

In order to carve wood easily and change its color, many wood products’ producers treat wood with heat as well as chemicals, e.g., ammonia (Anonymous 1987, Schuerch 1964, Stamm 1964). Nowadays, though most of manufacturer of wood products use steam as heat source, some of them use traditional method such as manure.

Yet, the durability of wood may be defined as the capacity of resisting environmental stresses. It is well known that wood strength decreases when it is heated (Maclean 1955, Millet and Gerhards 1972, Seborg et al. 1953, Feist and Sell 1987). It was reported that for the limited exposure of temperature up to 65.5 °C, this sudden effect of temperature is reversible (Green and Evans 2001). However, prolonged heating at temperatures can lead a irreversible loss in strength. For example when wood was heated at 65.5 °C for 2 months, mean residual property is 0.95 (Green and Evans 2001). However, it is heated at the same temperature for 48 months, residual property decreases to the level of 0.41. Some manufacturers of wood products make heat treatment by using direct combustion procedure or indirect ones such as manure.

In wood carving industry established in Turkey, Black walnut wood (Juglans nigra L.) has
been traditionally used as typical raw material because of their dark color after treatment with steam/manure in Turkey (Bektaş et al. 2002). However, no study has been done on the effect of manure treatment on the wood properties. Therefore, in this study it was aimed at investigating the relationship between physical and mechanical properties of wood and duration of manure treatment.

MATERIAL AND METHODS

Black walnut (Juglans nigra L.) was collected from the Bitlis province in the Eastern part of Turkey. The black walnut logs (40 cm (diameter) x 300 cm (length)) were cut to the timbers (8 cm (thickness) x 25 cm (width) x 100 cm (length)). Fresh cow manure collected in Kahramanmaraş city was used. The gas properties of the manure were given in Table 1 (Unal and Baskaya 1981).

<table>
<thead>
<tr>
<th>Gases</th>
<th>Percentage</th>
<th>Gases</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₄</td>
<td>34</td>
<td>HCHO</td>
<td>1</td>
</tr>
<tr>
<td>CO₂</td>
<td>21</td>
<td>HC</td>
<td>1</td>
</tr>
<tr>
<td>NOₓ</td>
<td>9</td>
<td>NH₃</td>
<td>1</td>
</tr>
<tr>
<td>SOₓ</td>
<td>3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Tab. 1: Gas contents produced from the manure during the manure treatment of wood

The sapwood parts (having moisture content of 57%) of walnut timber with white surface were soaked into the manure for 45 and 60 days. The temperature changes in the manure during the treatment were recorded every day and illustrated in Fig. 1.

Fig. 1: Temperature changes in the manure during the treatment of the walnut wood
Determination of Shrinkage and Swelling

Wood specimens (with 57% moisture content) having a dimension of 30 x 30 x 15 mm and 30 x 30 x 100 mm were prepared for swellings and shrinkages in both tangential and radial, and longitudinal directions, respectively, based upon Turkish Standards (TS 2472). For the measurement of shrinkage, the specimens were first immersed in distilled water to constant weight, and their dimensions were briskly measured on a caliper to a accuracy of 0.001 mm after being wrapped with tissue paper. Then, the specimens were oven-dried at 103 ± 2 °C up to constant weight and cooled in a desiccator for a while. Finally, the dimensions in radial, tangential and longitudinal directions were measured as described above. The shrinkages of the wood specimens were separately calculated in percentage for tangential (t), radial (r) and longitudinal (l) directions as follows:

\[
\beta_{r/t/l} = \frac{V_d - V_0}{V_d} \times 100
\]

where, \( \beta \) is shrinkages in radial (r), tangential (t), and longitudinal (l) directions (%), \( V_d \) is the dimension of saturated specimen (mm) and \( V_0 \) is the dimension of oven-dry specimen (mm).

Then, the volumetric shrinkage was calculated for the radial, tangential and longitudinal directions by the following equation:

\[
\beta_v = \beta_r + \beta_t + \beta_l
\]

where, \( \beta_v \) is the volumetric shrinkage (%), \( \beta_r \) is radial shrinkage, \( \beta_t \) is tangential shrinkage and \( \beta_l \) is longitudinal shrinkage.

For the swelling test, the specimens whose oven-dry dimensions have already known in the shrinkage test were immersed in distilled water up to reaching the constant weight. Then, the dimensions in radial, tangential and longitudinal directions were measured on a micrometer to the nearest 0.001 mm. For both shrinkage and swelling test, a total of about 1000 measurements were done. Finally, the swellings in the radial, tangential and longitudinal directions of the calabrian pine wood were calculated by the following equation:

\[
\alpha_{r/t/l} = \frac{V_d - V_0}{V_0} \times 100
\]

where, \( \alpha \) is swellings, radial (r), tangential (t) and longitudinal (l) directions (%). Then, the volumetric swelling was calculated by the following equation:

\[
\alpha_v = \alpha_r + \alpha_t + \alpha_l
\]

where, \( \alpha_v \) is the volumetric swelling (%), \( \alpha_r \) is radial swelling (%), \( \alpha_t \) is tangential swelling (%), and \( \alpha_l \) is longitudinal swelling.

Determination of Mechanical Properties

Both salvaged and new juniper wood specimens to be tested for mechanical properties were conditioned at 20±2 °C and 65±5% relative humidity (RH) to the moisture content of about 12% before all specimens were tested.
Compressive Strength Parallel to Grain

The compressive strength parallel to grain was measured by following Turkish Standards (TS 2595). The dimension of specimens was 40 x 40 x 60 mm and crosshead motion or rate of loading was 0.6 mm/min. The test was performed on a tone universal testing machine (Losenhausen). The test was performed on the at least 69 specimens. The compressive strength was calculated by the following Equation [5]:

\[ \sigma_{cpl} = \frac{P_{\text{max}}}{F} \]  \hspace{1cm} (MPa) \hspace{1cm} [5]

where, \( \sigma_{cpl} \) is the compressive strength (MPa), \( P_{\text{max}} \) is the maximum load at the break point (N) and \( F \) is area of cross-section of specimen on which force was applied (mm²).

Finally, at the end of the test the moisture content of the broken specimen was determined by following Turkish Standard (TS 2472) and then, the compressive strength of the specimen whose moisture content deviated from 12% was converted by the following Equation [6]:

\[ \sigma_{\text{cpl12}} = \sigma_{cpl} \left[ 1 + 0.06(w - 12) \right] \] \hspace{1cm} (MPa) \hspace{1cm} [6]

where, \( w \) is the moisture content (g), \( \sigma_{\text{cpl12}} \) is the compressive strength at the moisture content of 12% (MPa) and \( \sigma_{cpl} \) is the compressive strength at w% moisture content level (MPa).

Static Bending Strength

The test of bending strength perpendicular to grain, i.e., modulus of rupture (MOR), was performed according to Turkish Standard (TS 2474). The size of specimens was 20 x 20 x 360 mm and loading speed was 1 mm/min. For the test, at least 67 samples were used. The MOR of the specimen was calculated by the following Equation [7]:

\[ MOR = \frac{3 \ P_{\text{max}} \ l}{2 \ b \ h^2} \] \hspace{1cm} (MPa) \hspace{1cm} [7]

where, \( P_{\text{max}} \) is the maximum load at break point (N), \( l \) is the length of span (300 mm), \( b \) is the width of specimen (mm), and \( h \) is the thickness of specimen (tangential to annual ring) (mm).

Eventually, after termination the test the moisture contents of the broken specimens were determined by following Turkish Standard (TS 2472). The bending strength of the specimen whose moisture content deviated from 12% was converted by the following Equation [8]:

\[ MOR_{12} = MOR \left[ 1 + 0.04(w - 12) \right] \] \hspace{1cm} (MPa) \hspace{1cm} [8]

where, \( w \) is the moisture content (%), \( MOR_{12} \) is the bending strength at the moisture content of 12% and MOR is the bending strength at w% moisture content.

Impact Bending Strength

Impact bending strengths were measured by following Turkish Standard (TS 2477). The specimen (20 x 20 x 300 mm) were equilibrated at 20±2 °C and 65 ± 5% RH for 4 months to
the moisture content of about 12%. Impact bending strength of the specimens was tested on an impact tester (Model HPSW 10). The speed of hammer was 28.67 in./sec. The test was conducted on at least 50 specimens. The impact bending strength was calculated by the following Equation [9]:

\[ W = \frac{A}{F} \text{ (J/mm}^2\text{)} \]  

[9]

where, \( W \) is the impact bending strength (J/mm\(^2\)), \( A \) is the energy absorbed by the specimen (J) and \( F \) is the cross sectional area of the specimen (mm\(^2\)).

**Determination of Janka Hardness (H\( j \))**

Janka hardness of eastern beech wood specimens (50 x 50 x 50 mm) was performed using a universal tester (Losenhausen) according to Turkish Standard (TS 2479). In this test, hemispherical head with a diameter of 11.28 mm was forced into the center of the specimens to the depth of 1 in. with a head speed of 0.248 in./min. The load required was recorded in kgf and reported as N. The hardness was performed on cross, radial and tangential sections of the wood specimens.

**RESULTS AND DISCUSSION**

Table 2 shows the shrinkage and swelling ratios of walnut wood treated with manure as well as those of untreated ones. As indicated in Table 1, after the treatment process, both volumetric shrinkage and swelling ratios of walnut wood have an obvious increase. This tendency is valid for all the shrinkage and swelling ratios measured at all the directions except for shrinkage ratio at tangential direction. As the treatment time is increased from 45 days to 60 days, the increases in the volumetric shrinkage and swelling ratios increase. Also, the increase in volumetric swelling ratio is higher than that of the volumetric shrinkage ratio.

<table>
<thead>
<tr>
<th>Fertilizing Time</th>
<th>Shrinkage (%)</th>
<th>Swelling (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>radial</td>
<td>tangential</td>
</tr>
<tr>
<td>Untreatment</td>
<td>4.62</td>
<td>8.56</td>
</tr>
<tr>
<td>45 days</td>
<td>5.44</td>
<td>7.80</td>
</tr>
<tr>
<td>60 days</td>
<td>5.66</td>
<td>8.03</td>
</tr>
</tbody>
</table>

*The amounts of shrinkage and swelling in longitudinal direction were ignored.

As also can be seen from Table 3, changes in the shrinkage ratio are not significant according to ANOVA and Tukey’s mean separation test. However, changes in shrinkage ratios are found to be significant on the basis of the ANOVA and Tukey’s mean separation test (\( P<0.05 \)).

Fig. 2 demonstrates the results of compression strength parallel to grain (CS) and static bending strength (SBS) tests for the walnut wood as a function of manure–treatment time. As shown in this figure, the compression strength parallel to grain of walnut wood is significantly affected by
the manure treatment. Moreover, the compression strength of the treated walnut wood does not significantly differ when increasing treatment time from 45 days to 60 days.

*Tab. 3: The results of ANOVA and Tukey’s mean separation test for shrinkage and swelling percents of walnut wood treated as a function of manure-treatment time*

<table>
<thead>
<tr>
<th>Shrinkage</th>
<th>Fertilizing Time</th>
<th>N²a</th>
<th>Volumetric (%)</th>
<th>SDᵇ</th>
<th>SEᶜ</th>
<th>COVᵈ</th>
<th>Pᵉ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreatment</td>
<td>44</td>
<td></td>
<td>13.18ᵇ</td>
<td>2.593</td>
<td>0.39</td>
<td>13.99</td>
<td>NS</td>
</tr>
<tr>
<td>45 days</td>
<td>44</td>
<td></td>
<td>13.24ᵇ</td>
<td>0.988</td>
<td>0.14</td>
<td>0.25</td>
<td></td>
</tr>
<tr>
<td>60 days</td>
<td>44</td>
<td></td>
<td>13.68ᵇ</td>
<td>1.676</td>
<td>0.25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Swelling</th>
<th>Fertilizing Time</th>
<th>N²a</th>
<th>Volumetric (%)</th>
<th>SDᵇ</th>
<th>SEᶜ</th>
<th>COVᵈ</th>
<th>Pᵉ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreatment</td>
<td>44</td>
<td></td>
<td>12.32ᵇ</td>
<td>4.980</td>
<td>0.75</td>
<td>33.94</td>
<td>*</td>
</tr>
<tr>
<td>45 days</td>
<td>44</td>
<td></td>
<td>13.85ᵇ</td>
<td>6.231</td>
<td>0.94</td>
<td></td>
<td></td>
</tr>
<tr>
<td>60 days</td>
<td>44</td>
<td></td>
<td>15.06ᵇ</td>
<td>1.322</td>
<td>0.20</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Number of samples; b Standard deviation; c Sampling error; d Coefficient of variation; e Non significant (for ANOVA). Values having the same letters are not significantly different and vice versa (for Tukey test).

*Fig. 2: Compression strength parallel to grain (CS) and static bending strength (SBS) and for the walnut wood treated with manure as a function of treatment time as well as control (untreated walnut wood)*
As also indicated in the same figure, static bending strength parallel to grain of walnut wood significantly decreases when the wood is treated with manure (Fig. 2). However, it is clear from the table that no significant change in the strength is determined as the treatment time is increased.

Fig. 3 shows impact bending strength of the walnut wood as a function of manure-treatment time. As can be seen from Fig. 3, no significant difference is found for impact bending strength of walnut wood after treatment with manure for two different times, 45 and 60 days.

![Fig. 3: Impact bending strength of the walnut wood treated with manure as a function of treatment time along with control (untreated walnut wood)](image)

Janka hardness values of untreated and treated walnut wood are given in Table 4. It is evident from Table 4 that the Janka hardness values of walnut wood determined for axial, radial and tangential directions clearly decrease due to manure treatment. However, no obvious differences in Janka hardness values determined for three directions between 45 and 60 days treatments are determined. Furthermore, the ANOVA test shows that the mean values of Janka hardness is significantly affected by the manure treatment ($P<0.05$) as also indicated in Table 4. Yet, no meaningful change is determined when increasing treatment time from 45 to 60 days.

All these changes in the mechanical strengths and some physical properties studied here can be ascribed to the heat of manure as shown in Figure 1. It is well known that heat treatment tremendously affect the mechanical strengths of the wood. For example, when wood was heated at 75 °C for 6 months, a 7% loss in MOE (modulus of elasticity) and 33.8% loss in MOR (modulus of rupture/static bending strength) were determined (Green and Evans 2001).

On the other hand, this changing mechanism resulted from heat treatment may be due to the particular loss in hemicellulose, which acts as a binder between cellulose and lignin ratios (Fengel and Wegener 1984). Green and Evans (2001) reported that the greatest loss was found in the amount of arabinose, followed by galactose, xylose and mannose respectively when wood was
heated for 6 months. Also, it was reported by Winandy and Lebow (2001) that wood is heated over long periods, significant strength loss is possible without a decrease in the amounts of cellulose or lignin, except for hemicellulose.

Tab. 4: The results of ANOVA and Tukey’s mean separation test for Janka hardness of walnut wood treated as a function of manure-treatment time

<table>
<thead>
<tr>
<th>Fertilizing Time</th>
<th>D$_{12}$$^a$ (g/cm$^3$)</th>
<th>arw$^b$ (mm)</th>
<th>Janka Hardness (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>axial</td>
</tr>
<tr>
<td>45 days</td>
<td>0.628</td>
<td>5.8</td>
<td>73.6$^x$</td>
</tr>
<tr>
<td>60 days</td>
<td>0.640</td>
<td>5.9</td>
<td>74.6$^x$</td>
</tr>
<tr>
<td>Untreatment</td>
<td>0.681</td>
<td>5.3</td>
<td>75.5$^y$</td>
</tr>
</tbody>
</table>

ANOVA and Tukey’s mean separation test for mean hardness

<table>
<thead>
<tr>
<th>Fertilizing Time</th>
<th>N$^a$</th>
<th>Mean (MPa)</th>
<th>SD$^b$</th>
<th>SE$^c$</th>
<th>COV$^d$</th>
<th>P$^e$</th>
</tr>
</thead>
<tbody>
<tr>
<td>45 days</td>
<td>15</td>
<td>58.8$^x$</td>
<td>11.052</td>
<td>2.85</td>
<td>17.55</td>
<td>*</td>
</tr>
<tr>
<td>60 days</td>
<td>15</td>
<td>60.2$^x$</td>
<td>11.254</td>
<td>2.91</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Untreatment</td>
<td>15</td>
<td>68.5$^y$</td>
<td>10.584</td>
<td>2.73</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$^a$Air dry density, $^b$Annual ring width, $^c$Number of samples; $^d$Standard deviation; $^e$Sampling error; $^f$Coefficient of variation; $^g$Non significant (for ANOVA). $^h$Values having the same letters are not significantly different and vice versa (for Tukey test).

CONCLUSIONS

Some physical and mechanical properties of manure-treated black walnut wood treated with fresh cow manure for several days were investigated. It was found that the manure treatment significantly affected the swelling percent and several mechanical properties (compressive strength parallel to grain, static bending strength and Janka hardness) of the walnut wood. Yet, shrinkage percent and the impact bending strength of the walnut wood did not significantly vary with the manure treatment.

ACKNOWLEDGEMENT

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