

THE IMPROVE OF COMBUSTION PROPERTIES ON WOODEN MATERIAL BY USING LIQUED NITROGEN AND BORIC ACID

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Abstract: In this study, the effects of combustion properties on oriental beech (*Fagus orientalis* L.) wood species which treated with liquid nitrogen and impregnated with boric acid. The dipping method was used for the impregnation and liquid nitrogen process. The combustion test was performed according to the procedure defined in the ASTM-E 69 standards and during the test the mass reduction, temperature were determined for each 30 seconds. According to the test results, it was found that the samples treated with liquid nitrogen retention was increase. Moreover, sample treated with liquid nitrogen and impregnated with boric acid was more successfully to combustion retardant than samples only impregnated with boric acid.

Key words: Combustion, Beech, Liquid Nitrogen, Boric acid, Impregnated, Wood.

Introduction

The wood materials widely used from wood construction sector, urban furniture and interior design to furniture sector are renewable resource. To increase service life of wood materials which especially used to urban furniture, wood materials are treated coating and impregnated materials against to biological and mechanical factors. Wood materials used in wood construction sector should be treated by using fire retardant chemicals. Beside, the wood materials that are used in place where can be attack biological pets should be used to some impregnate materials. Many researchers have studied effects of combustion properties of wooden materials. Uysal et. al (2009) studied combustion properties of the oriental spruce (*Picea orientalis* L.) impregnated with boron compounds. A borax-boric acid 10% solution was found to be the most successful fire retardant chemical . Yapıcı et. Al (2011) studied effects of pretreatment with boric acid, borax and Tanalith-E on combustion properties of varnished Oriental beech. It was shown that the most mass reduction occurred in samples impregnated with boric acid and varnished with polyurethane varnish. Borates have several great advantages as wood preservatives as well as imparting flame retardancy, providing sufficient protection against all forms of wood destroying organisms, have low mammalian toxicity and low volatility, they are moreover colorless and odorless (Murphy,1990;Yalınkılıç et.al, 1999; Drysdale, 1994; Chen et al., 1997). In this study, it was investigated properties of the beech wood treated with liquid nitrogen and impregnated with borax.

Material and Method

Wood Species

Oriental beech (*Fagus orientalis* L.) was chosen randomly from timber suppliers of Ankara, Turkey. Blending process was made to represent control simples on other groups. A special emphasis was put on the selection of the wood material. Accordingly, non-deficient, whole, knotless, normally grown (without zone line, reaction wood, decay, insect or fungal infection) wood materials were selected.

Chemical Materials

Boric acid were used as impregnation chemical. Properties of impregnation chemical and test plan are given in Table 1.

Table 1. Properties of impregnation chemical and test plan

| Impregnation Chemicals | Solution Conc. (%) | Solvent | Purity (%) | pH | | Density (g/ml) | |
|------------------------|-----------------------|------------|---------------|------|------|----------------|------|
| | | | | BI | AI | BI | AI |
| Boric Acid | 5 | Pure water | 98 | 5.23 | 5.30 | 1.02 | 1.02 |

BI:Before impregnation AI: After impregnation

Nitrogen gas composes 78% of the Earth's atmosphere. It is a colorless, odorless and non-flammable gas. It is used in the electrical industry, producing chemicals safely, the food packaging industries and in the drying and preparation of refrigeration systems. Nitrogen gas is also used in at temperatures below -196 °C, nitrogen is a liquid. When liquid nitrogen comes into contact with objects at room temperature it boils rapidly from the heat energy emitted by the objects.

Determination of density

Wood materials were kept in the room at $20 \pm 2^\circ\text{C}$ and $65 \pm 3\%$ relative humidity until their weight became stable. Air dry densities of wood materials before and after impregnated used for the preparation of treatment samples were determined according to TS 2472. Afterwards, the dimensions of wood materials were measured by a compass of $\pm 0,001$ sensitivity and volumes were determined by a stereo-metric method. The air dry density (δ_{12}) was calculated by the following equation:

$$\delta_{12} = M_{12}/V_{12} \text{ g/cm}^3 \quad (1)$$

where M_{12} is the perfect air dry weight (g) and V_{12} is the volume (cm^3) of the wood material.

Preparation of Test Samples

The oversized test samples were acclimatized until they were stable at $20 \pm 2^\circ\text{C}$ and $65 \pm 3\%$ relative humidity in climate room. Later on they were cut with the dimensions of $9 \times 19 \times 1016$ mm according to the procedure of ASTM E – 69 then, specimens were treated to liquid nitrogen in tank during 4 hours. Impregnation processes stated at ASTM D 1413-76 was applied to the prepared test samples. Using the dipping method, the samples were dipped into the impregnation solution for 48 h for long-term dipping. Before the impregnation process, all samples were weighed and then kiln-dried at a temperature of $103 \pm 2^\circ\text{C}$ until they reached constant weight. Then, the samples were weighed in an analytic balance with 0.01-g sensitivity. After the impregnation, all impregnated samples were held for 15 days in circulating air for evaporation of the solvent. Impregnated test samples were kept at a temperature of $20 \pm 2^\circ\text{C}$ and $65 \pm 3\%$ relative humidity until they reached constant weight. After this period, the impregnated samples were kiln-dried at $103 \pm 2^\circ\text{C}$ until they reached constant weight. After cooling, all dried samples in the dessicator were weighed on the scale. The dry weight of the samples was determined and recorded. The amount of retention (R, kg/m^3) was calculated as follow:

$$R(\text{kg/m}^3) = \frac{GC}{V} \times 10 \quad (2)$$

where, $G = T_2 - T_1$, T_2 is the sample weight after impregnation (g), T_1 is the sample weight before impregnation (g), V is the volume of sample (cm^3), and C is the concentration of solution (%).

Execution Test

The combustion test was carried out according to the principles of the ASTM E –69. But some changes were made in the stand. For this purpose, a digital balance having 0.01 g sensitiveness has been used for determination of mass reduction of materials when they are burnt. Butane gas was used to make an ignition flame. The gas flow is standard as the high of flame is 25 cm, the temperature must be 1000°C . The distance between the bottoms of the test samples, which were hanged inside of the fire tube and the top of the gas pipe must be adjusted as 2.54 cm. During the test, mass reduction, temperature were determined in every 30 seconds. The test was made under a chimney where the flow of air blown was drawn with natural draft. At the beginning of combustion test flame source was used for 4 minutes then flame source was taken away and it was continued 6 minutes. Totally 10 minutes, the test was lasted. Testo 350 M and XL flue gas analyzers was used for measuring concentration of the released gasses (CO , NO , O_2) and temperature variation. The probe was inserted into the first hole from the top of the fire tube.

Data Analyses

By using untreated control samples and treated with liquid nitrogen, impregnation chemical and unimpregnated control sample a total of 120 ($2 \times 2 \times 30$) samples were prepared using 30 samples for each parameter.

Results and Discussion

The averages of density are given in Table 2. The air dry density (0.695 gr/cm³) was obtained in control oriental beech wood. The air dry density (0.737 gr/cm³) was obtained in treated with impregnated oriental beech wood samples.

Table 2. Average values of density (gr/cm³).

| Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|---------|------------------------------|--------------------------|--|
| 0.695 | 0.627 | 0.737 | 0.704 |

Retention amount of boric acid was obtained from control samples 1.60% when treated with liquid nitrogen samples was determined retention amounts 1.83% and also these samples impregnated with boric acid.

The averages of mass reduction are given in Table 3. The highest mass reduction occurred in control samples with 45.90% (8th measurement) as a result of flame source while the lowest one occurred in treated with liquid nitrogen and impregnated samples with 22.83%. The highest mass reduction occurred in control samples with 98.32% as a result of the end of combustion while the lowest one occurred in treated with liquid nitrogen and impregnated samples with 31.37%.

Table 3. Average of mass reduction(%)

| Measured of time | Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|------------------|---------|------------------------------|--------------------------|--|
| 1* | 1,52 | 2,71 | 2.40 | 2,85 |
| 2* | 2,78 | 5,37 | 6.43 | 4,17 |
| 3* | 5,55 | 7,32 | 8.87 | 5,70 |
| 4* | 10,20 | 12,59 | 11.45 | 8,99 |
| 5* | 17,19 | 19,05 | 15.08 | 13,42 |
| 6* | 26,92 | 29,92 | 17.34 | 16,80 |
| 7* | 37,33 | 39,54 | 19.46 | 18,96 |
| 8* | 45,90 | 46,70 | 23.66 | 22,83 |
| 9 | 52,94 | 50,96 | 24.02 | 23,97 |
| 10 | 61,68 | 58,73 | 25.38 | 24,08 |
| 11 | 72,07 | 67,53 | 26.26 | 25,09 |
| 12 | 76,56 | 76,44 | 26.89 | 25,70 |
| 13 | 86,83 | 85,66 | 27.09 | 26,14 |
| 14 | 91,71 | 92,17 | 28.45 | 26,40 |
| 15 | 95,03 | 94,31 | 29.61 | 26,86 |
| 16 | 95,47 | 95,65 | 30.10 | 27,42 |
| 17 | 96,67 | 96,38 | 31.73 | 28,97 |
| 18 | 97,21 | 96,80 | 32.59 | 30,39 |
| 19 | 97,78 | 97,31 | 34.99 | 30,99 |
| 20 | 98,02 | 98,66 | 36.07 | 31,37 |

* Flame source combustion

The averages of temperature are given in Table 4. The highest temperature (740 °C) variation was observed in the treated with liquid nitrogen samples, the lowest (219 °C) in treated with liquid nitrogen and impregnated samples.

Table 4. Average of temperature values (°C)

| Measured of time | Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|------------------|---------|------------------------------|--------------------------|--|
| 1* | 75 | 80 | 72 | 81 |
| 2* | 91 | 100 | 84 | 95 |
| 3* | 108 | 118 | 95 | 107 |
| 4* | 135 | 136 | 109 | 122 |
| 5* | 164 | 158 | 130 | 142 |
| 6* | 199 | 186 | 156 | 157 |
| 7* | 239 | 213 | 178 | 165 |
| 8* | 268 | 238 | 189 | 177 |
| 9 | 313 | 266 | 200 | 184 |
| 10 | 417 | 327 | 208 | 187 |
| 11 | 556 | 495 | 215 | 195 |

| | | | | |
|----|-----|-----|-----|-----|
| 12 | 680 | 654 | 224 | 206 |
| 13 | 723 | 740 | 236 | 219 |
| 14 | 711 | 724 | 217 | 205 |
| 15 | 641 | 707 | 209 | 198 |
| 16 | 531 | 561 | 186 | 184 |
| 17 | 452 | 473 | 174 | 172 |
| 18 | 398 | 412 | 163 | 161 |
| 19 | 358 | 367 | 151 | 145 |
| 20 | 318 | 329 | 139 | 132 |

* Flame source combustion

The averages of O₂ amounts are given in Table 5. The highest reduction of O₂ concentration (% 19.14) was measured in treated with liquid nitrogen and impregnated samples, the lowest change of O₂-concentration (%16.42) in combustion of control samples.

Table 5. Average of O₂ amounts(%)

| Measured of time | Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|------------------|---------|------------------------------|--------------------------|--|
| 1* | 20,85 | 20,75 | 20,42 | 20,48 |
| 2* | 20,46 | 20,45 | 20,26 | 20,21 |
| 3* | 19,82 | 20,02 | 20,04 | 20,06 |
| 4* | 19,26 | 19,41 | 19,37 | 19,73 |
| 5* | 18,60 | 18,94 | 19,33 | 19,65 |
| 6* | 17,93 | 18,28 | 19,19 | 19,39 |
| 7* | 17,39 | 18,34 | 18,90 | 19,34 |
| 8* | 17,09 | 18,33 | 18,80 | 19,25 |
| 9 | 16,94 | 18,38 | 18,74 | 19,14 |
| 10 | 16,42 | 18,17 | 19,15 | 19,47 |
| 11 | 16,82 | 17,92 | 19,40 | 19,88 |
| 12 | 17,23 | 17,41 | 19,60 | 20,11 |
| 13 | 17,75 | 17,05 | 19,83 | 20,27 |
| 14 | 18,82 | 16,28 | 19,95 | 20,43 |
| 15 | 19,41 | 17,64 | 20,28 | 20,83 |
| 16 | 20,48 | 20,07 | 20,67 | 20,84 |
| 17 | 20,55 | 20,40 | 20,85 | 20,92 |
| 18 | 20,60 | 20,51 | 20,96 | 20,98 |
| 19 | 20,67 | 20,60 | 20,97 | 20,99 |
| 20 | 20,79 | 20,77 | 20,97 | 20,99 |

* Flame source combustion

The averages of variations of CO are given in Table 6. The highest increase in CO concentration was (822 ppm) observed in the treated with liquid nitrogen samples and the lowest in (363 ppm) treated with liquid nitrogen and impregnated samples.

Table 6. Variation of CO (ppm)

| Measured of time | Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|------------------|---------|------------------------------|--------------------------|--|
| 1* | 11 | 32 | 3 | 40 |
| 2* | 43 | 99 | 52 | 112 |
| 3* | 120 | 178 | 137 | 167 |
| 4* | 195 | 269 | 203 | 216 |
| 5* | 283 | 322 | 283 | 255 |
| 6* | 324 | 372 | 328 | 288 |
| 7* | 372 | 418 | 360 | 304 |
| 8* | 490 | 501 | 361 | 341 |
| 9 | 526 | 558 | 365 | 363 |
| 10 | 549 | 639 | 368 | 345 |
| 11 | 561 | 750 | 326 | 315 |
| 12 | 692 | 822 | 306 | 274 |
| 13 | 707 | 798 | 285 | 260 |

| | | | | |
|----|-----|-----|-----|-----|
| 14 | 664 | 728 | 238 | 242 |
| 15 | 596 | 603 | 206 | 210 |
| 16 | 513 | 534 | 161 | 188 |
| 17 | 322 | 413 | 96 | 115 |
| 18 | 256 | 299 | 65 | 77 |
| 19 | 192 | 209 | 38 | 49 |
| 20 | 105 | 136 | 16 | 26 |

* Flame source combustion

The averages of variation of NO are given in Table 7. In this study, the highest increase in NO concentration was observed in the experiment of (17.2 ppm) control samples and the lowest in those of (11,8 ppm) treated with impregnated samples.

Table 7. Variation of NO (ppm).

| Measured of time | Control | Treated with liquid nitrogen | Treated with impregnated | Treated with liquid nitrogen and impregnated |
|------------------|---------|------------------------------|--------------------------|--|
| 1* | 0,3 | 1,0 | 0,8 | 0,8 |
| 2* | 1,3 | 2,6 | 1,8 | 2,8 |
| 3* | 4,4 | 4,2 | 3,6 | 3,6 |
| 4* | 8,3 | 7,9 | 5,4 | 6,0 |
| 5* | 9,6 | 9,9 | 7,2 | 8,4 |
| 6* | 10,6 | 10,2 | 9,4 | 10,6 |
| 7* | 11,4 | 12,4 | 11,8 | 11,4 |
| 8* | 12,6 | 13,1 | 10,6 | 12,0 |
| 9 | 13,7 | 14,4 | 9,4 | 10,6 |
| 10 | 14,1 | 15,4 | 8,4 | 8,8 |
| 11 | 14,5 | 16,0 | 7,0 | 7,2 |
| 12 | 13,2 | 17,2 | 5,8 | 6,2 |
| 13 | 12,1 | 14,0 | 4,4 | 5,0 |
| 14 | 9,2 | 10,8 | 3,4 | 4,2 |
| 15 | 7,0 | 6,9 | 2,1 | 3,6 |
| 16 | 4,4 | 5,5 | 1,3 | 2,1 |
| 17 | 2,9 | 3,4 | 0,8 | 1,1 |
| 18 | 1,9 | 2,2 | 0,6 | 0,7 |
| 19 | 1,3 | 1,6 | 0,4 | 0,5 |
| 20 | 0,6 | 1,0 | 0,2 | 0,3 |

*Flame source combustion

Conclusion

The highest value was found in treated with liquid nitrogen samples at the end of flame source combustion (8th measurement or 4th minute). It was observed that mass reduction values in % occurred in the control samples 2% lower, treated with impregnated samples 49% lower and in the treated with liquid nitrogen-impregnated samples 51% lower compared with the treated with liquid nitrogen samples.

The highest value was found in treated with liquid nitrogen samples at the end of combustion. It was observed that mass reduction values in % occurred in the control samples 1% lower, treated with impregnated samples 63% lower and in the treated with liquid nitrogen-impregnated samples 68% lower compared with the treated with liquid nitrogen samples.

Take into account the temperature values occurred at the end of combustion tests. The highest values were found in the treated with liquid nitrogen samples while the lowest one was observed in the treated with liquid nitrogen-impregnated samples.

In such places, service life of industrial wood materials depends on retention ratio of impregnate chemicals. To use wood materials in exterior service, penetrating thickness on wood materials is very important. Impregnate chemicals which stay on surface of wood will be leached during time. This progress will cause wood material unprotected against to biological factors. Because of this reason, anatomical structure of wood material (wood cell) would change physically (by increasing volume of frozen liquid) by treating liquid nitrogen and penetration ratio would be easier and increased.

Consequently, treated with liquid nitrogen-impregnated was found to be the most successful according to the CO amounts and mass reduction. It can be proposed that protecting impregnating of historical wooden structure due to fire risk.

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