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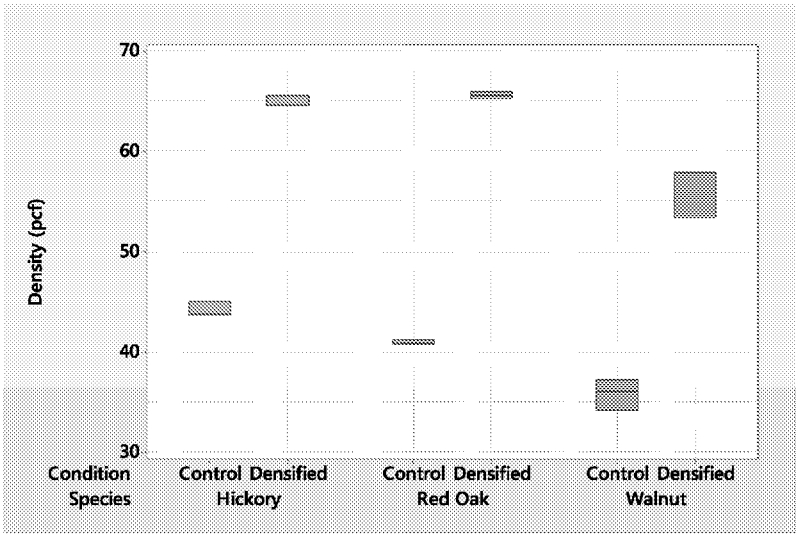
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(54) Title: DENSIFIED WOOD INCLUDING PROCESS FOR PREPARATION

FIG 1.




(57) Abstract: A wood substrate or member is included, having an increased density with respect to natural, untreated wood. The process includes drying the wood prior to application of heat and pressure, which are controlled to reduce or eliminate color change on a surface of the wood member where heat and pressure are applied.

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# DENSIFIED WOOD INCLUDING PROCESS FOR PREPARATION

## BACKGROUND OF THE INVENTION:

### Field of the Invention

The invention includes a compressed wood member providing dimensional stability and not destroying or crushing the cellular structure of untreated wood. The process includes compression of wood after specific conditioning to increase the density of the wood member.

### Summary of Related Art

There have been numerous attempts to improve properties of wood by increasing the density. Each of these processes focuses on maintaining or increasing the moisture content of the wood and/or are limited to veneer thicknesses.

US Patent No. 7,404,422 to Kamke et al. includes a process of increasing the temperature and moisture content of wood followed by mechanosorption, i.e., rapid movement of water out of the wood cell wall. The process is conducted with lower density veneers or composite panels.

Diouf et al. also describe a process for increasing the density of wood veneers in "Effects of thermo-hygro-mechanical densification on the surface characteristics of trembling aspen and hybrid poplar wood veneers." Applied Surface Science, vol. 257, issue 8, February 1, 2011, p. 3558. The process uses thermo-hygro-mechanical densification, which includes the introduction of heat, steam and pressure to increase density. The article notes that a significant color change in the wood is observed above 200°C (approx. 390°F).

Arruda et al. describe another process for increasing wood density in "Utilization of a Thermomechanical Process to Enchance Properties of Hardwood Used for Flooring." Ciencia da Madeira, Brazillian Journal of Wood Science, vol. 6, no. 3, (2015). These processes were conducted on 30mm x 30mm square wood samples having a thickness of 20 to 25mm. There was no pre-treatment of the samples which are subjected to both heat and pressure. Before

treatment, the samples had a before treatment moisture content of 9.49% to 12.48% and an after treatment moisture content of 5.36% to 9.36% depending on the sample.

## SUMMARY OF THE INVENTION:

One common feature of previous attempts to successfully increase the density of wood is the required introduction of water to increase and/or maintain a high moisture content in the wood prior to compression. The inventors have noted that as heat is applied in the prior processes, the water becomes steam, which is trapped in the porous structure of the wood. As the steam escapes, the cellular structure of the wood is damaged.

The issues of cellular structure damage and darkening of the wood surface have been resolved by the process provided herein. The result of this process is a treated wood member having a density about 10% to about 150% greater than untreated wood wherein the cellular structure of the wood is substantially intact. Furthermore, the treated wood member exhibits dimensional stability with a significant reduction or elimination of the darkening of the wood surface, which resulted from previous processes.

A process for preparing a treated wood member having an increased density includes:

(a) providing a wood member having a moisture content less than about 19%;

(b) preheating the wood member;

(c) optionally, applying water to a surface of the wood member;

(d) applying pressure to the wood member for a press time; and

(e) providing a post-treatment conditioning of the wood member.

## BRIEF DESCRIPTION OF THE DRAWINGS:

FIG. 1 is a chart showing comparison of density for similar species.

FIG. 2 is a chart showing the Dry Side Gap during the time after treatment.

FIG. 3 is a chart demonstrating the vertical density profile.

**DETAILED DESCRIPTION OF THE INVENTION:**

The process for preparing a treated wood member having an increased density is useful with a variety of different wood species. For example, the process may be used with either hardwood or softwood. However, the increase in density will depend on various factors including the original, untreated density of the wood, as well as other factors. Any of a variety of different wood species may be included. Suitable examples include, but are not limited to Red Oak, White Oak, Hickory, Walnut, Aspen, Basswood, Maple, Poplar, Pine, Cherry, and Ash.

The treated wood member will have advantages over untreated wood, such as an increased dimensional stability throughout a variety of temperature and humidity conditions, increased strength, and especially important for a flooring application, resistance to denting, which is a current drawback of existing wood floors.

The treated wood member will have advantages over untreated wood, including an increased dimensional stability throughout a variety of temperature and humidity conditions, increased strength, and/or increase resistance to denting. Susceptibility to denting and other physical deformation is a well-know drawback of wood products in almost any application, including, for example, flooring, construction, cabinetry, moldings, finishes, counter tops, furniture, walls, ceilings, decking. Tilus, the improved properties of the treated wood member of the present process make it useful for any application in which a wood surface is left exposed or is subject to physical insult

When the treated wood is used in flooring, it may be used by itself, as a solid hard wood floor or as a component of flooring such as a veneer for engineered hardwood, laminate, or any other core material, such as plastic-based flooring substrates. The flooring may also include a tongue in groove, connection or a locking profile, many of which are known to those of skill in the art.

Depending on the species and type of wood being subjected to the present process, the increase in densification may be about 10% to about 150% greater than untreated wood. This includes an about 40% to about 100% comparative density increase. The density increase is typically uniform throughout the thickness of the wood. This can be observed by the vertical density profile. The density of the treated wood will typically be greater than 50 pcf (pound per cubic foot). Suitable densities may be about 50 pcf to about 85 pcf, or about 60 pcf to about 70 pcf.

The thickness of the treated wood will be less than the untreated wood. For example, the treated wood may have a thickness that is about 30% to about 70% less than untreated wood. The treated wood member may have a thickness greater than about 0.025 in, such as greater than about 0.25 in, including a range of about 0.5 in to about 8 in, and about 0.5 in to about 3 in. Various other suitable thicknesses may also be provided.

The color change of the top and bottom surfaces of the treated wood member may be significantly less than that seen with previous processes. For example, the surface color difference ($\Delta E$) of a treated wood member may be less than 20 compared to a surface color of untreated wood. In order to provide a minimal color change, the process/press temperature may be reduced to about 400°F.

Other benefits have been observed by the densification process. The present process reduces the bowing, cupping, and other lumber defects (e.g., distortion out of a flat plane) after the process is completed. The process has been observed to fuse knots that were loose prior to pressing. Also, after pressing the surface of the material is "smooth" compared to untreated material. Saw marks, rough fiber, and other surface imperfections are pressed smooth by the present process.

One significant difference between the current and previous processes is the pre-treatment of the wood to reduce the moisture content (MC). Previous attempts to increase density of wood did not reduce the MC of wood prior to compression.

The overall process includes (a) providing a wood member having a moisture content (MC) less than about 19%; (b) preheating; (c) optionally applying surface water; (d) applying pressure; and optionally cooling the treated wood member prior to (e) providing post-treatment conditioning.

Providing reduced MC wood member (Initial moisture content of wood)

The wood member to which the present treatment is applied has a reduced moisture content (MC) compared to green wood. The reduction in MC may be achieved by any suitable method such as treatment in a kiln. The MC of the wood member will be less than about 19% or less than about 15%. Suitable ranges include MC of about 3% to about 19%, about 5% to about 15%, about 5% to about 12%, and about 5% to about 10%.

Pre-heating

The pre-heating step may also be conducted by any suitable method. The pre-heating step is included to increase the temperature of the wood throughout the thickness of the wood and may also result in a further MC reduction. The preheating may be conducted in any suitable temperature range including about 250°F to about 500°F. The preheating should be applied for a sufficient time to obtain a core temperature of about 250°F to about 500°F. The MC of the wood member may about 5% or less at the completion of the preheating, such as about 2% to about 5%

Optional water application

Water may be applied to the surface only of the wood member, if desired. If used, the water may be applied in the form of liquid or steam. The addition of water may provide additional resistance and/or reduction of cupping and/or bowing in the wood member.

Applying Pressure

During the application of pressure, heat may also be applied to maintain the preheated core temperature of the wood member. The pressure may be applied by any known device

such as a heated platen press, a continuous press, a series of mills, or a combination thereof. The pressing time may be about 10 seconds to about 60 minutes about 30 seconds to about 10 min. The pressure may be selected based on the species of wood and desired increase in density. Suitable ranges of pressures include about 500psi to about 5000 psi, such as about 1500 psi to about 3500 psi. The pressing process may include a single press cycle or multiple press cycles. If desired, the wood member may be cooled after pressing.

Post-treatment conditioning

The post-treatment conditioning is important to maintaining the dimensional stability of the wood member. The post treatment may include the introduction of steam, humidity, heat, or combinations thereof. The post-treatment conditioning should raise the MC of the wood member. The MC may be that of any suitable construction material and may depend on the desired end use of the wood member. For example, the MC may be about 5 to about 10%, or about 7% to about 8%.

The features and advantages of the present invention are more fully shown by the following examples which are provided for purposes of illustration, and are not to be construed as limiting the invention in any way.

**Examples:**

Color change of the material appearance

- Conditions
  - Control
  - 1.5 hour preheat at 375 F
  - 1.5 hour preheat at 475 F
  - Control
  - 1.5 hour preheat at 375 F and 4 minute pressing 375F and 3000 psi
  - 1.5 hour preheat at 425 F and 4 minute pressing 425F and 3000 psi
  - Three samples per board. Each group had untreated controls
  - Three replicates per condition
  - Measured the color change on both top and bottom face of the specimen using X-Rite Model SP64 Spectrometer
  - $\Delta E^*_{ab} = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$
- Material

- Red Oak
- Hickory
- 13" X 5.5" 4/4 Lumber

The color change table below indicates a greater color change for processing at 425F, compared to 375F.

| Color change table | | |
|---|---|---|
| Species and conditions | Top - ΔE | Bottom - ΔE |
| HICKORY - Control No Preheat/No Press | | |
| HICKORY - Preheat/Press 375/375/4min. | 3.645698 | 1.943353852 |
| HICKORY - Preheat/Press 425/425/4min. | 9.478496 | 8.378549736 |
| | | |
| HICKORY - Control No Preheat/No Press | | |
| HICKORY - Preheat 375/No Press | 2.637723 | 2.668513909 |
| HICKORY - Preheat 425/No Press | 5.336167 | 5.013005495 |
| | | |
| Red Oak - Control No Preheat/ No Press | | |
| Red Oak - Preheat/Press 375/375/4min. | 8.931624 | 6.42765546 |
| Red Oak - Preheat/Press 425/425/4min. | 21.99234 | 21.15111906 |
| | | |
| Red Oak - Control No Preheat/ No Press | | |
| Red Oak - Preheat 375/No Press | 2.571813 | 4.201487458 |

| | | |
|---|---|---|
| Red Oak - Preheat 425/No Press | 11.85161 | 7.752267843 |

Impact Resistance
Material

- Red Oak, Walnut, Hickory and Basswood
- 15" ball drop with a mass of 16 ounces
- Maximum depth measured with a Mitutoyo depth gauge
- 3 replicates per group
- 6 drops per replicate (specimen)

The table, below, indicates that a greater resistance to denting results from an increase in pressure, i.e., increase in density.

| Impact Resistance Table | | |
|---|---|---|
| Species | Pressure (psi) | Average Depth (inches) |
| Red Oak | control | 0.01997 |
| Red Oak | 1,529 | 0.00683 |
| Red Oak | 2,038 | 0.00622 |
| Red Oak | 2,548 | 0.00406 |
| Red Oak | 3,058 | 0.00372 |
| Walnut | control | 0.02525 |
| Walnut | 1,154 | 0.00813 |
| Walnut | 1,538 | 0.01363 |
| Walnut | 1,923 | 0.00654 |
| Hickory | control | 0.01058 |
| Hickory | 1,529 | 0.00753 |
| Hickory | 2,038 | 0.00744 |
| Hickory | 2,548 | 0.00489 |
| Hickory | 3,058 | 0.00506 |
| Basswood | Control | 0.03781 |
| Basswood | 769 | 0.01044 |
| Basswood | 1,019 | 0.00719 |
| Basswood | 1,154 | 0.01531 |
| Basswood | 1,529 | 0.00686 |

FIG. 1 demonstrates the density increase (Mass/(Width, Length & Thickness)) for several treated samples and controls.

Environmental Chambers – 4.6% EMC Conditions

Red Oak control and densified 5/8" X 5" X RL material were machined and finished into finished flooring. This material was installed using mechanical fasteners to an already conditioned to 4.6 MC OSB subfloor in the environmental chamber. Measurements were taken using a set of feeler gauges at the side seams of joints of the flooring planks. This was completed initially, after 1 day, 7 day, 14 day and 21 day (28 day data is being collected today and tomorrow). The results can be seen in FIG. 2.

Vertical Density Profile (VDP)

2" X 2" specimens were measured for the density through the thickness using an X-Ray analyzer to measure density at .001" frequency through the thickness of the material. Quintek Measurement System Model QDP-01X was used for measuring VDP. FIG 3 shows the reduction in variation in the VDP for Red Oak and Hickory.

Janka Testing

Resistance to denting was measure by a test commonly referred to as the Janka test. According to ASTM D143-14, the amount of force was measured as needed to embed a 0.444" steel ball to half of its diameter into a wood sample. The densified values represent the average of six sample and the control values represent the published values from the Wood Handbook. The Janka values in the table, below demonstrate that significantly more force was required to dent the densified samples

| Wood Type | Janka Ball value (lb-f) |
|---|---|
| Densified Hickory | 3687 |
| Densified Red Oak | 3228 |
| Brazilian Cherry - Control | 2350 |
| Mesquite- Control | 2345 |
| Hickory- Control | 1820 |
| Hard Maple- Control | 1450 |
| White Oak- Control | 1360 |
| Ash- Control | 1320 |
| Red Oak- Control | 1290 |
| Black Walnut- Control | 1010 |
| Black Cherry- Control | 950 |

While there have been described what are presently believed to be the preferred embodiments of the invention, those skilled in the art will realize that changes and modifications may be

made thereto without departing from the spirit of the invention, and it is intended to include all such changes and modifications as fall within the true scope of the invention.

**What is claimed is:**

1. A treated wood member comprising a density about 10% to about 150% greater than untreated wood wherein the cellular structure of the wood is substantially intact.

2. The treated wood member of claim 1, wherein the density is about 40% to about 100% greater than untreated wood.

3. The treated wood member of claim 1, wherein the density of the treated wood member has been increased throughout a thickness of said wood member.

4. The treated wood member of claim 1, having a thickness that is 30% to about 70% less than the untreated wood.

5. The treated wood member of claim 1, wherein said member has a surface color difference ($\Delta E$) less than 20 compared to a surface color of untreated wood.

6. The treated wood member of claim 1, wherein said member has density greater than about 50 pcf ($lbs/ft^3$).

7. The treated wood member of claim 6, wherein said member has a density about 50pcf to about 85pcf.

8. The treated wood member of claim 6, wherein said member has a density about 60pcf to about 70 pcf.

9. The treated wood member of claim 1, wherein said treated wood member is a hardwood or softwood.

10. The treated wood member of claim 1, wherein the treated wood thickness is greater than about 0.025 in.

11. The treated wood member of claim 1, wherein the treated wood thickness is greater than about 0.25 in.

12. The treated wood member of claim 1, wherein the treated wood thickness is about 0.5 in to about 8 in.

13. The treated wood member of claim 1, wherein the treated wood thickness is about 0.5 in to about 3 in.

14. The treated wood member of claim 1, further comprising a core, wherein the treated wood member is a veneer.

15. Flooring comprising the treated wood member of claim 1.

16. A method for increasing the density of a wood member including:

(a) providing a wood member having a moisture content less than about 19%;

(b) preheating the wood member;

(c) optionally, applying water to a surface of the wood member;

(d) applying pressure to the wood member for a press time; and

(e) providing a post-treatment conditioning of the wood member.

17. The method of claim 16, further comprising:

cooling the wood member before the post-treatment conditioning.

18. The method of claim 16, wherein the post-treatment conditions includes the introduction of steam, humidity, heat or combinations thereof.

19. The method of claim 16, wherein the preheating is conducted at temperature of about 250°F to about 500°F.

20. The method of claim 16, wherein the preheating is for a time sufficient to obtain a temperature for a core of the wood member to about 250°F to about 500°F and a moisture content of 0 to about 5%.

21. The method of claim 16, wherein heat is applied during pressing to maintain a pre-heat temperature.

22. The method of claim 16, wherein the moisture content of the wood prior to pre-heating is about 0 to about 10%.

23. The method of claim 16, wherein said pressure is about 500 psi to about 5,000 psi.

24. The method of claim 16, wherein the density of the wood member is increase by about 10% to about 150%.

25. The method of claim 16, wherein the water is applied in the form of steam to the surface of the member.

26. The method of claim 16, wherein said press time is about 10 seconds to about 60 minutes.

27. The method of claim 16, wherein the applying pressure is conducted with a heated platen press, a continuous press, a series of mills, or a combination thereof.

28. The method of claim 16, wherein the applying pressure is a single press cycle or multiple press cycles.

FIG 1.

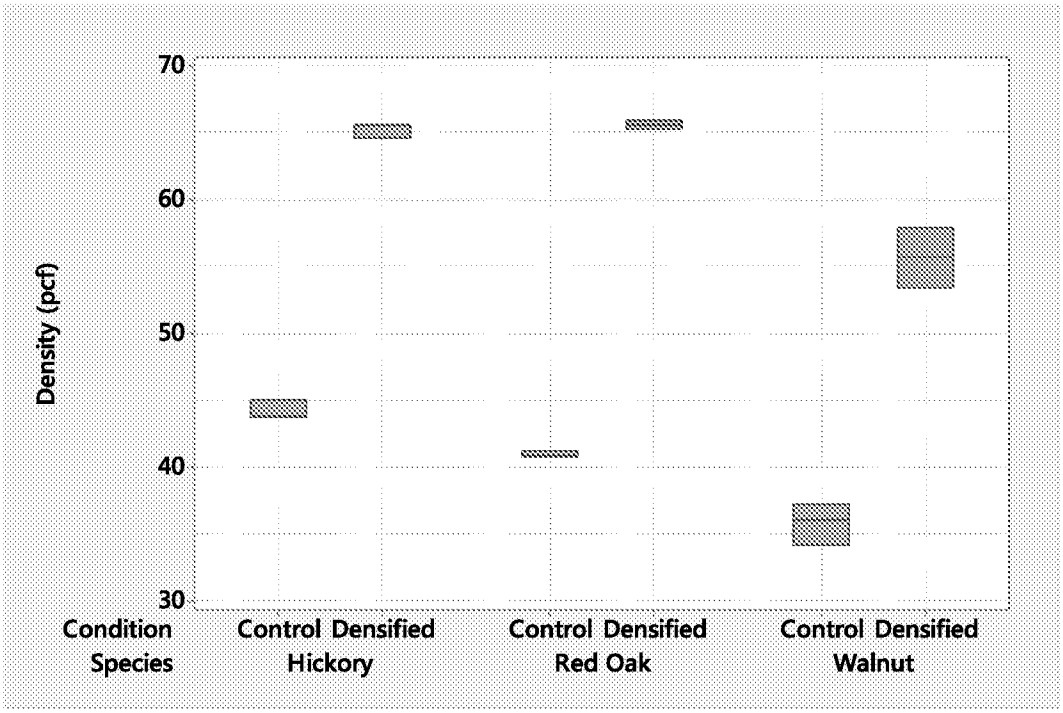

70
60
50
40
30
Density (pcf)
Condition
Species
Control Densified
Hickory
Control Densified
Red Oak
Control Densified
Walnut

FIG. 2

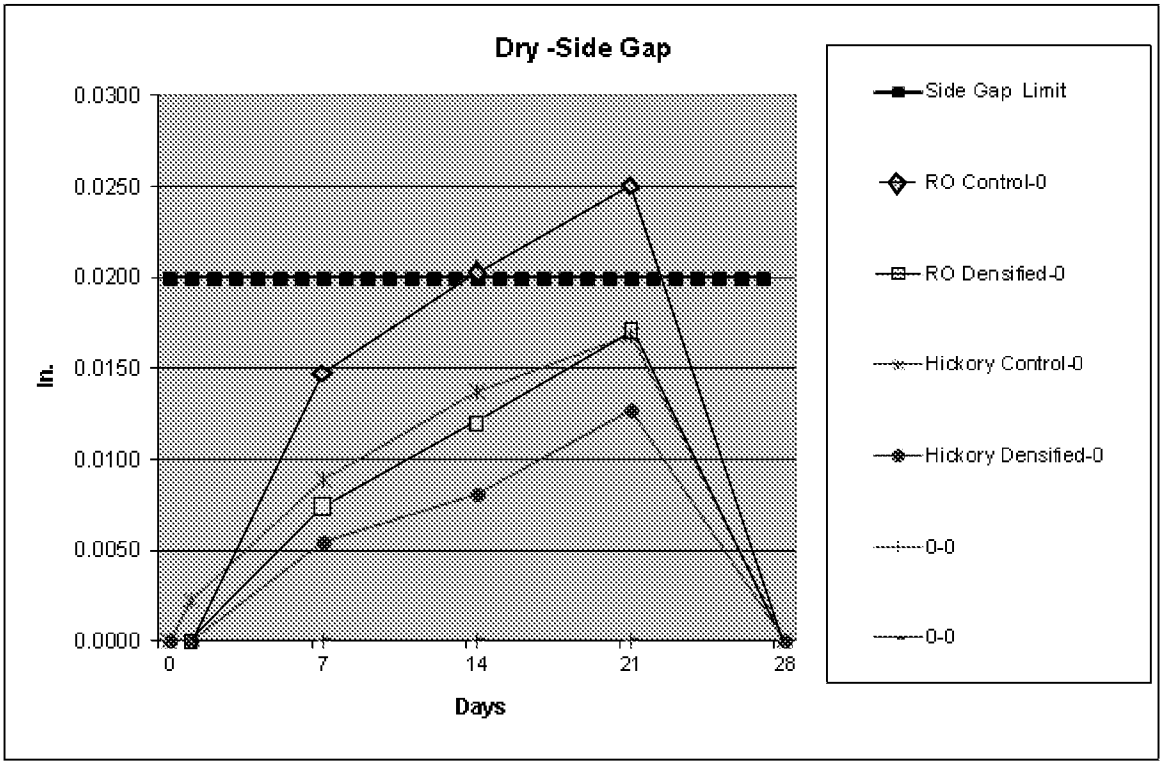
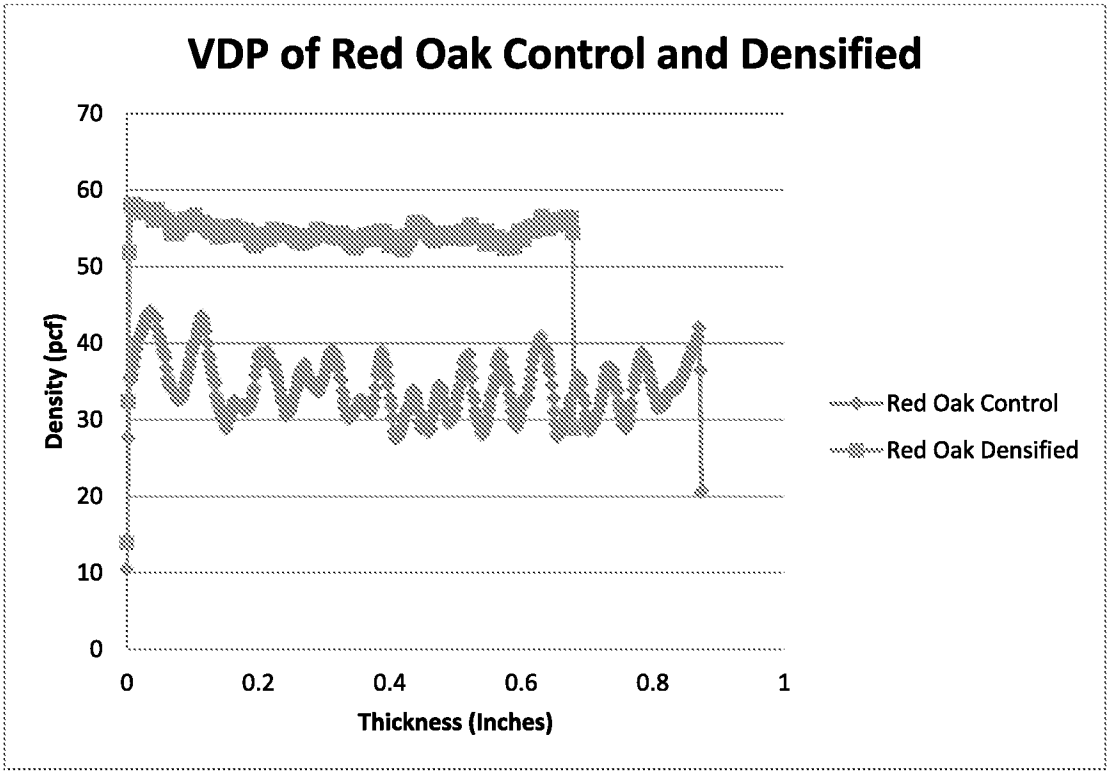
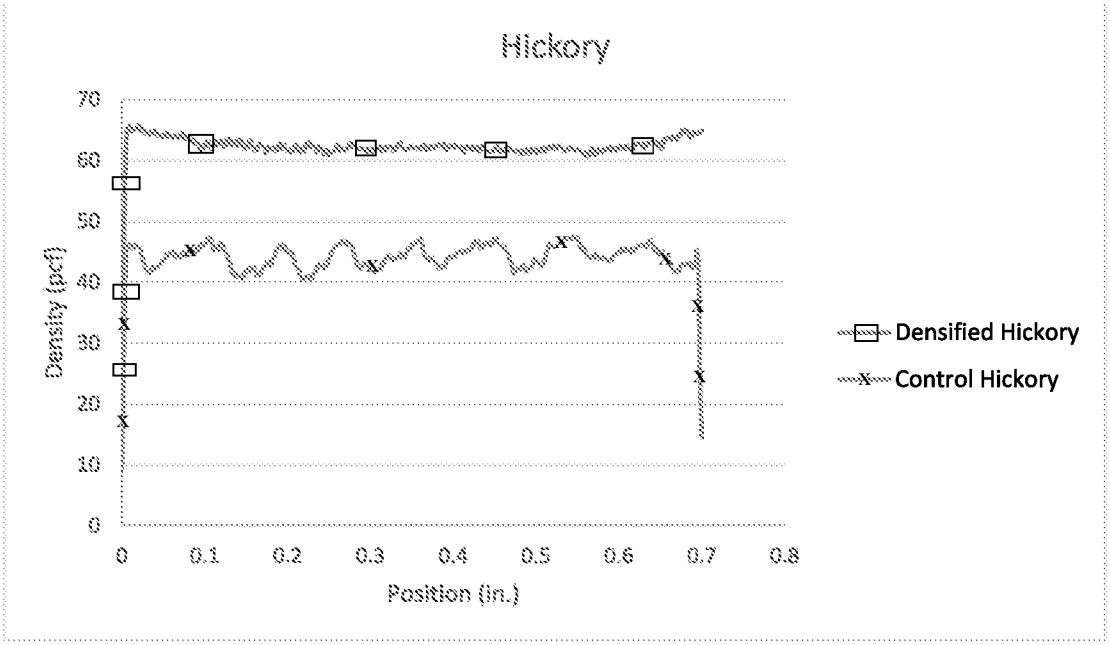

Dry -Side Gap
0.0300
0.0250
0.0200
0.0150
0.0100
0.0050
0.0000
In.
0
7
14
21
28
Days
Side Gap Limit
RO Control-0
RO Densified-0
Hickory Control-0
Hickory Densified-0
0-0
0-0

FIG. 3


VDP of Red Oak Control and Densified
Density (pcf)
70
60
50
40
30
20
10
0
0
0.2
0.4
0.6
0.8
1
Thickness (Inches)
Red Oak Control
Red Oak Densified


Hickory
Density (pcf)
70
60
50
40
30
20
10
0
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
Position (in.)
Densified Hickory
Control Hickory

# INTERNATIONAL SEARCH REPORT

International application No.
PCT/US18/67849

A. CLASSIFICATION OF SUBJECT MATTER

IPC - B27K 5/00, 5/06; B27N 3/00, 3/08 (2019.01)

CPC - B27K 5/00, 5/0085, 5/06; B27N 3/00, 3/08, 3/086

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

See Search History document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

See Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X<br>--<br>Y | US 2016/0039113 A1 (ARMSTRONG WORLD INDUSTRIES, INC.) 11 February 2016; abstract; paragraphs [0002], [0008]-[0009], [0011], [0017], [0024], [0027]-[0028], [0030], [0035], [0040]-[0042] | 1, 3-4, 9-10, 14-15<br>--<br>2, 5-8, 11-13 |
| X<br>--<br>Y | US 2011/0262727 A1 (TU, D et al.) 27 October 2011; paragraphs [0017], [0024]-[0025], [0033], [0035], [0069] | 16-18, 23-24, 26-28<br>--<br>21-22, 25 |
| X<br>--<br>Y | CN 103,753,664 A (RES INST WOOD INC CAF) 30 April 2014; see machine translation; abstract; claims 1, 5; paragraphs [0008], [0025], [0029] | 16, 19<br>--<br>20 |
| Y | (BLOMBERG, et al.) Effects of semi-isostatic densification of wood on the variation in strength properties with density. Wood Science and Technology, Vol. 39, No. 5, pp. 339-350. 01 August 2005; abstract; page 341 | 2, 6-8 |
| Y | WO 2014/160938 A1 (ARMSTRONG WORLD INDUSTRIES, INC) 02 October 2014; paragraphs [0030], [0038]-[0039], [0045]; table 3 | 5, 11-13 |
| Y | CN 107,414,981 A (RES INSTITUTE OF WOOD INDUSTRY CHINESE ACADEMY OF FORESTRY) 01 December 2017; see machine translation; abstract; paragraphs [0005], [0031]-[0032] | 20 |
| Y | US 7,404,422 B2 (KAMKE, FA et al.) 29 July 2008; column 5, lines 26-32, 40-44; column 6, lines 63-67; column 7, lines 5-12; column 11, lines 30-35 | 21, 25 |

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 14 February 2019 (14.02.2019) | 04 MAR 2019 |

| Name and mailing address of the ISA/ | Authorized officer |
|---|---|
| Mail Stop PCT, Attn: ISA/US, Commissioner for Patents<br>P.O. Box 1450, Alexandria, Virginia 22313-1450<br>Facsimile No. 571-273-8300 | Shane Thomas<br>PCT Helpdesk: 571-272-4300<br>PCT OSP: 571-272-7774 |

## INTERNATIONAL SEARCH REPORT

| International application No. |
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| PCT/US18/67849 |

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
| --- | --- | --- |
| Y | US 7,258,761 B2 (LIU, F et al.) 21 August 2007; column 3, lines 41-47; column 6, lines 8-10; column 7, lines 34-36 | 22 |