J. Korean Wood Sci. Technol. 2020, 48(6): 780~790
pISSN: 1017-0715 eISSN: 2233-7180
https://doi.org/10.5658/WOOD.2020.48.6.780

Original Article

Evaluation of Cell-Wall Microstructure and Anti-Swelling Effectiveness of Heat-Treated Larch Wood[1]

Yonggun PARK[2] · Woo-Seok JEON[2] · Sae-Min YOON[2] ·
Hyun Mi LEE[2] · Won-Joung HWANG[2,†]

ABSTRACT

In this study, the cell-wall microstructure and anti-swelling effectiveness (ASE) of heat-treated larch wood were evaluated and the correlation between them was analyzed. For this purpose, some larch lumbers were heat-treated for 12, 18, and 24 hours at temperatures of 190°C and 220°C. By observing the scanning electron microscopy cross-sectional image of the heat-treated larch, it was confirmed that the shape of heat-treated wood cell changed, the cut-section of the wood cell wall was rough, and the intercellular space has become wide as the intercellular bonds had broken because of heat-treatment. In addition, the evaluation of the swelling for each treatment condition revealed that, as the heat-treatment temperature and duration increased, the amount of absorbed water and swelling decreased and the ASE increased. The decrease in the amount of absorbed water is thought to be affected by the chemical change in the cell wall by heat-treatment. On the contrary, the decrease in the swelling and the increase in the ASE are thought to be due to a combination of chemical changes and physical changes such as structural changes in the cell wall.

Keywords: heat-treatment, cell wall microstructure, anti-swelling effectiveness, amount of absorbed water, dimensional stability

1. INTRODUCTION

As a result of an increasing interest in the environment, such as climate change and certified emission reduction (CER), both in Korea and around the world, the attention and demand for wood, which is an eco-friendly and sustainable material, are increasing as well. Unlike other materials, wood has higher specific strength, is easy to process (including cutting and bonding), and acts as an insulator against electricity, sound, and heat. However, wood has its weakness in that it changes in size and/or decays when exposed to humid conditions. Because wood is a hygroscopic material, it always contains some moisture in its natural state. The moisture in the wood affects its properties; in particular, itis directly involved in the dimensional change of wood. In other words, when the amount of moisture in the wood increases, the wood swells, and when it decreases, the wood shrinks. This change in the dimension of wood serves as a disadvantagethat hampers the structural stability of the wood in use. Therefore, there have been a variety of

---

[1] Date Received July 29, 2020, Date Accepted September 7, 2020

[2] Timber Engineering Division, Forest Products Department, National Institute of Forest Science, 57 Hoegi-ro, Dongdaemun-gu, Seoul 02455, Republic of Korea

† Corresponding author: Won-Joung HWANG (e-mail: wonjoung@korea.kr, ORCID: 0000-0001-6848-5778)

studies to reduce these dimension changes of wood in accordance with its moisture content.

Heat-treatment of wood is a process that involves applying heat ranging 160–260°C to improve its physico-mechanical properties and resistance against wood rot fungi (Militz, 2002; Park *et al.*, 2014). Various heat-treatment methods have been studied and their effects have been evaluated (mainly in Europe) because of the advantages of improved durability in an environmentally friendly method. Numerous studies have reported that heat-treatedwood has enhanced hydrophobicity, which leads to improved dimensional stability, darkened surface color, and increased decay resistance against various fungi, bacteria, and insects. (Cho *et al*., 2015; Esteves *et al*., 2008; Kang *et al*., 2018; 2019; Kim, 2016; Kim and Kim, 2019; Kim *et al*., 2018; Lee and Lee, 2018; Park *et al*., 2012; 2014; 2015; 2016; 2018; Repellin and Guyonnet, 2005; Yoon *et al*., 2008; 2009). These characteristic changes enable the application of heat-treated wood for different uses that require dimensional stability or biological durability such as outdoor furniture, fences, columns, cabinet, window frame, doors, decks, exterior, and floors (Boonstra *et al*., 1998; Esteves and Pereira, 2009; Kim, 2016).

The changes in wood properties due to heat-treatment depend on the temperature, duration, and heat-transfer media of heat-treatment (Kim, 2016). Specifically, as the heat-treatmenttemperature and duration are increased, the effect of heat-treatment improves. However, the relationship between the temperature or duration and the effect of heat-treatment is not linear; this makes it exceedingly difficult to identify heat-treatment conditions (temperature, duration, etc.) that result in specific material properties. Accordingly, a lot of time and energy is used in the process of identifying the suitable heat-treatment conditions to achieve properties that meet the purposes of using heat-treated wood. To this end, this study aimed for presenting the measurements of swelling and anti-swelling effectiveness (ASE) in larch wood, which accounts for the majority of artificial coniferous forests in Korea, as basic database. In addition, this study attempted to analyze the causes of the changes in swellings by heat-treatment through observation of the cell-wall microstructure. To achieve this, changes in the cell-wall microstructure of larch wood heat-treated at 190°C and 220°C for 12, 18, and 24 hours were observed through scanning electron microscopy (SEM), and the amount of absorbed water, swelling, and ASE of the heat-treated wood were measured.

2. MATERIALS and METHODS

2.1. Materials

For this study, the lumber of larch wood (*Larix kaempferi*) that was sawn and dried in the Jungbu Lumber Distribution Center (Yeoju-si, Gyeonggi-do) of the National Forestry Cooperative Federation was purchased. The dimensions of the larch lumber were 50 mm (thickness; radial direction) × 150 mm (width; tangential direction) × 2,400 mm (length; longitudinal direction).

2.2. Heat-treatment of wood

As shown in Fig. 1, depending on the heat-treatment duration, 2,400-mm long larch lumber was divided and cut into four sets: non-treated, 12-hour heat-treatment, 18-hour heat-treatment, and 24-hour heat-treatment. During heat-treatment performed by putting the other threesets excluding the non-treated set into the reactor, the experiment was conducted in a method such that each set was taken out from the reactor in accordance with the heat-treatment duration. The heat-treatment temperature was set to two conditions of 190°C and 220°C, and 12 lumbers were used for each heat-treatment temperature.

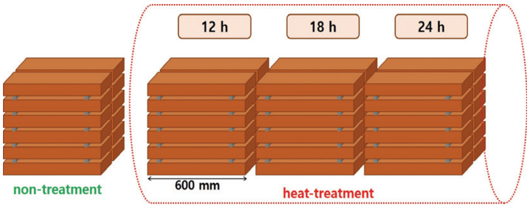

Fig. 1. Four sets of larch lumber for heat-treatment.

2.3. Evaluation of changes in cell-wall microstructure

Changes in the cell-wall microstructure of wood by heat-treatment were evaluated. Some cubical specimens with a side length of 5 mm were made to avoid any knots or cracks from the middle of the lumber. The specimens were softened and oven-dried to remove moisture. Then, the specimens were coated with gold and their cell-wall microstructures were observed through 20-kV SEM (SEMCOX EM-30, COXEM, Korea).

2.4. Evaluation of anti-swelling effectiveness

The ASE of the heat-treated larch wood was measured according to the ASTM D4446 “Anti-Swelling Effectiveness of Water-Repellent Formulations and Differential Swelling of Non-treatedWood When Exposed to Liquid Water Environments.” However, because ASTM D4446 is for determining the ASE for different coating applications on wood specimens, the coating applications were replaced with heat-treatment conditions for this study. The specimen was made in the dimensions of 127 mm (length; tangential direction) × 38 mm (width; radial direction) × 6 mm (thickness; longitudinal direction) from the middle of the lumber and ten specimens were used for each heat-treatment condition. The specimens were humidified in a room at a constant temperature of 23±2°C and a relative humidity of 50±5% until they reached the constant weight. The humidified specimen was immersed in distilled water for 30 minutes to absorb water and swell as shown in Fig. 2. The weight change in the specimen before and after immersion was measured to determine the amount of water absorbed by the specimen, and the tangential length of the specimen before and after immersion was measured using a swellometer to determine the swelling (β). In addition, the ASE was calculated using Equation (1) with swelling measurements of non-treated and heat-treated larch wood in each condition to quantify the effects of heat-treatment on the changes in wood dimensions.

Fig. 2. Experiment for the anti-swelling effectiveness.

$$ASE(\%) = \frac{\beta_n - \beta_h}{\beta_n} \times 100 \qquad (1)$$

where, β_n indicates tangential swelling (mm) of non-treated wood, and β_h indicates tangential swelling (mm) of heat-treated wood.

3. RESULTS and DISCUSSION

3.1. Heat-treatment of Wood

Figs. 3 and 4 show the graphs of temperature changes for the surface and core of the wood during heat-treatment at 190°C and 220°C, respectively. At

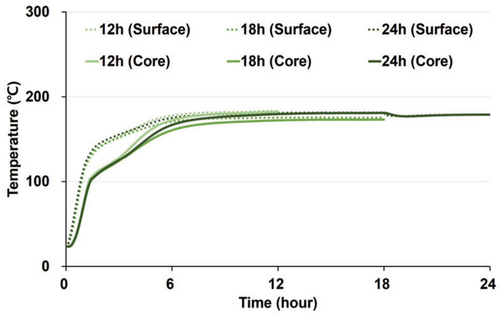

Fig. 3. Change in wood temperature during the heat-treatment at 190°C.

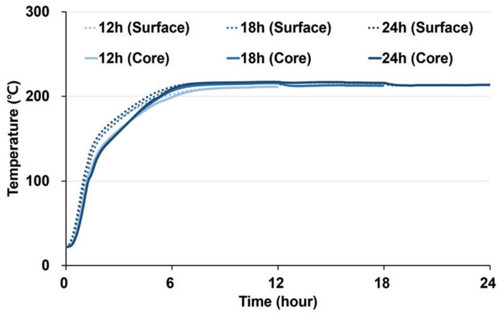

Fig. 4. Change in wood temperature during the heat-treatment at 220°C.

the 6-hours into the process at both temperature conditions, the temperature on the surface and in the core reached the target temperature of 190°C and 220°C, and the temperature remained constant until the end of the process. The temperature difference between the surface and core did not vary significantly; thus, it is thought that the effect of heat-treatment would be similar for all the wood specimens. The moisture content was 0% at the end of heat-treatment.

3.2. Evaluation of changes in cell-wall microstructure

Fig. 5 shows the 1,000-magnified cross-sectional SEM images of the non-treatedand heat-treated larch wood at 190°C and 220°C. For non-treated wood, the thick S2 layer in the cell wall was cut smoothly, while the ones in heat-treated wood specimens were cut roughly. Moreover, the cells were deformed out of the original shapes with breakages and dents, and the intercellular spaces were widened as though the cells were disconnected. These tendencies were more remarkable in the larch wood that was heat-treated at 220°C than in those heat-treated at 190°C. It is known that heat-treatment of wood results in the decomposition of hemicellulose, which is one of the major elements of cell walls,and the mass decreases as heat-treatment is continued (Kim, 2016; Park *et al*., 2017; Welzbacher *et al*., 2009). This pyrolysis of the major element of wood cell walls weakens the cell walls, causing rough cutting surfaces. Moreover, as hemicellulose is decomposed, the number of hydroxyl groups decreases. Accordingly, it is considered that the hydrogen bonds between cells weakened, disconnecting cells from each other, eventually widening the intercellular spaces.

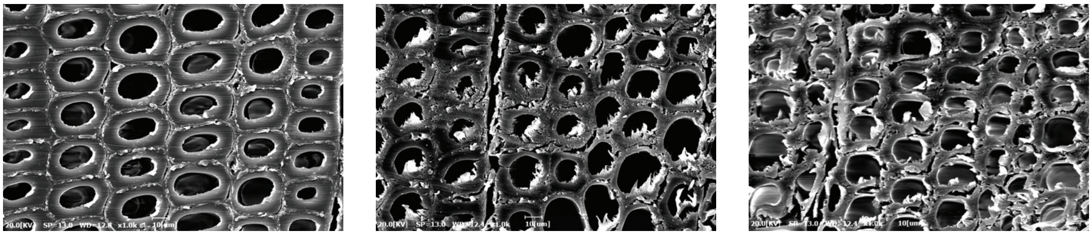
(A) Non-treated (B) Heat-treated at 190°C (C) Heat-treated at 220°C

Fig. 5. SEM images of cross-section for the treated larch wood (×1,000).

3.3. Evaluation of anti–swelling effectiveness

Fig. 6 shows the results for the amount of absorbed water, swelling, and ASE measurements for different heat-treatment conditions. Table 1 shows moisture content of specimens before and after immersion. Both amount of absorbed water and swelling measurements tended to decrease as the heat-treatment temperature and duration increased. In heat-treated wood, the number of hydroxyl groups decreased, increasing the hydrophobicity (Esteves and Pereira, 2009; Kim, 2016; Park *et al.*, 2014; 2015; 2016). Therefore, it seemed that as heat-treatment continued, the increasing hydrophobicity reduced the amount of absorbed water, which in turn decreased the swelling.

However, although the amount of absorbed water showed a tendency to decrease continuously as heat-treatment continued, the swelling did not. The amount of absorbed water was largely affected by the chemical changes in wood caused by heat-treatment, while swelling was affected by the chemical changes as well as by physical changes such as the cell-wall microstructure. When wood is heat-treated, the amorphous regions of cellulose (where the moisture can penetrate into and cause swelling) decompose (Esteves and Pereira, 2009; Kim, 2016). Furthermore, if moisture penetrates the gaps (that have been widened due to heat-treatment) between the cells, the cell walls do not swell. This indicates that swelling is influenced by a

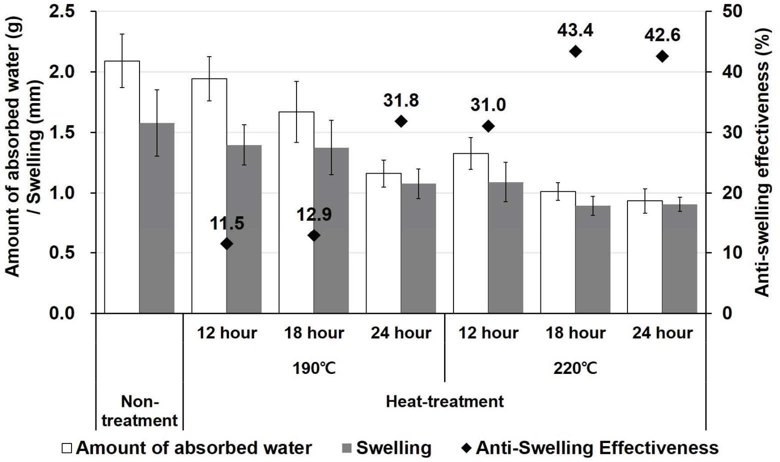

Fig. 6. Absorbed water, swelling, and anti-swelling efficiency (ASE) depending on the treatments.

Table 1. Changes and difference in moisture contents of specimen before and after swelling experiment

	Non-treatment	Heat-treatment					
		190°C			220°C		
		12 hour	18 hour	24 hour	12 hour	18 hour	24 hour
Before	8.5 %	7.3 %	7.0 %	6.6 %	6.5 %	5.6 %	5.1 %
After	24.6 %	22.9 %	19.6 %	15.8 %	17.6 %	14.7 %	13.9 %
Difference	16.1 %	15.6 %	12.6 %	9.2 %	11.1 %	9.1 %	8.8 %

combination of chemical changes such as an increase in hydrophobicity due to reduction in the number of hydroxyl groups and physical changes such as changed cell-wall microstructure.

The ASE showed similar tendencies with swelling. At 190°C heat-treatment, the ASE was similar for 12-hour (11.5) and 18-hour (12.9) heat-treatments; at 220°C heat-treatment, it was similar for 18-hour (43.4) and 24-hour (42.6) heat-treatments. Moreover, the ASE was similar for 24-hour heat-treatment at 190°C (31.8) and 12-hour at 220°C (31.0). The effect of heat-treatment increases both when the heat-treatment duration is increased while keeping the temperature constant and when the heat-treatment temperature is increased while keeping the duration constant. In other words, similar properties can be observed in case of heat-treatmentat lower temperatures for a longer duration and in case of heat-treatmentat higher temperatures for a shorter duration. Yang *et al*. (2018) applied the concept of heat-treatment degree to explain this, and Chung *et al.* (2019) conducted a study to quantify the heat-treatment degree in a cumulative function. In this regard, heat-treatment at 190°C for 24 hours and the one at 220°C for 12 hours would have similar heat-treatment degree.

4. CONCLUSION

In this study, the cell-wall microstructure and ASE of heat-treated larch wood were evaluated, and the causes of change in the swelling with heat-treatment were anlayzed through changes in the cell-wall microstructure. As heat-treatment decomposed major element of cell walls, including hemicelluloses, cell walls wereweakened and cut roughly, resulting in failure to maintain their original shapes. In addition, the cells were detached from each other, which widened their intercellular spaces. These phenomena became more severe when the wood was heat-treated at higher temperatures. As the heat-treatment temperature and duration was increased, the amount of absorbed water and swelling decreased. However, the reduction in the amount of absorbed water was not proportional to the decrease in swelling because the decomposition of the major chemical element due to heat-treatment was not proportional to temperature or duration of heat-treatment. Moreover, there were additional effects such as the absorbed water was not bound within the cell walls but penetrated the widened gaps between the cells, failing to contribute to the swelling. Overall, the effects of decreased swelling and improved dimensional stability were considered to be associated with the increase in hydrophobicity and intercellular space due to decomposition of the cell-wall element.

REFERENCES

ASTM D4446. Standard Test Method for Anti-Swelling Effectiveness of Water-Repellent Formulations and Differential Swelling of Untreated Wood When Exposed to Liquid Water Environments.

Boonstra, M.J., Tjeerdsma, B.F., Groeneveld, H.A.C. 1998. Thermal modification of non-durable wood species. 1. The PLATO technology: thermal modification of wood. The International Research Group on Wood Preservation, Document no. IRG/WP 98-40123.

Cho, B.G., Hwang, S.W., Kang, H.Y., Lee, W.H. 2015. Change of dimensional stability of thermally compressed Korean pine (*Pinus koraiensis* Sieb. et Zucc.) wood by heat treatment. Journal of the Korean Wood Science and Technology 43(4): 470-477.

Chung, H., Yang, S.Y., Kim, H., Park, Y., Park, J.S., Yeo, H. 2019. Quantification of wood heat treatment degree and calibration method. In: 2019 Proceedings of The Korean Society of Wood Science and Technology Spring Meeting. The Korean Society of Wood Science and Technology, Cheongju, Korea. pp. 54.

Esteves, B.M., Graça, J., Pereira, H.M. 2008. Extractive composition and summative chemical analysis of thermally treated eucalypt wood. Holzforschung 62(3): 344-351.

Esteves, B.M., Pereira, H.M. 2009. Wood modification by heat treatment: a review. Bioresources 4(1): 370-404.

Kang, C.W., Li, C., Jang, E.S., Jang, S.S., Kang, H.Y. 2018. Changes in sound absorption capability and air permeability of Malas (*Homalium foetidum*) specimens after high temperature heat treatment. Journal of the Korean Wood Science and Technology 46(2): 149-154.

Kang, C.W., Jang, E.S., Jang, S.S., Cho, J.I., Kim, N.H. 2019. Effect of heat treatment on the gas permeability, sound absorption coefficient, and sound transmission loss of *Paulownia tomentosa* wood. Journal of the Korean Wood Science and Technology 47(5): 644-654.

Kim, A.R., Kim, N.H. 2019. Effect of heat treatment and particle size on the crystalline properties of wood cellulose. Journal of the Korean Wood Science and Technology 47(3): 299-310.

Kim, Y.K., Kwon, G.J., Kim, A.R., Lee, H.S., Purusatama, B., Lee, S.H., Kang, C.W., Kim, N.H. 2018. Effects of heat treatment on the characteristics of royal paulownia (*Paulownia tomentosa* (Thunb.) steud.) wood grown in Korea. Journal of the Korean Wood Science and Technology 46(5): 511-526.

Kim, Y.S. 2016. Research trend of the heat-treatment of wood for improvement of dimensional stability and resistance to biological degradation. Journal of the Korean Wood Science and Technology 44(3): 457-476.

Lee, J.M., Lee, W.H. 2018. Dimensional stabilization through heat treatment of thermally compressed wood of Korean pine. Journal of the Korean Wood Science and Technology 46(5): 471-485.

Militz H. 2002. Heat treatment technologies in Europe: scientific background and technological state of art. In: Proceedings of conference on enhancing the durability of lumber and engineered wood products. Forest Products Society, Kissimmee, Orlando, Madison, USA.

Park, Y., Eom, C.D., Han, Y.., Park, J.H., Chang, Y.S., Yang, S.Y., Choi, J.W., Yeo, H. 2014. Combined treatment of green pitch pine wood by heat and superheated steam and the effects on physical properties of the products. Holzforschung 68(3): 327-335.

Park, Y., Eom, C.D., Park, J.H., Chang, Y.S., Kim, K.M., Kang, C., Yeo, H. 2012. Evaluation of physical properties of Korean pine (*Pinus koraiensis* Siebold & Zucc.) lumber heat-treated by superheated steam. Journal of the Korean Wood Science and Technology 40(4): 257-267.

Park, Y., Han, Y., Park, J.H., Chung, H., Kim, H., Yang, S.Y., Chang, Y.S., Yeo, H. 2018. Evaluation of deterioration of Larix kaempferi wood heat-treated by superheated steam through field decay test for 12 months. Journal of the Korean Wood Science and Technology 46(5): 497-510.

Park, Y., Han, Y., Park, J.H., Chang, Y.S., Yang, S.Y., Chung, H., Kim, K., Yeo, H. 2015. Evaluation of physico-mechanical properties and durability of *Larix kaempferi* wood heat-treated by hot air. Journal of the Korean Wood Science and Technology 43(3): 334-343.

Park, Y., Jang, S.K., Park, J.H., Yang, S.Y., Chung, H., Han, Y., Chang, Y.S., Choi, I.G., Yeo, H. 2017. Changes of major chemical components in larch wood through combined treatment of drying and heat treatment using superheated steam. Journal of Wood Science 63(6): 635-643.

Park, Y., Park, J.H., Yang, S.Y., Chung, H., Kim, H., Han, Y., Chang, Y.S., Kim, K., Yeo, H. 2016. Evaluation of physico-mechanical properties and durability of *Larix kaempferi* wood heat-treated by superheated steam. Journal of the Korean Wood Science and Technology 44(5):776-784.

Repellin, V., Guyonnet, R. 2005. Evaluation of heat treated wood swelling by differential scanning calorimetry in relation to chemical composition. Holzforschung 59(1): 28-34.

Welzbacher, C.R., Brischke, C., Rapp, A.O. 2009. Estimating the heat treatment intensity through various properties of thermally modified timber (TMT), International Research Group on Wood Preservation, Document no. IRG/WP 09-40459.

Yang, S.Y., Han, Y., Chang, Y.S., Park, J.H., Park Y., Chung, H., Yeo, H. 2018. Classification of the hot air heat treatment degree of larch wood using a multivariate analysis of near-infrared spectroscopy, Journal of Wood Science 64(3): 220-225.

Yoon, K.J., Eom, C.D., Park, J.H., Kim, H.Y., Choi, I.G., Lee, J.J., Yeo, H. 2009. Color control and durability improvement of yellow poplar (*Liriodendron tulipifera*) by heat treatments. Journal of the Korean Wood Science and Technology 37(6): 487-496.

Yoon, K.J., Eom, C.D., Park, J.H., Lee, Y.W., Choi, I.G., Lee, J.J., Yeo, H. 2008. Control of discolorration of cedar wood (*Cryptomeria japonica*) by heat and UV treatment. Journal of the Korean Wood Science and Technology 36(6): 33-40.

APPENDIX

(Korean Version)

낙엽송 열처리재의 세포벽 미세구조 및 항팽윤효율 평가

초록 : 본 연구에서는 낙엽송 열처리재의 세포벽 미세구조와 항팽윤효율을 평가하고, 이들 간의 상호관계를 분석하고자 하였다. 이를 위하여 낙엽송 판재를 190°C와 220°C의 온도 조건에서 12시간, 18시간, 24시간 동안 열처리하였다. 열처리된 낙엽송재의 횡단면을 1,000배 확대하여 관찰한 결과, 열처리재의 경우 세포가 원래의 형태를 유지하지 못 했으며, 세포의 단면이 거칠게 절단되었고, 세포 간 결합이 끊어지면서 세포간극이 늘어났음을 확인할 수 있었다. 또한, 처리 조건 별 팽윤 실험 결과, 열처리 온도와 시간이 증가함에 따라 수분 흡수량과 팽윤량이 감소하였으며, 항팽윤효율은 증가하는 경향을 보였다. 수분 흡수량의 감소는 열처리에 의한 세포벽의 화학적 변화에 영향을 받은 것으로 보이며, 팽윤량의 감소 및 항팽윤효율의 증가는 화학적 변화 뿐만 아니라 세포벽의 구조적인 변화와 같은 물리적인 변화가 복합적으로 영향을 미쳤기 때문인 것으로 생각된다.

1. 서 론

최근 국내뿐만 아니라 전 세계적으로 기후변화와 탄소배출권 등 환경에 관한 관심이 증가하면서 친환경적이며 지속 가능한 재료인 목재에 관한 관심과 수요가 증가하고 있다. 목재는 다른 재료와는 달리 높은 비강도와 전기, 소리, 열 등에 대한 절연성을 갖고 있으며, 절삭과 접착 등 가공하기 쉽다는 장점이 있다. 반면에, 목재는 고습한 환경에 노출되면 치수가 변하거나 열화가 되는 단점도 있다. 목재는 흡습성 재료로서 자연 상태에서는 늘 수분을 함유하고 있으며, 목재 내 수분은 목재의 성질에 많은 영향을 미친다. 특히, 목재 내 수분은 목재의 치수 변화에 직접적으로 관여되어있다. 즉, 목재 내 수분의 양이 증가하면 목재는 팽윤하고, 반대로 줄어들면 목재는 수축한다. 이러한 목재의 치수 변화는 이용 중인 목재의 구조적 안정성을 저해하는 단점으로 작용할 수 있기 때문에 수분 함량 변화에 따른 목재의 치수 변화를 줄이기 위한 다양한 연구가 이루어져 왔다.

목재 열처리는 목재에 160~260°C 온도 범위의 열을 가하여 목재 세포벽을 구성하는 주요 성분의 화학적 변화를 통해 목재의 물리-역학적 특성과 부후균에 대한 저항성을 개선하는 방법이다(Militz, 2002; Park *et al*., 2014). 목재 열처리는 친환경적인 내구성 향상 방법이라는 장점으로 인해 2000년대 초반부터 유럽을 중심으로 다양한 처리 방법과 그 효과에 관한 연구가 수행되어왔다. 열처리에 의해 목재는 소수성이 증가함에 따라 치수 안정성이 향상되고, 표면 재색이 어두워지며, 균이나 곤충에 의한 저항성이 증가하는 것으로 보고되고 있다(Cho *et al*., 2015; Esteves *et al*., 2008; Kang *et al*., 2018; 2019; Kim, 2016; Kim and Kim, 2019; Kim *et al*., 2018; Lee and Lee, 2018; Park *et al*., 2012; 2014; 2015; 2016; 2018; Repellin and Guyonnet, 2005; Yoon *et al*., 2008; 2009). 이러한 다양한 특성 변화로 인해 열처리 목재는 정원용 가구, 울타리, 기둥, 수납장, 창문틀, 문, 데크재, 외장재, 마루판 등 치수 안정성이나 내후성이 요구되는 다양한 용도에 적용 가능한 것으로 소개되고 있다(Boonstra *et al*., 1998; Esteves and Pereira, 2009; Kim, 2016).

열처리에 의한 목재의 물성 변화는 열처리 온도와 열처리 시간, 그리고 열처리에 사용된 열전달 매개체의 종류에 따라 달라진다(Kim, 2016). 즉, 열처리 온도가 증가할수록 그리고 열처리 시간이 길어질수록 열처리 효과는 증가한다. 하지만 온도나 시간에 따른 열처리 효과는 선형적인 관계를 갖지 않기 때문에 특정 물성을 발현하는 열처리 조건(온도와 시간 등)을 특정하는 것은 매우 어렵다. 이에 따라, 열처리 목재의 이용 목적에 맞는 물성을 맞추기 위한 열처리 조건을 찾는 과정에서 많은 시간과 에너지가 소비되고 있다. 이에 본 연구에서는 국내 침엽수 인공림 중 가장 많은 양을 차지하고 있는 낙엽송에 대하여 열처리 조건별 팽윤량 및 항팽윤효율 측정 결과를 기초 자료로 제시하고자 하였다. 또한, 열처리에 의한 팽윤량 변화의 원인을 세포벽 미세구조를 통해 분석하고자 하였다. 이를 위하여 190°C와 220°C의 온도 조건에서 12시간, 18시간, 24시간 동안 열처리한 낙엽송의 세포벽 미세구조 변화를 주사전자현미경을 통해 관찰하고, 수분 흡수량, 팽윤량 및 항팽윤효율을 측정하였다.

2. 재료 및 방법

2.1. 공시재료

본 연구에서는 산림조합중앙회 중부 목재 유통센터(경기도 여주시)에서 제재 및 건조한 낙엽송(*Larix kaempferi*) 제재목을

구매하여 사용하였다. 건조된 낙엽송 제재목의 치수는 두께 50 mm (방사방향), 폭 150 mm (접선방향), 길이 2,400 mm (섬유방향)이었다.

2.2. 목재 열처리

Fig. 1과 같이 길이 2,400 mm의 낙엽송 제재목을 4등분하여 처리 시간별로 무처리, 12시간, 18시간, 24시간의 4세트로 만들었다. 무처리 세트를 제외한 나머지 3세트를 한꺼번에 반응기에 투입하여 열처리하는 동안, 처리 시간에 맞추어 반응기로부터 하나의 세트씩 꺼내는 방식으로 실험하였다. 열처리 온도는 190°C와 220°C 두 가지 조건으로 설정하였으며, 각 처리 온도별로 12개의 제재목이 사용되었다.

2.3. 세포벽 미세구조 변화 평가

열처리에 의한 목재 세포벽의 미세구조 변화를 평가하였다. 열처리 및 무처리 판재의 가운데 위치에서 옹이나 할렬과 같은 결함이 없도록 한 변의 길이가 5 mm인 정육면체 시험편을 제작하였다. 제작된 시험편을 연화시킨 다음, 전건하여 수분을 모두 제거하였다. 수분이 제거된 시험편은 금으로 코팅한 다음, 주사전자현미경(scanning electron microscope (SEM), SEMCOX EM-30, COXEM, Korea)을 이용하여 세포벽의 미세구조를 관찰하였다.

2.4. 항팽윤효율 평가

열처리된 낙엽송재의 항팽윤효율은 ASTM D4446「Anti-Swelling Effectiveness of Water-Repellent Formulations and Differential Swelling of Untreated Wood When Exposed to Liquid Water Environments」에 따라 측정하였다. 다만, ASTM D4446 규격은 목재 시험편의 도장 처리에 따른 항팽윤효율에 관한 규격이므로 본 연구에서는 도장 처리 대신 열처리를 적용하였다. 열처리 및 무처리 판재의 길이 방향으로 가운데에서 길이 127 mm (접선방향) × 폭 38 mm (방사방향) × 두께 6 mm (섬유방향)인 시험편을 처리 조건별로 10개씩 제작하였다. 제작된 시험편들은 23±2°C, 상대습도 50±5%인 항온항습실에서 항량에 도달할 때까지 조습되었다. 조습된 시험편의 침지 전 접선방향 길이를 swellometer를 이용하여 측정하였고, 증류수에 30분 동안 침지 시킨 후 시험편의 접선방향 길이 변화를 측정하여 팽윤량을 계산하였으며, 무게 변화를 측정하여 수분 흡수량을 계산하였다(Fig. 2). 또한, 열처리가 목재의 치수 변화에 미치는 영향을 정량적으로 평가하기 위하여 무처리재의 팽윤량과 각각의 조건에서 열처리한 낙엽송재의 팽윤량을 이용하여 식 (1)에 따라 항팽윤효율(ASE)을 계산하였다.

$$ASE(\%) = \frac{\beta_n - \beta_h}{\beta_n} \times 100 \tag{1}$$

여기에서, β_n는 무처리재의 접선방향 팽윤량(mm), β_h는 열처리재의 접선방향 팽윤량(mm)을 의미한다.

3. 결과 및 고찰

3.1. 목재 열처리

Fig. 3과 Fig. 4는 각각 190°C와 220°C에서 열처리하는 동안 목재 표면(Surface)과 중심부(Core)의 온도 변화를 나타낸 그래프이다. 두 온도 조건에서 모두 공정이 시작된 지 약 6시간 만에 목재 표면과 중심부 온도가 각각 목표 온도인 190°C와 220°C에 도달하였으며, 이후 공정이 종료되는 시점까지 유지되었다. 전체적으로 목재의 표면과 중심부 사이의 온도 차이가 크지 않았기 때문에 목재 시편 전 부분에 걸쳐서 열처리 효과는 유사할 것으로 생각하였다. 열처리가 종료된 시점에서 모든 판재의 함수율은 0%였다.

3.2. 세포벽 미세구조 변화 평가

Fig. 5는 각각 무처리, 190°C 열처리 및 220°C 열처리한 낙엽송의 횡단면을 1,000배 확대한 SEM 이미지 사진이다. 무처리재의 경우, 세포벽 내의 두꺼운 S2층이 매끈하게 절단된 반면에 두 열처리재는 거칠게 절단되었음을 확인할 수 있다. 또한, 열처리에 의해 세포의 형태가 깨지거나 찌그러지는 등 원 형상과는 다른 변형이 발생했고, 세포와 세포 사이의 결합이 끊어진 듯 세포간극(intercellular space)의 크기가 커졌음을 확인할 수 있다. 이러한 현상은 190°C에서 열처리한 낙엽송에서보다 220°C에서 열처리한 낙엽송에서 더 두드러지게 나타났다. 목재를 열처리하면 세포벽의 주요 성분인 헤미셀룰로오스가 다량 분해되며, 열처리가 진행됨에 따라서 질량감소가 커지는 것으로 알려져 있다(Kim, 2016; Park *et al*., 2017; Welzbacher *et al*., 2009).

이러한 목재 세포벽 주요 성분의 열분해 결과로 세포벽이 약해짐에 따라 세포벽이 거칠게 절단된 것으로 보인다. 또한, 헤미셀룰로오스가 분해되면서 헤미셀룰로오스의 하이드록실 그룹의 수가 감소한다. 이에 따라 세포와 세포 사이의 수소결합이 약해지면서 세포 간 결합이 끊어지고, 결국 세포간극의 크기가 증가한 것으로 생각된다.

3.3. 항팽윤효율 평가

Fig. 6은 열처리 조건에 따른 수분 흡수량과 팽윤량 및 항팽윤효율을 측정한 결과를 나타낸 그래프이고, Table 1은 침지 전/후 시험편의 함수율 변화와 그 차이를 정리한 표이다. 수분흡수량과 팽윤량 모두 열처리 온도가 증가할수록 그리고 열처리 시간이 길어질수록 감소하는 경향을 보였다. 열처리에 의해 목재는 하이드록실 그룹의 수가 감소하여 소수성이 증가한다(Esteves and Pereira, 2009; Kim, 2016; Park *et al*., 2014; 2015; 2016). 즉, 열처리가 진행될수록 소수성이 증가함에 따라 수분 흡수량은 감소하게 되고, 수분 흡수량이 감소함에 따라 팽윤량 또한 감소하는 것으로 생각되었다.

하지만 수분 흡수량은 열처리 시간이 증가함에 따라 일정하게 감소하는 경향을 보였지만, 팽윤량은 일정하게 감소하지 않았다. 수분 흡수량은 열처리에 의한 목재의 화학적 특성 변화에 크게 영향을 받는다. 하지만, 팽윤량은 열처리에 의한 화학적 특성 변화뿐만 아니라 세포벽의 구조 변화와 같은 물리적 특성 변화에도 영향을 받을 수 있다. 목재를 열처리하면 수분이 침투하여 목재가 팽윤되는 셀룰로오스의 비결정영역이 분해된다(Esteves and Pereira, 2009; Kim, 2016). 또한, 앞서 SEM 이미지를 통해 관찰한 바와 같이 열처리에 의해 넓어진 세포 공극 내로 수분이 침투하게 되면, 세포벽이 팽윤되지 않게 된다. 즉, 팽윤율은 열처리에 의한 하이드록실 그룹의 감소에 따른 소수성의 증가와 같은 화학적 특성 변화뿐만 아니라 세포벽의 구조 변화와 같은 물리적인 특성 변화가 복합적으로 영향을 미치는 것으로 생각된다.

항팽윤효율은 팽윤량과 유사한 경향을 보였다. 190°C 조건에서는 12시간 동안 열처리한 것(11.5)과 18시간 동안 열처리한 것(12.9)에서 유사하게 평가되었고, 220°C 조건에서는 18시간 동안 열처리한 것(43.4)과 24시간 동안 열처리한 것(42.6)에서 유사하게 평가되었다. 특이한 점은 190°C 조건에서 24시간 동안 열처리한 것(31.8)과 220°C 조건에서 12시간 동안 열처리한 것(31.0)에서 항팽윤효율이 유사하게 평가되었다는 것이다. 열처리에 의한 목재의 물성 변화는 같은 온도에서 열처리할 때는 시간이 길어질수록, 같은 시간 동안 열처리할 때는 온도가 증가할수록 변화폭이 커지게 된다. 다시 말해서 저온에서 장시간 동안 열처리하는 경우와 고온에서 단시간 동안 열처리하는 경우에 유사한 물성이 발현될 수 있다. Yang 등(2018)은 이를 설명하기 위해 열처리도라는 개념을 적용하였으며, Chung 등(2019)은 열처리도를 온도와 시간의 누적 함수로 정량화하는 연구를 수행한 바 있다. 이 개념으로 보았을 때, 190°C 조건에서 24시간 동안 열처리한 경우와 220°C 조건에서 12시간 열처리한 경우는 항팽윤효율 측정에 있어 유사한 열처리도 값을 가질 것이라 생각된다.

4. 결 론

본 연구에서는 낙엽송 열처리재의 세포벽 미세구조와 항팽윤효율을 평가하고, 열처리에 의한 팽윤량 변화의 원인을 세포벽 구조 변화로 분석하였다. 열처리에 의해 목재의 세포벽을 구성하는 주요 성분이 열분해됨에 따라 세포벽이 약해지면서 거칠게 절단되고, 세포의 원래 형태를 유지하지 못 했으며, 세포와 세포 사이의 결합이 끊어짐에 따라 세포간극이 넓어지는 경향을 보였다. 이러한 현상은 높은 온도에서 열처리했을 때 더 심하게 나타났다. 열처리 온도와 시간이 증가함에 따라 수분 흡수량과 팽윤량은 감소하는 경향을 보였다. 하지만, 수분 흡수량 감소와 팽윤량 감소는 비례하지 않는 것으로 나타났다. 이는 열처리에 따른 화학적 주성분의 분해가 열처리 온도 또는 시간과 비례하지 않기 때문인 것으로 생각된다. 또한, 흡수된 수분이 세포벽과 결합하지 않고, 열처리에 의해 늘어난 세포 내 공극으로 침투하면서 팽윤에 관여하지 못하는 부수적인 효과도 있을 것으로 생각된다. 즉, 열처리에 의한 팽윤량의 감소 및 치수 안정성의 개선 효과는 세포벽 구성성분의 분해에 따른 소수성의 증가와 세포 공극의 증가에 따른 영향인 것으로 생각된다.