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ORIGINAL ARTICLE

Evaluating the effect of heat treating temperature and duration on selected wood properties using comprehensive cluster analysis

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Abstract In this study, twenty-five heat treatments were conducted at the various treating conditions of five temperatures and five durations. At each treatment, 15 poplar specimens were used. Twelve variables that represented wood color, physical and mechanical properties and durability were measured for both treated and untreated specimens. To evaluate the effect of heat treatment on the wood performance, a comprehensive cluster analysis was applied to the measured variables of treated and untreated specimens. Cluster analysis showed that treating and control specimens can be distinctly classified into 2, 3 or 4 clusters according to the intended purposes. Two clusters can represent the control group and twenty-five testing groups. Four clusters represented the control group and three groups of testing specimens having mild, moderate and severe treating conditions, respectively. At the mild treatment, modulus of rupture (MOR) was reduced <11 %. Wood durability increased to moderate resistant. At the moderate treatment, EMC decreased by 28 %, and MOR was decreased by more than 12 %. In the severe treatment, wood durability increased to resistant or highly resistant; however, its MOR was reduced half.

Keywords Cluster analysis · Factor analysis · *Populous tomentosa* · Thermal treatment · Wood properties

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Introduction

Heat treatment improves wood durability [1, 2] and its dimensional stability [3–5]. Heat-treated wood is darker and more uniform in color, which is preferred in many European countries [6–8]. However, heat treatment can also cause some unfavorable changes. Mass loss, reduced strength and increased brittleness, especially in high temperature, limit its application in structural area [4, 6, 9]. The volume of heat-treated lumber is increasing and the commercialization of heat-treated wood is also growing. It is essential to know the correlations among wood properties so that treating conditions can be effectively selected and controlled.

Heat treating condition includes treating temperature and duration, which affect the properties of heat-treated wood. MOR and hardness of Chinese fir decreased slightly when it was heat treated at 170 and 185 °C. At the temperature above 200 °C, MOR and hardness decreased rapidly [10]. Main compositions of wood, hemicellulose, cellulose and lignin, undergo different complex chemical transformations as temperature increases [11]. Chemical reaction rates of wood constituents vary with treating temperature. Therefore, heat treatment can be divided into different levels.

Classification and quality control of heat treatment have been studied over the last few decades [12–14]. Finnish Wood Preserving Association classified heat-treated wood into three heat treatment classes based on their durability according to EN 335-1 [12]. In this study, we applied cluster analysis to classify heat-treated wood with various properties into consideration in addition to the durability.

Cluster analysis is a statistical technique to group a set of objects according to their similarity. Similarity can be computed based on several specific variables. The cluster analysis has been widely applied to wood researches [13, 15]. For example, it was used to process the data from infrared spectroscopy or near infrared spectroscopy for species identification [16].

Many researchers had successfully separated the treatment intensities of unknown samples based on some non-destructive methods, such as color measurement and NIR spectroscopy. Schnabel et al. [17] measured the color of beech wood from 3 different heat treatments and control group. They classified those samples into 4 groups using cluster analysis based on wood color. However, classification of heat-treated wood only with color has certain limit, because other properties, such as wood physical and mechanical properties, are crucial for the end use. Bachle et al. [14] successfully classified some samples according to their treatment intensities using SIMCA classification method based on NIR spectroscopy. These researches defined classes by treatment intensities in advance, and then sorted some unknown samples into corresponding classes to prove their methods are feasible. This study aims to divide treatment intensities into different levels according to their comprehensive properties.

In this study, the relationships among heat-treated wood properties were analyzed. Comprehensive cluster analysis on effect of the heat treating temperatures and duration on wood properties was conducted to establish a novel approach of grading heat treatments according to the selected properties of heat-treated wood.

Experimental

Materials and heat treating process

Fifteen 20-year old white poplar (*Populous tomentosa*) trees were harvested from a planted forest in Shandong province, China. Clear samples were cut into size of 25 mm × 50 mm × 500 mm (radial × tangential × longitudinal). Twenty-five heat treatments were carried out at five treating temperatures and five treating durations. The five temperatures were 170, 185, 200, 215 and 230 °C and five treating duration were 1, 2, 3, 4 and 5 h. A control group of fifteen untreated specimens were used and not treated. To get reliable results, fifteen replicates from fifteen different trees were used for each treatment condition. Twenty-six clear specimens from each tree were randomly selected for twenty-five treatments and control group. The specimens were dried to 8 % moisture contents (MC) before they were treated. Steam heat treatments were conducted in an experimental kiln equipped with an electrical heating system and a zirconia oxygen analyzer. Steam was used to replace the oxygen. By regulating the amount of steam, the oxygen content in the kiln was controlled under 2 % during the treatment.

Table 1 Classes of decay resistance used in this study

Class	1	2	3	4
Weight loss	0–10 %	11–24 %	25–44 %	>45 %
DR	HR	R	MR	NR

DR decay resistance, *HR* highly resistant, *R* resistant, *MR* moderately resistant, *NR* nonresistant

Measurement of specimen properties

The objective of heat treatment is to minimize wood dimensional change, increase wood durability and modify wood color during thermal treatment. Twelve wood property variables were measured to evaluate the effect of heat treatment on wood properties. Eight indicators are related to wood physical and mechanical properties, such as oven-dry density (Den), volumetric shrinkage from water-swollen to air-dry condition (SH_{-air}), volumetric shrinkage from water-swollen to oven-dry condition (SH_{-ov}), volumetric swelling from oven-dry to water-swollen condition (SW_{-ov}), volumetric swelling from air-dry to water-swollen condition (SW_{-air}), equilibrium moisture content (EMC) at 20 °C and 65 % relative humidity, modulus of rupture (MOR), modulus of elasticity (MOE). The measurements were made according to ISO standards [18, 19].

Wood color was determined using a colorimeter (CR-300, Konica Minolta) according to CIE LAB color space established by the Commission International de Enluminure [17]. Prior to cutting other specimens, 15 samples of each treatment were scanned on tangential section after heat treatment. For control group, wood color is also recorded. The scanning area is a circle with diameter of 8 mm. Three color parameters were collected including lightness (L^*), chromatic coordinates on the green–red axis (a^*), chromatic coordinates on the blue-yellow axis (b^*). For each sample, the mean values of 3 points evenly distributed on the tangential section were recorded.

Wood durability was measured after heat treatment. A brown-rot fungi species (*Gloeophyllum trabeum*) was used to evaluate the decay resistance (DR). The test samples with size of 20 mm × 20 mm × 10 mm (radial × tangential × longitudinal) were sawn from the specimens of treatment and control groups. After 12-week exposure to

Table 2 Measured properties of heat-treated and control poplar woods by treating temperature and duration

Treating Temp.	Treating duration (h)	SH_{-air} (%)	SH_{-ov} (%)	SW_{-air} (%)	SW_{-ov} (%)	EMC (%)	MOR (Mpa)	MOE (Gpa)	Den (g/m^3)	Color L^* (%)	Color a^* (%)	Color b^* (%)	DR
Control		6.47	10.98	2.63	11.52	10.23	73.32	10.20	0.446	89.53	0.92	19.14	NR
170 °C	1	6.41	10.87	2.53	11.21	8.74	78.17	11.71	0.443	77.11	5.36	23.86	NR
	2	6.36	10.82	2.49	10.84	8.85	78.83	11.81	0.442	72.89	6.41	24.47	MR
	3	6.27	10.75	2.37	10.69	8.64	81.59	11.67	0.437	69.86	7.27	24.72	MR
	4	6.01	10.34	1.93	10.53	8.45	76.99	11.35	0.434	68.51	7.34	25.52	MR
	5	5.82	10.21	1.82	10.29	8.27	76.48	11.30	0.430	65.91	7.73	25.98	MR
185 °C	1	6.36	10.77	1.99	10.66	8.43	80.30	11.43	0.441	72.36	6.14	25.17	MR
	2	5.95	10.39	1.86	9.60	8.13	73.89	11.23	0.432	68.93	6.54	25.20	MR
	3	5.71	9.90	1.65	9.53	7.74	73.14	11.08	0.429	63.25	7.69	25.56	MR
	4	5.45	9.19	1.52	9.20	7.57	66.48	10.95	0.424	62.51	7.86	25.81	MR
	5	5.40	8.96	1.48	9.11	7.41	66.17	10.74	0.421	61.77	8.03	26.06	MR
200 °C	1	6.16	10.08	1.79	9.11	7.34	64.44	11.49	0.433	57.68	8.72	24.42	MR
	2	5.14	8.56	1.77	8.16	6.31	59.63	11.43	0.427	48.70	9.66	22.99	MR
	3	4.50	7.84	1.51	7.75	5.82	51.71	11.38	0.424	43.68	9.64	21.42	MR
	4	4.25	7.54	1.44	7.39	5.82	48.71	11.27	0.416	42.05	9.83	21.25	MR
	5	4.17	7.32	1.39	6.70	5.44	47.51	11.04	0.411	40.49	9.74	20.45	R
215 °C	1	4.50	8.09	1.48	7.76	5.56	54.88	11.48	0.424	43.62	9.48	21.31	MR
	2	3.25	6.63	1.38	6.84	5.60	49.10	11.35	0.417	40.66	9.15	19.89	R
	3	3.18	6.37	1.27	6.12	5.39	47.18	11.21	0.413	45.83	8.66	21.65	R
	4	3.02	6.10	1.21	5.97	5.10	41.63	10.95	0.409	31.27	5.38	16.10	R
	5	2.53	5.47	1.14	5.81	5.02	36.97	10.69	0.396	30.58	7.60	14.01	R
230 °C	1	3.29	6.14	1.39	6.59	5.03	43.32	11.22	0.412	33.27	8.06	15.87	R
	2	2.66	5.33	1.33	5.70	4.76	42.25	11.10	0.411	39.32	7.80	18.07	HR
	3	2.43	5.20	1.25	5.53	4.89	37.69	10.81	0.397	29.17	6.88	12.75	HR
	4	2.34	5.21	1.21	5.22	4.67	36.01	10.70	0.392	29.39	6.84	12.81	HR
	5	2.12	4.89	1.09	5.02	4.77	33.58	10.48	0.385	28.30	6.82	12.45	HR

DR, HR, R, MR, NR are defined in Table 1

SH_{-air} volumetric shrinkage from water-swollen to air-dry condition, SH_{-ov} volumetric shrinkage from water-swollen to oven-dry condition, SW_{-air} volumetric swelling from air-dry to water-swollen condition, SW_{-ov} volumetric swelling from oven-dry to water-swollen condition, *EMC* equilibrium moisture content, *MOR* modulus of rupture, *MOE* modulus of elasticity, *Den* oven-dry density, L^* lightness, a^* chromatic coordinates on the green–red axis, b^* chromatic coordinates on the blue-yellow axis

fungi, test samples were weighed to calculate the percent weight losses (WL). Weight loss of each treatment was the average value of 15 samples. Four DR classes (Table 1) were used as described in ASTM D2017 [20]. Twenty-six test results were classified accordingly.

Data analysis

Using IBM SPSS Statistics 19.0, twenty-six observations with twelve factors were analyzed for data dimensionality reduction. Average values of 15 replicates for each observation were reserved for further analysis. In factor analysis, principal component method was used for data extraction, varimax method for rotation and regression method for computing factor scores. Factor scores for 26 observations were then utilized in the cluster analysis. Hierarchical cluster analysis was performed using between-group linkage method, and data were standardized by *Z* scores method to compute the squared Euclid distances.

Results and discussion

Property changes of heat-treated wood

Table 2 shows the average values of wood properties of 390 specimens from twenty-five treatments and control group. The results indicated that volumetric shrinkage, volumetric swelling, equilibrium moisture content, density and lightness decreased and decay resistance increased

Table 3 Correlation coefficients and significance levels among measured twelve variables corresponding to the wood properties of poplar specimens

	SH_{-air}	SH_{-ov}	SW_{-air}	SW_{-ov}	EMC	MOR	MOE	Den	L^*	a^*	b^*	DR
SH_{-air}	1.00	0.99***	0.86***	0.98***	0.94***	0.97***	0.45*	0.95***	0.94***	−0.25	0.87***	0.90***
SH_{-ov}		1.00	0.88***	0.98***	0.96***	0.98***	0.45*	0.95***	0.95***	−0.29	0.85***	0.89***
SW_{-air}			1.00	0.91***	0.91***	0.86***	0.40*	0.89***	0.90***	−0.52**	0.58**	0.78***
SW_{-ov}				1.00	0.98***	0.98***	0.40*	0.94***	0.97***	−0.38	0.81***	0.89***
EMC					1.00	0.95***	0.25	0.89***	0.98***	−0.52**	0.73***	0.83***
MOR						1.00	0.47*	0.93***	0.95***	−0.29	0.87***	0.83***
MOE							1.00	0.54**	0.28	0.41*	0.58**	0.38
Den								1.00	0.92***	−0.27	0.83***	0.89***
L^*									1.00	−0.48*	0.78***	0.84***
a^*										1.00	0.13	−0.22
b^*											1.00	0.77***
DR												1.00

Variables are defined in Table 2

* Correlation is significant at the 0.05 level, ** correlation is significant at the 0.01 level, *** correlation is significant at the 0.001 level

consistently as treatment temperature and duration increased. Chromatic coordinates (a^*, b^*) increased to the maximum and then gradually declined during the treatment. These results were consistent with many studies reported in a review [11].

MOE showed an increase compared with untreated specimens. MOR rose initially and declined afterwards. Shi et al. [21] studied the mechanical behavior of aspen (*Populus* spp.) after heat treatment using ThermoWood process. Their research showed that after a treatment at 200 °C for 3 h, MOR decreased by 35 % and MOE increased by 15 %. For a same temperature and duration, MOR decreased by 29 % and MOE increased by 12 % in this present study. Heat-treated specimens had a higher strength than control specimens (untreated wood) when the specimens were treated at the temperature at or below 185 °C for no more than 2 h. Many researchers also found the increase in MOR at moderate treatments [22, 23]. The reasons for the initial improvement in mechanical properties are attributed to the increased cellulose crystallinity. Another important reason is that lower equilibrium moisture content of heat-treated wood when placed in service conditions, since mechanical properties increase with decreasing moisture content [24].

Twelve variables changed differently during heat treatment. Plus, treatment temperature and duration have an interaction on the wood properties. Treatment at 215 °C for 1 h or treatment at 200 °C for 3 h has the same effect on the most wood properties, as presented in Table 2. The effect of the treating conditions on the wood properties is complicated and can be classified using cluster analysis.

Correlations between variables

Pearson correlation coefficients between above twelve variables are presented in Table 3. It proved out clearly that nine variables (EMC, L^*, SW_{-air}, SH_{-ov}, SW_{-ov}, MOR, DR, SH_{-air} and Den) are highly correlated with one another at a significant level of 0.001 (correlation coefficient > 0.77). Similar results were reported by Robert Welzbacher et al. [25] that durability of beech wood correlated well with L^*, EMC, dimensional stability, etc. The other three variables, MOE, a^*, b^*, are poorly correlated. This suggested that with a known property among the nine variables, other properties can be calculated. L^* can be easily obtained; therefore, it has the potential to be used to predict other properties. The feasibility of predicting wood properties using lightness has been discussed by González-Peña and Hale [26].

Factor analysis

Factor analysis was used for evaluating the relations among the observed variables. Factor analysis of the data from Table 2 can interpret 92.17 % of the total variance. After rotation, Factor 1 and Factor 2 account for 77.64 and 14.53 % of the total variance, respectively (Table 4). Table 5 shows the factor loadings for twelve variables. Each variable can be expressed by a formula of the extracted factors and factor loadings. For instance, SH_{-air} can be calculated using the formula, 0.976Factor 1 + 0.142Factor 2.

Figure 1 shows the distribution of variables on the factor loading plot. In the figure, EMC, L^*, SW_{-air}, SH_{-ov}, SW_{-ov},

Table 4 Variance explained after extracted two factors and rotated using varimax method

Factor	Initial variance explained			Extraction sums of squared loadings			Rotated sums of squared loadings		
	Total	% of Variance	Cumulative (%)	Total	% of Variance	Cumulative (%)	Total	% of Variance	Cumulative (%)
1	9.368	78.07	78.07	9.368	78.07	78.07	9.317	77.64	77.64
2	1.693	14.11	92.17	1.693	14.11	92.17	1.744	14.53	92.17
3	0.486	4.05	96.22						
4	0.236	1.97	98.19						
5	0.088	0.73	98.92						
6	0.059	0.49	99.41						
7	0.035	0.29	99.70						
8	0.017	0.14	99.85						
9	0.008	0.07	99.92						
10	0.005	0.04	99.96						
11	0.004	0.03	99.99						
12	0.001	0.01	100.00						

Table 5 Loadings on factor 1 and factor 2 for twelve measured variables

	Factor 1	Factor 2
SH_{-air}	0.976	0.142
SH_{-ov}	0.986	0.106
SW_{-air}	0.923	−0.122
SW_{-ov}	0.994	0.018
EMC	0.978	−0.152
MOR	0.974	0.118
MOE	0.397	0.782
Den	0.958	0.161
L^*	0.981	−0.105
a^*	−0.406	0.873
b^*	0.806	0.483
DR	0.891	0.126

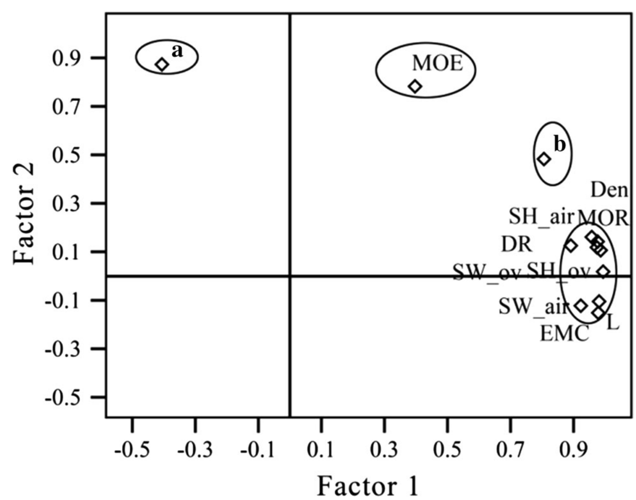

Fig. 1 Factor loading plot for twelve measured variables in rotated space. *L* lightness, *a* chromatic coordinates on the *green–red* axis; *b* chromatic coordinates on the *blue-yellow* axis

MOR, DR, SH_{-air} and Den distributed heavily and had strong positive loadings on Factor 1. This indicated that these variables were highly linear correlated with one another and Factor 1 had major effect on these properties. The other three variables, MOE, a^* and b^*, were comparatively scattered and had strong loadings on Factor 2. Their variation was different from other nine variables.

Cluster analysis with twelve measured variables

Cluster analysis of twenty-five treatments and the control samples was done based on computed factor scores. Figure 2 exhibited the dendrogram of those clusters. Treatments are represented as "Temperature-duration". For instance, "170-1" means treatment at 170 °C for 1 h; "0-0" symbolized the control group. According to cluster distances, treated specimens were divided into 2, 3 or 4 clusters.

Specimens could be divided into 2 clusters: one cluster consisted of the control specimens and another cluster consisted of specimens in twenty-five treatments. This classification differentiates untreated wood from heat-treated wood. The result showed that the heat treatment has significant effect on wood properties.

If the specimens were divided into 3 clusters, one cluster consisted of the specimens in the control group, another cluster the specimens from five treatments of higher temperature and longer duration (at 215 °C for 4–5 h or 230 °C for 3–5 h) and last cluster of specimens from the remaining twenty heat treatments of lower intensities. The result suggested that the treatments of higher temperature above 215 °C and duration influenced wood properties dramatically. The treatments significantly increased

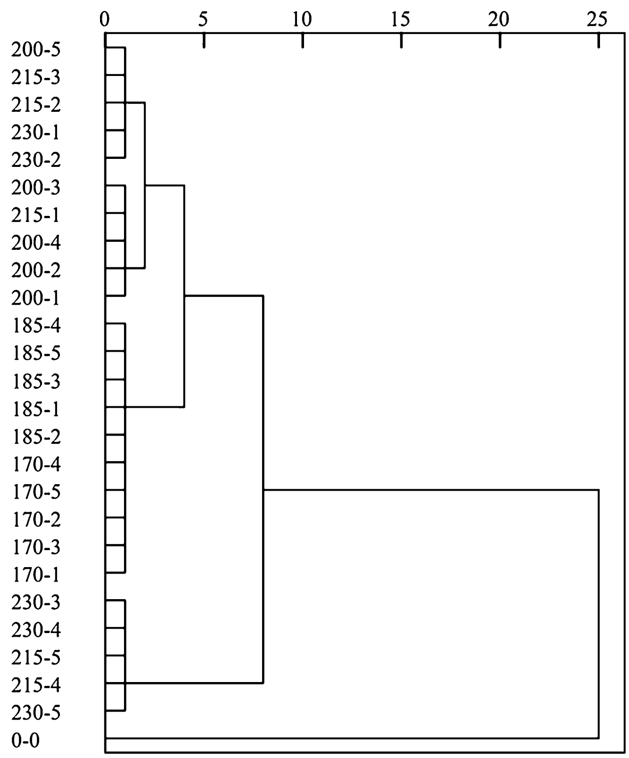

Fig. 2 Cluster dendrogram based on twelve measured variables. The *horizontal axis* shows the distances of clusters; the *vertical axis* represents twenty-five treatments and the control group

dimensional stability, darkened wood, increased decay resistance, but decreased bending strength (about 50 % in this study).

If the specimens were divided into 4 clusters, Cluster 1 consisted of the specimens from control group. Cluster 2 had the specimens from ten treatments at the lower temperature of 170 and 185 °C. Cluster 3 had specimens from ten mild treatments, with five treatments at 200 °C, and the other five treatments at the temperature and duration of 215 °C, 1–3 h and 230 °C, 1–2 h. Cluster 4 had specimens from the remaining five treatments with higher temperature and longer duration of 215 °C, 4–5 h and 230 °C, 3–5 h. Clustering results of these four clusters are presented in Table 6.

Wood properties of the specimens in each cluster are listed in Table 7. MOE of the specimens from three tested clusters showed an increase <17 %. For the specimens from cluster 2, EMC declined by 13–28 %, and the durability increased from nonresistant to moderate (except for the treatment at 170 °C for 1 h). In the cluster 2 MOR changed within ±11 %. In cluster 3, EMC decreased by more than 28 %, and the durability ranged from moderate to highly resistant. In cluster 3, MOR decreased by more than 12 %. In cluster 4, EMC decreased by more than 50 % and the durability changed to resistant or highly resistant. However, MOR reduced by 54 %. Lumber with severe treatments, as in cluster 4, can be applied in outdoor environment and not suggested for in the structural uses.

Specimens from treatments were grouped into other clusters when temperature surpassed 200 °C. The results indicated that wood properties changed significantly when the treating temperature exceeded 200 °C. This critical temperature was also identified in the previous research [10]. Wood properties significantly change at the temperature above 200 °C [27].

The results of cluster analysis provided evidence for classification of heat-treated wood with similar properties. According to the classification, treating temperature and duration can be selected for different end uses.

Cluster analysis with the selected variables

Previous cluster analysis with twelve wood properties produced a good result. However, large amount of data were collected. It is possible that fewer representative wood properties can be selected and the information loss can be minimized to obtain a reliable cluster analysis result. The result of factor analysis can be used for the selection of variables. In view of variable distribution in Fig. 1, twelve wood properties can be classified into four

Table 6 Clustering results of 4 clusters

	0 h	1 h	2 h	3 h	4 h	5 h
170 °C						
185 °C						
200 °C	Control					
215 °C						
230 °C						

Table 7 Properties' changes of heat-treated *Populous tomentosa* wood in the divided clusters

	EMC (%)	MOR (%)	MOE (%)	ΔE^*	DR
Cluster 1	0	0	0	0	NR
Cluster 2	−(13–28)	(+11 to −10)	+(5–16)	14–38	NR–MR
Cluster 3	−(28–53)	−(12–42)	+(8–13)	34–56	MR–HR
Cluster 4	−(50–54)	−(43–54)	+(3–7)	59–62	R–HR

"%" is the change rate compared to control group, "+" means increase, "−" means decrease, ΔE^* is total color difference, EMC, MOR, MOE and DR are the same in Table 2

categories. One category is nine densely distributed variables, and other variables belonged to other three categories separately. To reduce information loss, selected variables should contain variables of the four categories, that is, a^*, b^*, MOE and at least one of the above nine densely distributed variables.

Variable cluster analysis can be another useful tool by classifying variables according to their correlations. Using IBM SPSS Statistics 19.0, variable cluster analysis was conducted with data in Table 2. The result showed that nine highly correlated variables (SH_{-air}, SH_{-ov}, SW_{-ov}, MOR, Dens, DR, EMC, L^* and SW_{-air}) can be grouped together, the other 3 variables belonged to different groups, which is consistent with results from factor analysis. MOR is an indication of the strength and EMC is associated with the moisture exchange within the environment. EMC is also associated with the swelling and shrinkage variables (Table 3). From the factor analysis and variable cluster analysis, five representative variables were selected as follows: MOR, EMC, a^*, b^*, MOE. Factor analysis and cluster analysis of specimens from 25 treatments and control group were conducted with the five variables and results are shown in Fig. 3.

Figure 3 shows that clustering results using selected variables were same as those from previous analysis when divided into 2, 3 and 4 clusters. This result suggested that for the evaluation and classification of heat-treated wood, only using wood properties of MOR, EMC, a^*, b^* and MOE can show the changes in all other wood properties after heat treatment. Cluster analysis is a useful tool for classification and evaluation of heat-treated wood.

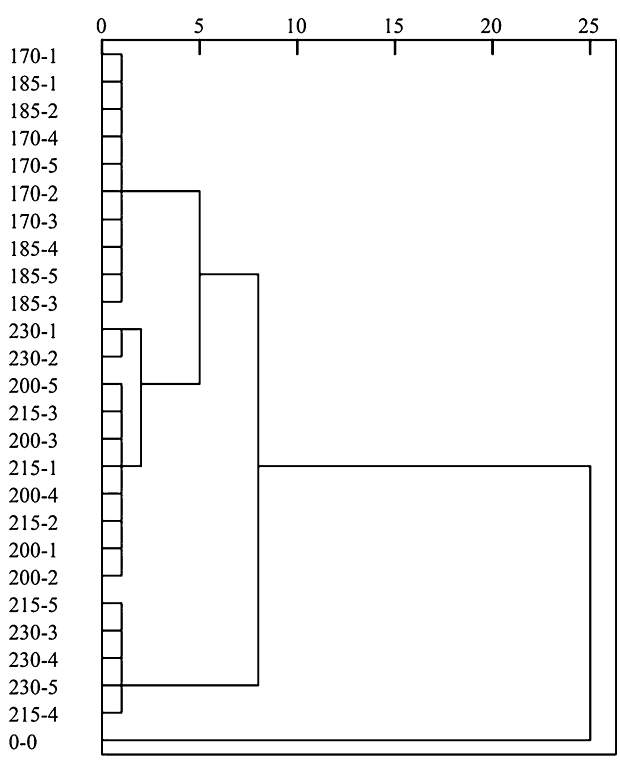

Fig. 3 Cluster dendrogram using five selected variables

Conclusions

The white poplar wood specimens were heat treated at five treating temperatures and five treating durations. Twelve wood property indicators representing color, durability, physical and mechanical properties were measured. Cluster analysis was used for the classification of heat-treated wood. The conclusions of the study are as follows.

1. The statistical analysis showed that variables, such as volumetric swelling, volumetric shrinkage, equilibrium moisture content, decay resistance, lightness, modulus of rupture and oven-dry density, can be grouped with high correlation with one another after heat treatment. Other variables, such as a^*, b^*, MOE, have relatively poor correlation and were enlisted as the separated group.
2. Cluster analysis can classify 26 treated and untreated groups into 2, 3 or 4 clusters. In 2 clusters, it can distinguish control specimens from heat-treated specimens. In 3 clusters, heat treatments are further divided into two intensities. In 4 clusters, four groups can represent the non-treated samples, samples with mild, moderate and severe treating conditions. At the mild

treatment, MOR was reduced <11 %. Wood durability increased to moderate resistant. At the moderate treatment, EMC decreased by 28 %, and MOR was decreased by more than 12 %. In the severe treatment, wood durability increased to resistant or highly resistant; however, its MOR was reduced half.

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