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Resin Impregnation of Sawdust Board for Making Woodceramics (I)[*1]

- Effect of Impregnation Method and Time on Physical and Mechanical Properties -

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ABSTRACT

This research work explored physical and mechanical properties of impregnated sawdust boards from three softwood species (*P. densifora, L. kaemferi,* and *P. koraiensis*) with phenol-formaldehyde (PF) resin by various vacuum treatment methods of combining pressure, vacuum, and ultrasonic waves. Simultaneous vacuum and ultrasonic wave treatments with no pressure resulted in the greatest increase in resin content, density, dimensional changes (thickness and length), bending strength, and hardness of impregnated board. This result seemed to be attributed to the ultrasonic wave treatment.

Keywords : sawdust board, impregnation, phenol- formaldehyde resin, physical and mechanical properties

1. INTRODUCTION

Woodceramics, which are made by impregnating thermosetting resin into wood or woody materials and burning in high temperature, are porous carbon materials and are evaluated as new materials for various industrial applications (Hokkirigawa *et al.*, 1995, 1996a, 1996b; Kano *et al.*, 1996; Kasai *et al.*, 1996). In addition, woodceramics can be manufactured from low-quality logs, thinned logs, and log residues. Thus, it can be recognized as environment-friendly material. The properties of woodceramics are greatly affected by density, resin content, burning temperature, *etc.* of the impregnated raw materials. Especially, uneven impregnation can be the cause of fissures and wrappings during the carbonizing process. So, impregnation methods and techniques need further researches. In general, resin impregnation has been mainly used for the preservation of ground beams and the like, but recently for developing high value-added products by improvement of water-resistance and moisture-proof, and toughness such as WPC (Wood Polymer Composite), *etc.* Vacuum and pressurization processes are usually used in impregnating, but some difficulties, such as uneven resin pene-

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Table 1. Characteristics of phenol formaldehyde resins

Power resin (Novolak type)		Liquid resin (Resol type)	
Solids content (%)	99	Solids content (%)	55-53
Melting point (℃)	80-95	Specific gravity	1.06
Gelation time (sec)	80-120	Gelation time (sec)	80-95
Plate flow (mm)	30-35	Viscosity (cps)	45-65

tration, long running time, a large amount of facility investment, must be solved for developing a new method of resin impregnation (Okabe & Saito, 1995a, 1995b; Okabe *et al.*, 1996a, 1996b). The purpose of this study, therefore, is to develop impregnation techniques with a wide variety of processes: no pressure, vacuum, no pressure just after vacuum, no pressure after vacuum with simultaneous ultrasonic waves.

2. MATERIALS and METHODS

2.1. Materials

This experiment used thinned logs of *P. densiflora, L. kaemferi,* and *P. koraiensis* as raw materials. Sawdust by species from a circular sawing was sorted in grain size of 1 mm or less, and was conditioned to moisture content of 6% or less. Powder and liquid phenol-formaldehyde resins (Kolon Chemical Co., Ltd.) were used as binder and impregnation agent. Table 1 shows the characteristics of resins.

2.2. Boards Preparation

Each board sized 26×26×1.4 cm was made with powder phenol-formaldehyde resin. Sawdust were mixed well in a zinc box, put into a square stainless die, and molded in thermal press. The conditions for making sawdust boards were as follows: resin content 10%; press temperature 190°C; pressure 40→20→10 kgf/cm^2; press time 6→5→4 min. Through thermal compression under these conditions, 10 boards of 0.6 g/cm^3 in target density were made.

2.3. Resin Impregnation

Ten boards of 0.6±0.01 g/cm^3 in density by the wood species were cut into 12×12×1.4 cm in size, and humidified for 3 weeks under the temperature and humidity of 20±1°C and 65±5% for constant test conditions. To examine the physical and mechanical properties of impregnated boards by the method and time of impregnation, the experiment was designed with combining four methods of impregnation: no pressure for 20 min, vacuum for 20 min, no pressure for 10 min after vacuum for 10 min, and no-pressure for 10 min after vacuum with ultrasonic waves for 10 min. Table 2 shows the methods and conditions of impregnation stated above. The impregnation apparatus for vacuum with ultrasonic waves used in this study consists of an impregnation tank, an ultrasonic vibration part, an oscillation part, a vacuum pump, *etc.* Test samples prepared were put into the impregnation tank containing liquid phenol-formaldehyde resin, impregnated in atmosphere air pressure under each condition, dried, and then hardened for 8 h at 60°C, 10 h at 100°C and 8 h at 135°C, followed by humidification in the temperature and humidity of 20±1°C and 65±5%.

2.4. Physical and Mechanical Properties

To investigate the physical and mechanical

Table 2. Methods of impregnation and total impregnation time

(unit: min.)

Impregnation method \ Number of cycle	1	2	3	4
1. No pressure	20	40	60	80
2. Vacuum	20	40	60	80
3. Vacuum /	10	20	30	40
no pressure	10	20	30	40
4. Vacuum with	10	20	30	40
ultrasonic waves				
/ no pressure	10	20	30	40

Table 3. Change of density after impregnation

(unit: g/cm^3)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Number of cycle \ Impregnation method	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	0.75[a] ±0.02[b]	0.75 ±0.03	0.76 ±0.03	0.85 ±0.04	0.75 ±0.03	0.77 ±0.03	0.74 ±0.04	0.81 ±0.04	0.74 ±0.03	0.72 ±0.03	0.75 ±0.04	0.83 ±0.04
2	0.75 ±0.03	0.76 ±0.03	0.78 ±0.04	0.83 ±0.05	0.76 ±0.04	0.77 ±0.03	0.75 ±0.02	0.82 ±0.04	0.71 ±0.02	0.70 ±0.03	0.75 ±0.03	0.83 ±0.04
3	0.76 ±0.03	0.77 ±0.03	0.80 ±0.04	0.86 ±0.05	0.75 ±0.03	0.78 ±0.03	0.75 ±0.02	0.83 ±0.04	0.75 ±0.02	0.72 ±0.02	0.76 ±0.03	0.84 ±0.04
4	0.78 ±0.04	0.79 ±0.03	0.81 ±0.04	0.87 ±0.05	0.77 ±0.02	0.79 ±0.03	0.77 ±0.02	0.86 ±0.04	0.76 ±0.03	0.73 ±0.03	0.76 ±0.03	0.86 ±0.04

a : Mean value of 5 replications
b : Standard deviation of 5 replications
1* : No pressure only 2* : Vacuum only 3* : No pressure after vacuum
4* : No pressure after vacuum with ultrasonic vibration

properties of impregnated boards by the method and time, their weights, lengths, and thicknesses were measured before and after impregnation. Also, bending strength was measured by a universal testing machine (Autograph, AGS 10KN, Shimadzu) at a cross head speed of 5 mm/min, and hardness was tested under the condition of 10 mm in steel diameter and 500 kg in load by Brinell hardness meter.

3. RESULTS and DISCUSSION

3.1. Variations of Density and Resin Content

Variation of density by the impregnation method and time for sawdust boards by wood species is shown in Table 3. The results from board having density of 0.6±0.01 g/cm^3 showed gradual increase in density with the increase of

Table 4. Resin content of board after impregnation

(unit: %)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Impregnation method / Number of cycle	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	33.1[a] ±4.3[b]	32.3 ±4.7	45.0 ±5.6	52.7 ±5.9	39.6 ±4.7	37.0 ±3.6	47.0 ±5.2	51.3 ±7.3	32.4 ±4.7	29.7 ±3.6	42.3 ±4.6	57.9 ±6.9
2	46.8 ±5.3	38.5 ±4.6	50.5 ±6.4	64.5 ±6.3	43.1 ±5.2	42.2 ±3.9	51.6 ±6.5	61.9 ±7.2	38.4 ±5.3	29.4 ±3.7	45.0 ±6.2	66.3 ±5.0
3	46.2 ±4.6	38.7 ±4.2	48.3 ±5.3	66.1 ±7.2	46.4 ±5.3	42.3 ±3.7	52.5 ±6.3	62.1 ±4.7	38.6 ±5.8	30.1 ±3.7	46.8 ±5.3	68.5 ±7.2
4	48.2 ±7.3	46.3 ±6.2	51.1 ±6.3	66.3 ±7.3	47.7 ±4.6	45.8 ±6.3	52.7 ±5.3	65.6 ±7.0	38.6 ±3.6	36.1 ±2.7	47.3 ±5.2	68.5 ±6.3

a : Mean value of 5 replications
b : Standard deviation of 5 replications
1*, 2*, 3*, 4* : See Table 2.

total impregnation time. Variation of board density by the wood species was not found to be clearly different between methods of no pressure, vacuum, and no pressure after vacuum. But the board density by the impregnation through no pressure after vacuum with ultrasonic waves were higher: 0.85 to 0.87 g/cm^3 for *P. densiflora*; 0.84 to 0.86 g/cm^3 for *L. kaemferi*; 0.83 to 0.86 g/cm^3 for *P. koraiensis*. These higher densities might be caused by a large amount of resin impregnated. From these results, the effect of ultrasonic waves, which was taken simultaneously with vacuum as a way of impregnation, was identified but no difference in density of boards after impregnation between wood species was found. Table 4 indicates the resin content by the impregnation method and time at a given density of boards. The resin content increased slowly, regardless of the impregnation method, with increasing the number of cycles. In the impregnation by vacuum, the respective resin content in the board from 1 to 4 cycles were as follows: 32.3 to 46.3% for *P. densiflora;* 37.0 to 45.8% for *L. kaemferi;* 29.7 to 36.1% for *P. koraiensis.* These resin contents were the lowest of four methods, so little effect was found in impregnation by vacuum only. On the other hand, in no pressure after vacuum with simultaneous ultrasonic waves, the resin content was 65% or higher. This resin content was higher than in other methods, and appeared to be higher than in no pressure after vacuum by 15.2% for *P. densiflora*; 12.9% for *L. kaemferi*; 21.2% for *P. koraiensis* 21.2%. This shows a positive effect of ultrasonic waves. The resin content of the board from *P. koraiensis* was lower than that of any other wood species in no pressure, vacuum, and no pressure after vacuum, but was relatively high in no pressure after vacuum with ultrasonic waves.

3.2. Dimensional Change

Results of the dimensional change in length and thickness by the method and time of impregnation in the board with density of 0.6± 0.01 g/cm^3 are shown in Tables 5 and 6. In the impregnation by no pressure after vacuum with

Table 5. Increase rate of length after impregnation

(unit: %)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Impregnation method / Number of cycle	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	0.7[a] ±0.03[b]	0.6 ±0.02	1.3 ±0.03	1.2 ±0.04	1.2 ±0.03	1.1 ±0.02	1.5 ±0.05	1.5 ±0.03	0.7 ±0.03	0.7 ±0.03	0.9 ±0.03	1.1 ±0.04
2	1.2 ±0.03	0.7 ±0.04	1.4 ±0.06	1.3 ±0.03	1.5 ±0.04	1.2 ±0.03	1.7 ±0.05	1.5 ±0.05	0.8 ±0.03	0.6 ±0.02	1.0 ±0.04	1.3 ±0.05
3	1.1 ±0.04	0.6 ±0.02	0.9 ±0.02	1.4 ±0.03	1.5 ±0.03	1.1 ±0.04	1.8 ±0.02	1.9 ±0.04	0.8 ±0.02	0.5 ±0.02	0.8 ±0.03	1.5 ±0.05
4	1.3 ±0.05	1.1 ±0.03	1.1 ±0.03	1.5 ±0.05	1.4 ±0.04	1.3 ±0.04	1.7 ±0.03	2.0 ±0.05	0.9 ±0.02	0.7 ±0.03	1.1 ±0.04	1.5 ±0.04

a : Mean value of 5 replications
b : Standard deviation of 5 replications
1*, 2*, 3*, 4* : See Table 2.

Table 6. Increase rate of thickness after impregnation

(unit: %)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Impregnation method / Number of cycle	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	6.5[a] ±0.7[b]	9.5 ±0.8	12.4 ±1.3	11.0 ±0.5	10.3 ±0.9	9.5 ±1.0	10.9 ±0.7	11.2 ±2.2	5.8 ±0.4	7.9 ±0.7	12.6 ±1.3	12.8 ±1.9
2	8.9 ±0.8	8.7 ±1.1	10.5 ±0.9	11.6 ±1.5	11.9 ±0.8	10.7 ±1.2	13.1 ±1.3	12.0 ±0.6	9.3 ±0.5	11.2 ±0.7	13.9 ±1.3	13.0 ±0.8
3	10.5 ±0.9	9.9 ±0.7	9.5 ±0.6	11.8 ±0.9	12.0 ±1.2	12.5 ±0.7	12.5 ±1.3	13.5 ±0.8	6.1 ±0.5	9.4 ±0.7	12.5 ±0.9	15.6 ±1.7
4	10.4 ±0.7	9.9 ±0.8	10.6 ±1.3	14.1 ±1.1	12.1 ±0.9	12.5 ±0.8	13.1 ±1.2	17.6 ±2.3	14.1 ±1.2	14.8 ±0.7	15.5 ±0.9	16.7 ±2.2

a : Mean value of 5 replications
b : Standard deviation of 5 replications
1*, 2*, 3*, 4* : See Table 2.

simultaneous ultrasonic waves, the increase rate of length increased as the total impregnation time increased, but showed no uniform trend in the other methods. And the increase rate of length in the impregnation by vacuum was shown as 0.6 to 1.1% in a board for *P. densiflora*, 1.1 to 1.3% for *L. kaemferi*, and 0.5 to 0.7% for *P. koraiensis* and was the lowest of four impregnation methods. Then the increase rate of length by no pressure after the simultaneous vacuum with ultrasonic waves reached up to 1.5% or more in all the wood species. And the increase rate of length of the board from *L. kaemferi* was the highest of all methods of

Table 7. Modulus of rupture after impregnation

(unit: kgf/cm^2)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Number of cycle \ Impregnation method	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	158.3[a] ±20.4[b]	135.1 ±18.2	140.3 ±13.5	156.1 ±23.2	177.6 ±22.7	145.1 ±19.3	145.9 ±17.6	158.3 ±15.3	149.8 ±16.2	156.9 ±17.7	132.0 ±13.2	176.3 ±20.5
2	115.9 ±11.3	133.9 ±16.2	128.1 ±16.3	134.6 ±17.3	162.1 ±17.7	134.6 ±14.5	142.6 ±16.9	134.7 ±14.3	139.3 ±14.78	144.6 ±20.3	123.8 ±10.5	165.6 ±22.4
3	126.0 ±17.0	122.7 ±19.3	148.7 ±20.7	165.6 ±27.2	144.6 ±16.5	112.4 ±10.1	116.3 ±12.3	165.4 ±17.2	154.6 ±18.8	134.1 ±10.7	126.3 ±9.3	179.2 ±19.3
4	146.0 ±12.2	133.9 ±10.3	138.7 ±12.9	154.8 ±17.5	157.8 ±14.3	143.9 ±10.7	143.9 ±12.3	155.2 ±14.1	140.5 ±18.5	132.1 ±12.7	130.8 ±9.4	154.7 ±15.4

a : Mean value of 6 replications
b : Standard deviation of 6 replications
1*, 2*, 3*, 4* : See Table 2.

impregnation. On the other hand, increase rate of thickness by no pressure after vacuum with ultrasonic waves was shown as follows: 11.0 to 14.1% for *P. densiflora*; 11.2 to 17.6% for *L. kaemferi*; 12.8 to 16.7% for *P. koraiensis*. Especially, in no pressure after vacuum with simultaneous ultrasonic waves, the increase rate of thickness was higher than any other impregnation method. Oh (2002) examined the increase rates of thickness and length of the board from a cryptomeria thinned log by impregnating through vacuum with ultrasonic waves in 1 atmospheric pressure for two hours. He reported that the increase rate of thickness in board reached up to 16.3% and that of length up to 1.42% after steaming, but reached up to 10.3% and 8.9% without steaming, respectively. This was similar to the result in this study. Also, Oh (2001) noted in the examination of physical property of woodceramics made from cryptomeria that the increase rate of weight decreased as board density increased. But the decrease rate of length and thickness showed decreasing tendency. From these results, he explained that the difference of the resin content might be affected by the size of manufactured woodceramics.

3.3. Bending Strength and Hardness

Table 7 shows the results of bending strength of impregnated boards by wood species. Bending strength of the board impregnated by no pressure after vacuum with simultaneous ultrasonic waves increased up to three cycles. However, bending strengths of the boards by other methods exhibited no consistent trend. Also, bending strength of the board by no pressure after vacuum with simultaneous ultrasonic waves, on the whole, was higher than that of any other impregnation method. Therefore, there was close correlation between board strength and board density as well as resin content. There was no distinct difference between wood species except for the board from *L. kaemferi* with relatively high bending strength. *P. koraiensis* board impregnated by 3 cycles of no pressure after vacuum with ultrasonic waves was higher in bending strength than any other species.

Table 8 shows the results of Brinell hardness by the impregnation method and time. The

Table 8. Brinell hardness after impregnation

(unit: kg/cm^2)

Species	*P. densiflora*				*L. kaemferi*				*P. koraiensis*			
Impregnation method / Number of cycle	1*	2*	3*	4*	1	2	3	4	1	2	3	4
1	544.0[a] ±62.7[b]	409.3 ±53.5	568.2 ±73.7	634.4 ±47.6	484.1 ±36.2	463.0 ±54.3	400.2 ±30.2	648.2 ±58.7	440.8 ±60.3	471.3 ±35.2	553.2 ±60.3	761.3 ±72.4
2	652.6 ±47.3	476.1 ±25.7	602.4 ±50.6	662.3 ±62.4	465.1 ±39.2	572.5 ±47.3	451.3 ±22.7	780.4 ±63.9	365.2 ±19.2	407.9 ±30.3	547.6 ±46.5	835.3 ±72.5
3	498.8 ±53.3	663.9 ±65.2	573.9 ±33.7	849.2 ±72.0	527.9 ±60.4	561.3 ±43.1	437.9 ±51.6	849.6 ±72.5	413.5 ±30.7	416.3 ±31.2	708.8 ±54.6	894.3 ±72.4
4	746.8 ±81.6	645.9 ±43.2	720.6 ±51.4	851.0 ±45.2	572.0 ±37.3	630.5 ±49.6	497.2 ±52.4	843.9 ±63.2	440.9 ±30.5	439.4 ±27.2	540.4 ±46.5	900.8 ±82.6

a : Mean value of 7 replications
b : Standard deviation of 7 replications 1*, 2*, 3*, 4* : See Table 2.

hardness values by no-pressure after vacuum with ultrasonic waves were as follows: the board from *P. densiflora* 634.4 to 851.0 kg/cm^2; *L. kaemferi* 648.2 to 843.9 kg/cm^2; *P. koraiensis* 761.3 to 900.8 kg/cm^2. The hardness by this method showed increasing trend as the impregnation time increased, and was greater than that of any other impregnation method. When considering these results, the resin content by no pressure after vacuum with ultrasonic waves was higher than that by any other impregnation method. Oh *et.al.* (2000) noted in the relationship between the resin content and bending strength of woodceramics made from Aomori hiba that the higher the resin content, the greater the bending strength. Thus, it was assumed that there was close correlation between the resin content in the board and the performance of woodceramics.

4. CONCLUSIONS

This study explored the physical and mechanical properties of resin impregnated sawdust boards made from the thinned softwood logs of *P. densiflora, L. kaemferi,* and *P. koraiensis* by the impregnation method and time. The sawdust boards were impregnated with phenol-formaldehyde resin by four methods: vacuum, no pressure, no pressure after vacuum, and no pressure after vacuum with ultrasonic waves. The results were as follows:

1) As the number of cycle in impregnation increased, the increase rate of density, thickness, length, and resin content increased.

2) The density, resin content, dimensional change, bending strength, and Brinell hardness of impregnated board by no pressure after vacuum with simultaneous ultrasonic waves were the highest.

3) The bending strength in 1 cycle impregnation was the highest.

4) There was no distinct difference in the physical and mechanical properties between the wood species.

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