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Formaldehyde emissions from particle board made with phenol–urea–formaldehyde resin prepared by different synthesis methods

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This paper suggests the optimum NAF resin preparation to reduce the quantity of pollutant emissions from the particle boards by using PUF resin. The pollutant emissions performance of the prepared NAF resin was evaluated by comparison with eMDI resin. Each PF and PUF resin was made by variation of the molar ratio and synthesis methods according to order of addition of the basic products. In addition, after verifying the properties of the resins, the quantity of pollutant emitted from boards made with the PUF resins were evaluated. Of the cases of boards made with each resin, the formaldehyde emission was the lowest for PB #7. The eMDI resin used in this board showed good quality as an eco-friendly resin, because it had less than half the formaldehyde emission of the other formaldehyde-based resins. In addition, the mechanical properties of the particle board met the KS criteria; therefore, there should be no restriction on use in manufactured goods.

Keywords: formaldehyde; volatile organic compounds; particle board; urea-formaldehyde resin; phenol–urea–formaldehyde resin

1. Introduction

A large amount of construction residue is discarded in the process of building construction, including wood-based products such as those used for molds or buttresses, etc. In addition, because most of the wood is used in cabinetry materials – which continuously need replacement throughout a building's life cycle – a large amount of wood residue is generated. This construction residue is made into particle board. A particle board is a wood-based panel made with raw materials to reduce the environmental load.[1–3] The main material of particle board is crushed chips of a certain size, which are made into eco-friendly wood-based panels through a hot gluing and molding process with synthetic resin adhesive. These wood-based panels have several advantages. First, particle board has no anisotropy of the physical properties because the adhesive is configured to achieve bonding between the particles. Second, it is possible to manufacture a homogenous product without knots, wood rot, distortion, or bending. Third, the specific gravity, thickness, size, and so on can easily be adjusted. Fourth, particle board can easily be machined through the processes of pegging, cutting, drilling, painting, printing, adhesion, and so on. However, because the main material is residue, particle board is only made of reused chips. As a result, a certain level of formaldehyde is emitted

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from the board due to the types of formaldehyde adhesive previously used in the manufacture of the chip board product.[4–6]

In the building environment, of all pollutant sources, wood-based panels having a large surface area are always exposed to indoor air, and affect the indoor air quality by emitting various pollutants. Nowadays, about 95% of newly constructed buildings consist of wood-based panels, including particle board, MDF, and veneer, which are used as the raw materials for cabinetry and flooring. In the case of wood-based building materials, the pollutant emission rate is high due to the effect of resins applied during the production process and present in the timber itself. Formaldehyde especially has a negative effect on human health, causing conditions such as asthma and eye irritation. In addition, the indoor air quality can be worse than that of outdoors due to the emission of pollutants from building materials. Because of these issues, source control, dilution control, and removal control are conducted for improvement of indoor air quality. Furthermore, in the case of source control, many studies have been conducted to reduce the pollutant emission rate of wood-based panels by methods such as control of the molar ratio of applied resin during the production process, the replacement of formaldehyde resins, or the application of additives such as bio-scavengers.[7–9]

With respect to source control, no added formaldehyde (NAF) resin has been used for reducing the formaldehyde emission. In particular, emulsifiable methylene diphenyl diisocyanate (eMDI) is a non-formaldehyde resin of the isocyanate type, and is used to manufacture zero-board which emits less than 1/10 of the formaldehyde of SE0 (super emission zero under 0.3 mg/L) board. This resin has many advantages, such as not requiring additional water in the manufacturing process because the moisture in the wood is used for curing, and adhesion is possible at strands of high moisture content, and increased production in the drying process. While eMDI is easy to manufacture, as outlined above, it also has a weakness. The surface of the particle board adheres to the surface of the press machine when eMDI is sprayed on the surface layer during manufacture.[10,11]

In the present study, resin was the major factor considered for reducing the pollutant emission rate of particle board during the production process. As eMDI has been used as a formaldehyde emission-free NAF resin, it was selected as a reference for evaluation of the suggested NAF resin. Phenol was previously added to reduce formaldehyde emission from the scrap which consists of crushed construction residue containing formaldehyde from the manufacturing phase.[12–15] The optimal resin synthetic method was confirmed through examination of the resin upon variation of the injection timing of phenol, urea, and sodium hydroxide.

2. Materials and methods

2.1. Resin synthesis

For synthesis of the resins, seven resins were prepared from phenol–formaldehyde (PF) and phenol–urea–formaldehyde (PUF). The components of each resin are listed in Table 1. Resins #1 and #2 were synthesized by varying the molar ratio of formaldehyde/phenol. The molar ratios of resins #1 and #2 were 1.6 and 1.8, respectively. Phenol, formaldehyde, and sodium hydroxide were used as the raw materials, and were added to the reaction vessel according to the calculated amounts. Formaldehyde and phenol were charged in the reaction vessel after which sodium hydroxide was

Table 1. Synthetic methods of resin applied to the surface layer.

Category	Classification	Sample	Remarks
Molar ratio	Changed to molar ratio	#1	Phenol/sodium hydroxide = 4.2, final molar ratio (FMR) = 1.6
		#2	Phenol/sodium hydroxide = 4.2, FMR = 1.8
	Changed to injection timing	#3	Put into twice of phenol and sodium hydroxide, condensation molar ratio (CMR) = 2.0, FMR = 1.6
		#4	Put into twice of phenol and sodium hydroxide, CMR = 2.0, FMR = 1.8
Added urea	Pre-added of urea	#5	Same condition with #4, pre-added of urea
	Post-added of urea	#6	Same condition with #4, post-added of urea
	Split injection	#7	Put into twice of HCHO, phenol, urea, and sodium hydroxide

added with heating. After reaction, the resins were cured with the cooling phase. The sodium hydroxide solution was added to adjust the pH value to the target level, and the solid content of all the synthesized resins was about 53% based on oven-dry measurements.

Resins #3 and #4 were prepared with different injection timings than #1 and #2. Resins #3 and #4 differed from #1 and #2 in that phenol and sodium hydroxide were divided according to the calculated amounts. Phenol and sodium hydroxide were injected into the reaction vessel before reaction of the added elements, after which they were then added again. In the first addition step, the condensation molar ratios (CMR) of #3 and #4 were adjusted to 2.0. In the second addition step, the final molar ratio (FMR) of #3 was adjusted to 1.6, while #4 was adjusted to 1.8.

In the preparation of samples #5 to #7, the CMR and FMR, as well as the method of synthesis were the same as resin #4, with the exception of urea. Before the reaction, urea was added with phenol in the phase of preparing resin #5, while urea was injected after the reaction in #4.

In the synthesis of resin #7, all elements were added in the two steps, and formaldehyde, phenol, urea, and sodium hydroxide were divided according to the calculated amounts. Formaldehyde, phenol, and urea were added first, followed by addition of sodium hydroxide with heating. After reaction of these elements, the second phase of formaldehyde, phenol, and urea was added, and the elements were reacted again. Finally, after cooling, sodium hydroxide was added again to make the PUF resin with a molar ratio of 1.8.

2.2. Characterization of prepared resins

For confirmation of the thermal properties of the prepared resins, the curing behavior of the resins was confirmed using a differential scanning calorimeter (DSC), Perkin-Elmer Pyris 1 DSC with a cooler. The prepared resins were heated from 20 to 250 °C at a heating rate of 10 °C/min in both the first and second heating phases, followed by cooling at the rate of 10 °C/min from 250 to 20 °C in a nitrogen atmosphere. In addition, the thermal degradation of the resins was examined using a thermogravimetric analyzer (TGA), the TGA 2950, TA instruments. The prepared resins were heated from room temperature to 800 °C at the rate of 10 °C/min in a nitrogen atmosphere. Finally, the FT-IR spectra of the samples were measured in the range of 400–4000 cm⁻¹.

2.3. Preparation of wood-based composite

The production and pressing conditions for preparation of the wood-based panel particle board are shown in Table 2. To compare the particle boards according to the resins used, a reference board was prepared using eMDI resin. The pressing conditions of the reference board were the same as those for the particle boards fabricated using the prepared resins. For comparison purposes, each of the prepared resin was applied as a surface layer, while the core layers of all the boards were prepared using eMDI resin. A schematic design of the fabricated board is shown in Figure 1.

2.4. Evaluation of formaldehyde emission rate

The formaldehyde emission rates of the resins according to the method of synthesis were estimated using the desiccator method. The measurement was performed with a desiccator in accordance with JIS A 1460 (JIS 2001). Each specimen was placed in a 10 L glass desiccator for 24 h. The quantity of formaldehyde emitted was determined from the concentration of formaldehyde absorbed in a Petri dish filled with a specified amount of distilled or deionized water. The absorbed formaldehyde was analyzed by means of the chromotropic acid method.[16]

A field and laboratory emission cell (FLEC) was used to determine the adsorption performances of formaldehyde and VOCs. The FLEC was made of acid-resistant stainless steel and had a diameter of 15 cm with the maximum surface area and volume of 0.0177 m² and 0.035 L, respectively. When material was placed over the FLEC, the surface of the material became the bottom of the FLEC. The sample load factor was up to 506 m²/m³ (area of materials/FLEC volume). Dry air (moisture content < 5 ppmv) from a gas cylinder was passed through a water bubbler in an air supply instrument to obtain a relative humidity of 50%. The dry air was then introduced into the inlet of the FLEC and a laminar flow was formed in the slit of the FLEC.[17,18] Following convective mass transfer of air onto the surface material, the air was discharged out of the FLEC. The rate of air exchange was controlled using an air pump, which was fitted with a sensor to monitor the pressure, temperature, and RH of the air.

Table 2. Pressing conditions of particle boards.

Variable	Unit	Prepared boards
Time	Sec	120
Temperature	°C	220
Target thickness	mm	15.0
Target dimension	cm ²	1350 (30 × 45)
Target density	g/cm ³	0.680

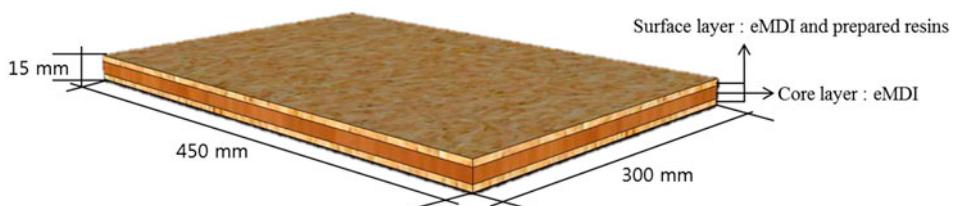


Figure 1. Schematic design of fabricated board.

2.5. Mechanical strength

The three-point bending strength (MOR) and internal bond strength (IB) of the particle boards prepared with seven different resins and with eMDI alone were estimated using a universal testing machine (Hounds Co.). Each value represents the average of eight samples.[19] The bending strength test was used to measure the approximate maximum load (P) at the surface of the specimen by applying a load of about 10 mm/min, and was expressed in the following Equation (1). In addition, for the particle boards of 18.0, 15.0, 13.0, and 8.0 types, the larger value of the horizontal or vertical bending strength was used. The values of two-way bending strength for the plates were 24.0–10.0, 17.5–10.5, and 30.0–15.0.

$$\text{Bending strength (N/mm}^2\text{)} = \frac{3}{2} \times \frac{PL}{bt^2} \quad (1)$$

In Equation (1), P is the maximum load (N), L is the span (mm), and b and t are the width (mm) and thickness (mm) of the specimen, respectively. The IB strength test was used to measure the specimen bonded to a steel or aluminum block, while tensile load was applied perpendicularly to the surface of the specimen. The maximum load (P) was measured when the pull out type fracture occurred, and was calculated with the following Equation (2). The tensile loading rate was 2 mm/min.

$$\text{Internal bond strength (N/mm}^2\text{)} = \frac{P}{bL} \quad (2)$$

In Equation (2), P is the maximum load (N) when the pull out type fracture occurred, and b and L are the width (mm) and length (mm) of the specimen, respectively. For the above, the criteria and qualifications complied with the KS F 3104 in Korea, based on ISO 768:1972: Fiber building boards – Determination of bending strength, ISO/DIS 3931: Fiber boards – Transversal IB, ISO/NP 16893: Wood-based panels – Particleboard, ISO 16893-1:2008: Wood-based panels – Particleboard, and so on.[20]

3. Results and discussion

3.1. Characterization of prepared resins

For confirmation of the heating temperature in the phase of board preparation, the thermal curing properties of the prepared resins were examined using a DSC, as shown in Table 3. The curing temperature ranged from 55 °C to 102 °C. Resin #7 displayed the lowest temperature at the start of curing, at 81 °C, while resin #6 exhibited the highest

Table 3. Curing characteristics of prepared resins by using a DSC.

Samples	Initial temperature (°C)	Peak temperature (°C)	Final temperature (°C)	Curing temperature range (°C)
#1 Resin	85.1	137.1	160.7	75.6
#2 Resin	89.6	142.8	191.2	101.6
#3 Resin	87.1	164.4	182.8	95.6
#4 Resin	88.6	144.4	183.9	95.4
#5 Resin	86.3	124.0	169.9	83.6
#6 Resin	99.6	128.0	154.9	55.3
#7 Resin	81.1	142.7	178.4	97.4

temperature at the start of curing, at 100 °C. Generally, the curing started at about 90 °C. The temperature at completion of curing ranged from 155 to 191 °C, with the highest temperature (191 °C) observed for resin #2 and the lowest (155 °C) for resin #6. Therefore, for the curing of all resins, a temperature of over 191 °C is needed.

The thermal properties of the cured resins were evaluated by the TG method, and the results of TG and DTG for the prepared resins are shown in Figure 2. From the

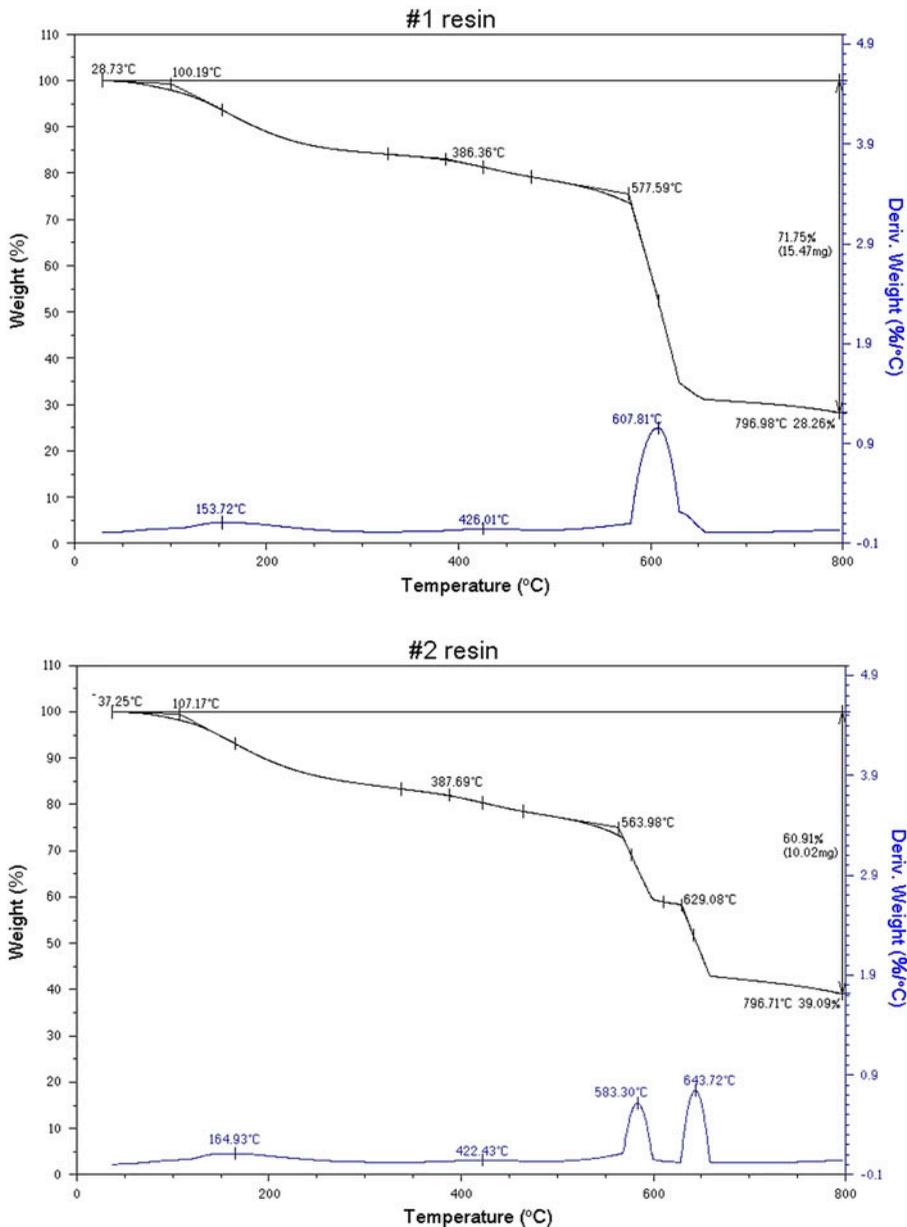


Figure 2. TG and DTG curves for prepared resins.

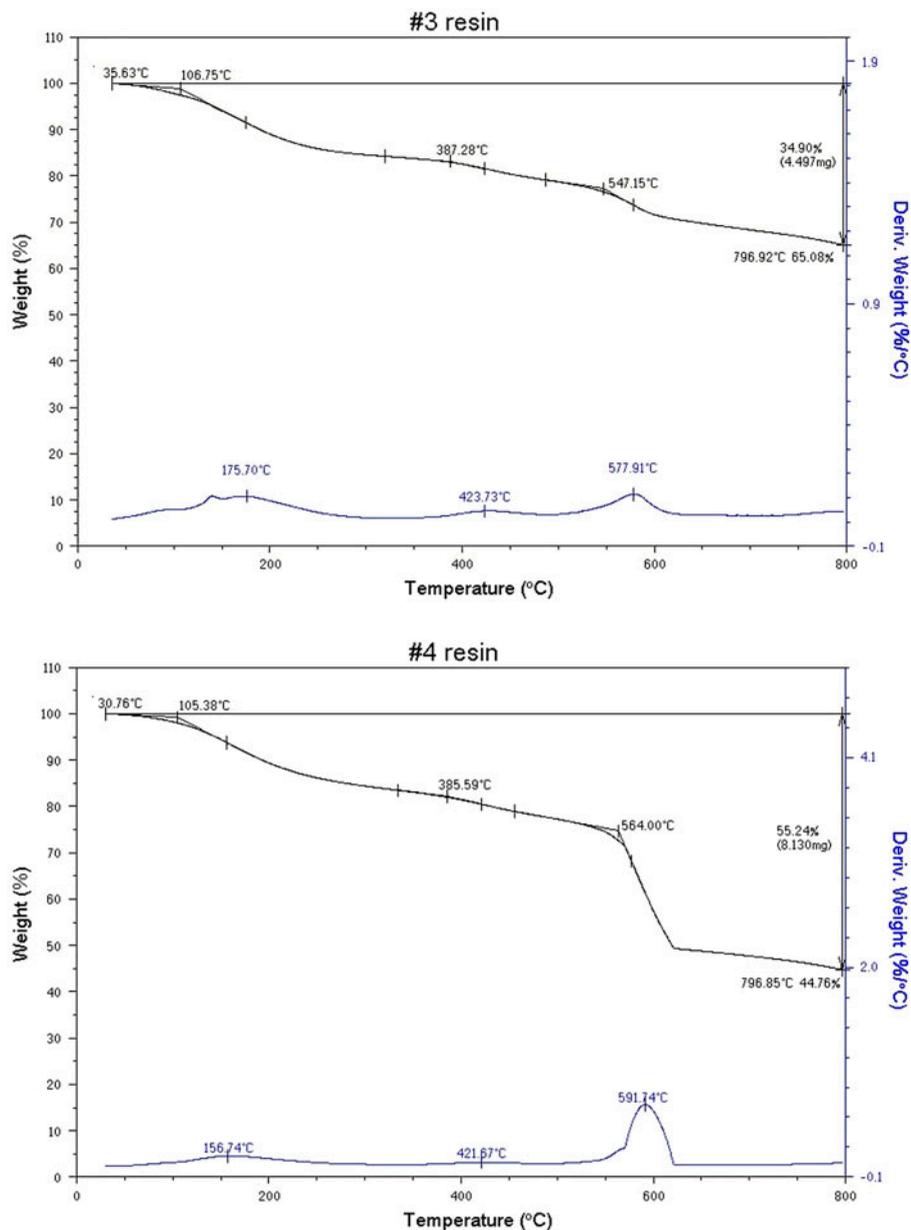


Figure 2. (Continued)

DTG profiles for the rate of weight loss, all prepared resins underwent thermal degradation and decomposition. From the range of temperature at 150–200 °C, the weight of all the resins was reduced by ca. 10% due to removal of the terminal groups and further cross-linking/condensation reactions. From the range of 200–550 °C, the weight of the PF and PUF resins decreased steadily by ca. 10% and 20%, respectively, because of the decomposition of bridged methylene into both phenol and cresol

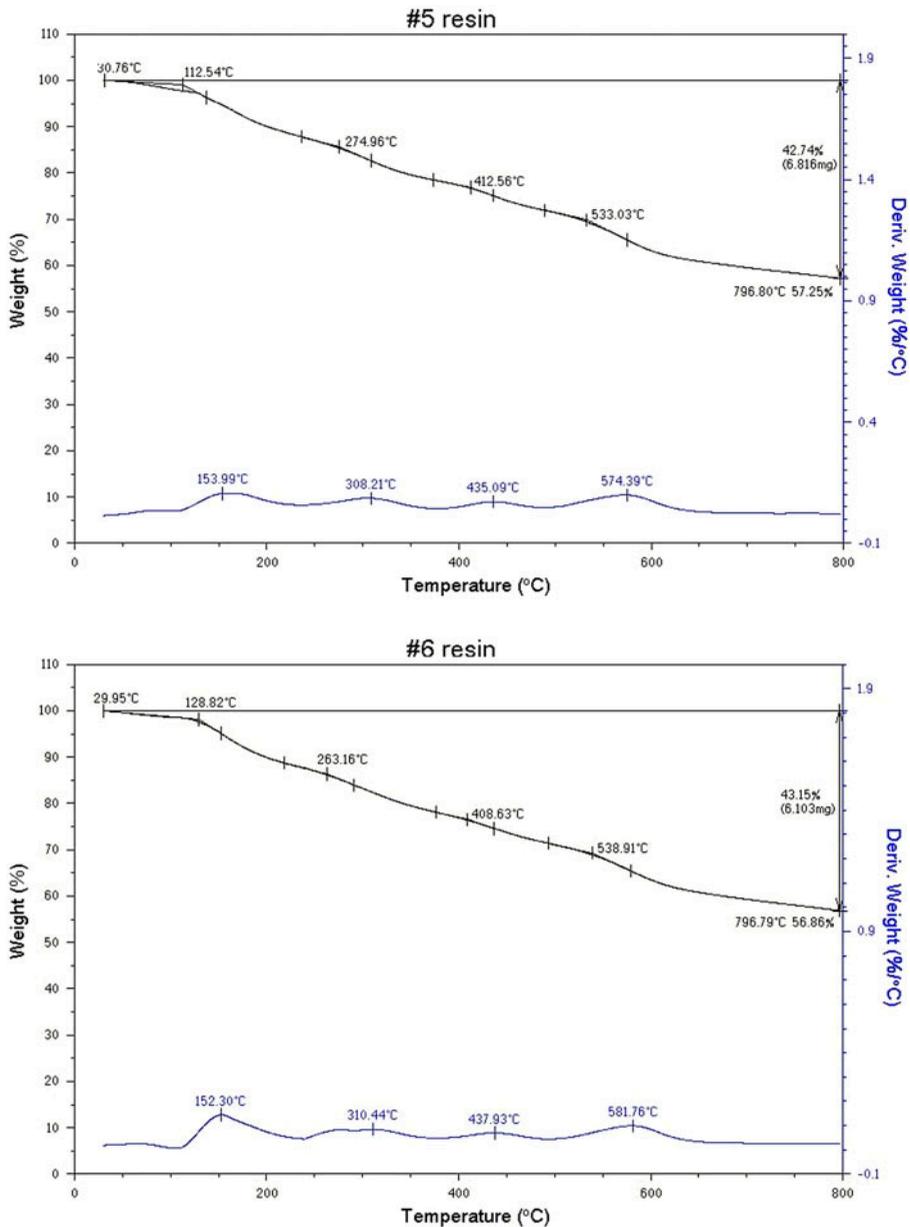


Figure 2. (Continued)

homolog. From the temperatures at 550–650 °C, the weight of PF dropped by ca. 50% from further degradation of the phenols to a carbonaceous structure.

From the DSC and TGA technical results, it could be determined that the curing of all prepared resins was completed at over 191 °C, and the weight loss of all resins was reduced by only ca. 10% at 220 °C. Therefore, the temperature of hot pressing was adjusted to the same temperature of 220 °C for all prepared boards.

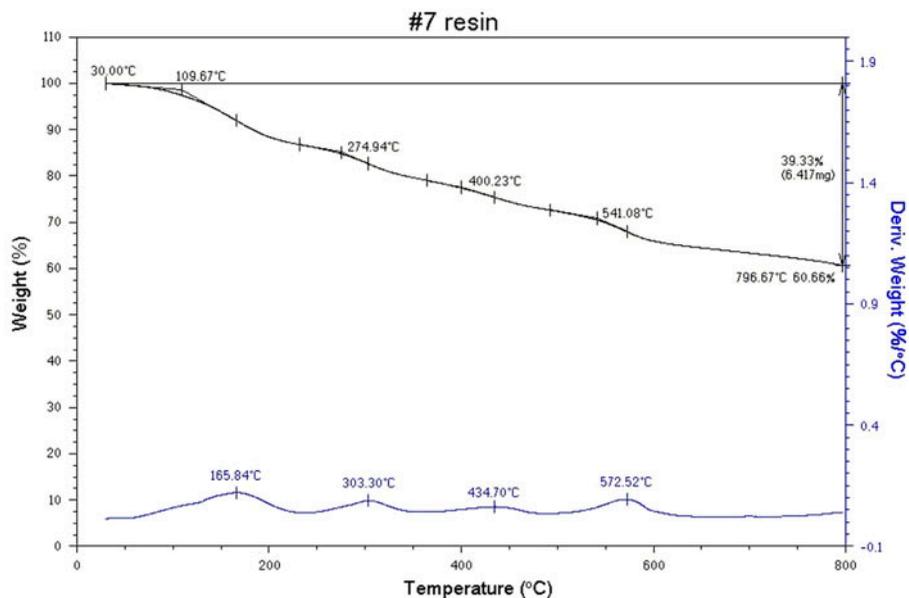


Figure 2. (Continued)

The prepared resins have the potential to contain higher amounts of formaldehyde than conventional resins, or may include other pollutants due to unintended chemical bonding because of alteration of the method of resin synthesis. These changes were also identified via FT-IR. The adequacy of the synthesis and physicochemical

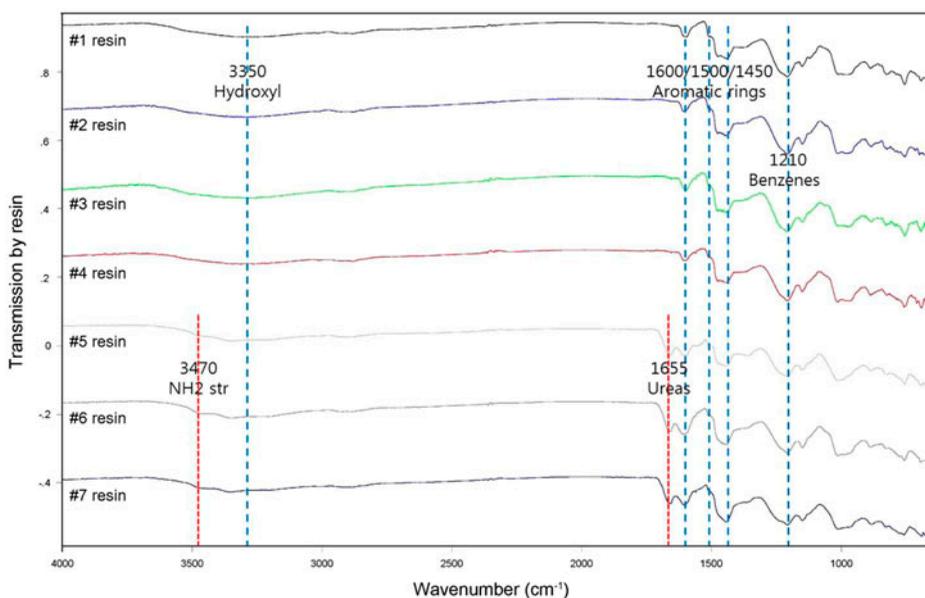


Figure 3. FT-IR spectra of prepared resins by synthesis.

combination of changes to the prepared resins were confirmed, the results of which are shown in Figure 3. Each resin had 5 peaks in common. These included the hydroxyl group at 3100–3700 cm^{-1} (stretching), the phenyl ring at 1600 cm^{-1} (skeleton), the condensation reaction, aliphatic CH_2 at 1480–1450 cm^{-1} (asymmetric bending), and 1,2,3-tribstituted benzenes at 1210 cm^{-1} .

In the case of PUF resins, two additional peaks appeared at 3470 and 1655 cm^{-1} . These were assigned to NH_2 stretching (3470 cm^{-1}) and carbonyl with urea (1655 cm^{-1}). The carbonyl group appeared due to oxidization.

Overall, major differences between the PF and PUF resins were not observed. In addition, there were no differences observed between the prepared resins due to the methods of synthesis. Therefore, the conditions of board production were appropriate and the stability of contaminants was confirmed through FT-IR analysis.

3.2. Formaldehyde emission rate

To reduce the formaldehyde emission rate of particle boards, PF resin was used along with urea as a scavenger. In the present study, particle boards were prepared using seven different types of resins that were prepared by changing either the molar ratio or synthesis method, and the formaldehyde emission rate of the particle board due to the resin applied on the surface layer is shown in Figure 4.

In the case of varying molar ratio, the formaldehyde emission rate of the particle boards prepared with each resin were within the range of 0.7–1.0 mg/L. Board #3 showed lower molar ratio values than 0.7 mg/L, and displayed the same emission rate as the reference board. Board #1 had the emission rate of 0.8 mg/L, which was lower than #2 at 0.9 mg/L. The highest value in the molar ratio category was obtained by board #4, at 1.0 mg/L. Boards with resins of low molar ratio emitted less formaldehyde than those with a high molar ratio used for both boards #1 and #2, due to increase of

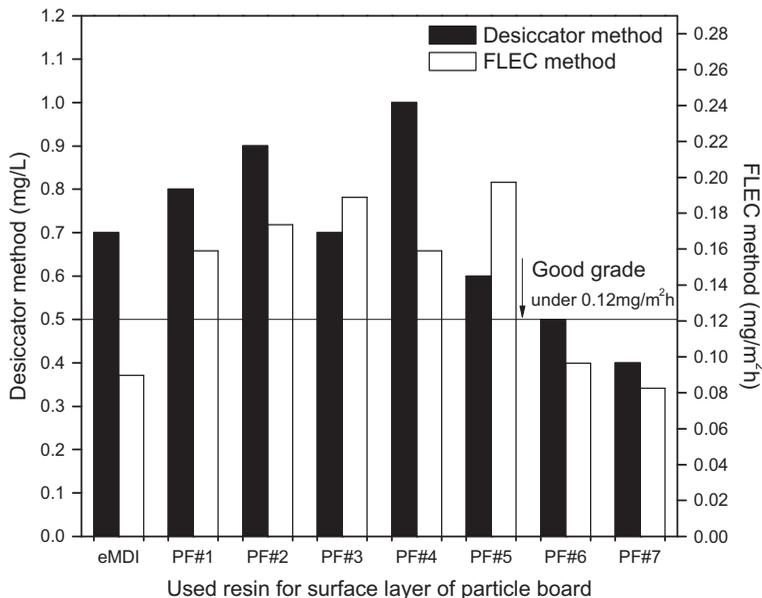


Figure 4. Comparison of formaldehyde emission rates of particle boards between desiccator method and FLEC.

formalin contained in the particle board from the increase of formaldehyde added to the resin. In the cases of boards #3 and #4, however, because the phenol and sodium hydroxide were added twice, the board with resins of a high molar ratio emitted less formaldehyde than that with resins of a low molar ratio.

In the case of PUF, the formaldehyde emission rate of the particle boards made with each resin were within the range of 0.4–0.6 mg/L. Especially, boards #6 and #7 met the value to the level of E0 (under 0.5 mg/L). While board #5 displayed higher values than those of the PUF category, at 0.6 mg/L, it had lower emission than the reference board. The emission rates of boards #6 and #7 were 0.5 mg/L and 0.4 mg/L, respectively. In the cases of #5–#7, the formaldehyde emission rate was reduced by about 20% because the urea added played the role of a scavenger. In addition, the particle boards made with resins #5, #6, and #7 showed a difference of emission rates of about 20%; nevertheless, the boards were made of resins of the same materials. Therefore, changing the order of resin synthesis is applicable for reducing the formaldehyde emissions.

The formaldehyde emission of board #7 was particularly reduced by about 40% compared with the reference board. To date, eMDI resin has been used as a formaldehyde-free eco-resin; however, from these results, it was confirmed that resin #7 can be substituted for eMDI. In other words, #7 has the advantages of a low formaldehyde emission rate, and not sticking to the surface of the press machine when eMDI is sprayed on the surface layer of the particle board during manufacture.

For confirmation of the formaldehyde emitted from the particle board, FLEC was utilized and compared to the desiccator method, as shown in Figure 4. Most of the values obtained by FLEC displayed a similar tendency as those determined by the desiccator method. However, the FLEC values for #3–#5 displayed a slightly different tendency compared to the values obtained by the desiccator method. The reason for the difference is that the desiccator method measures the dissolved formaldehyde concentration in water, while the FLEC method measures the formaldehyde emission rate from the surface of the specimen. In addition, the emission rates measured by FLEC can vary due to the differences of exposed surface area when each specimen has a different surface flatness.

In the case of formaldehyde emission from the particle boards, board #5 displayed a higher value, while #7 had a lower value than the other boards. The formaldehyde emission rate of the reference board met the grade for healthy building material (HB) mark of 0.0898 mg/m² h according to the Air Cleaning Association in Korea. The HB mark has various grades, whereby the best grade for formaldehyde emission rate is under 0.015 mg/m² h, excellent grade is under 0.05 mg/m² h and good grade is 0.12 mg/m² h. The emission rates of boards #1 to #4, made with PF resins, were above the level for good grade of the HB mark, with values of 0.0898, 0.1590, 0.1736, and 0.1890 mg/m² h, respectively. In contrast, all boards made with PUF resins, except #5 in which urea was added before the reaction, satisfied the good grade. The formaldehyde emission rate of board #6 was 0.0965 mg/m² h, while that of board #7 board was 0.0826 mg/m² h, displayed a lower value than the reference board. In other words, through both the desiccator method and FLEC method, board #7 was confirmed to have the lowest formaldehyde emission rate.

3.3. Mechanical properties

The mechanical properties of the particle boards made with eMDI and the prepared resins are shown in Figure 5. Of all the boards, those using the prepared resins showed higher values than that of the reference board.

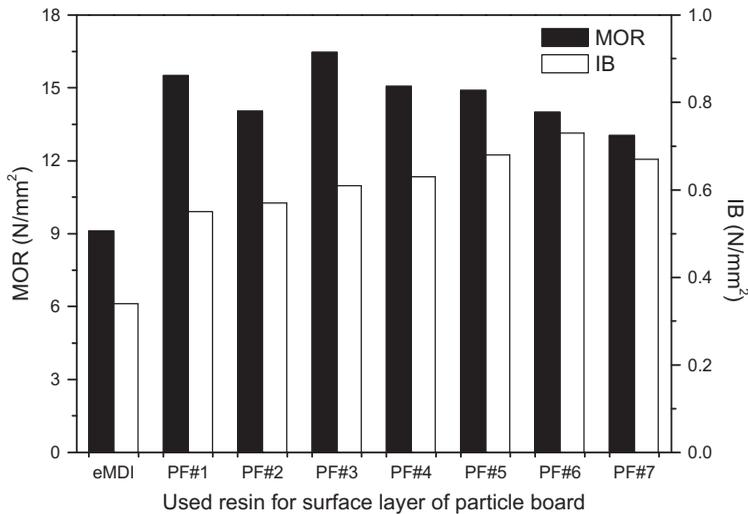


Figure 5. Mechanical properties of particle boards with various resins for surface layer using a universal testing machine.

In the case of bending strength, the reference board made with eMDI alone had a value of 9.10 N/mm^2 . When the PF resin was applied as a surface layer, all prepared boards displayed higher values than for those prepared with the PUF resin. Board #3 had the highest value of 16.46 N/mm^2 , while board #1 had the second highest value of 15.51 N/mm^2 . The next highest value was that of board #4 at 15.07 N/mm^2 , followed by #5 at 14.90 N/mm^2 . Board #5 displayed a higher value than #2 despite using PUF resin as the surface layer. Among the PF resins, board #2 had the fifth highest value at 14.04 N/mm^2 , and was the only board with a value lower than #4, made with PUF. The bending strengths of boards #6 and #7 were 13.99 and 13.04 N/mm^2 , respectively, which were lower than the values of the particle boards made with the other prepared resins. Board #3 displayed the highest value of 16.46 N/mm^2 , while #7 showed the lowest value of 13.04 N/mm^2 . However, the value of board #7 was still 1.4 times higher than that of the reference board, despite being the lowest.

In the case of IB strength, the reference board made with eMDI had a value of 0.34 N/mm^2 . When the PF and PUF resins were used as the surface layer, each of the prepared boards displayed higher IB strength than that of the reference board. Unlike the case of bending strength, the values of IB strength increased according to sample number. The IB strengths of boards #1 and #2 were 0.55 and 0.57 N/mm^2 , respectively. These values were lower than those of the other prepared boards; however, they were about 1.6 times higher than that of the reference board. The IB strength of board #3 was 0.61 N/mm^2 , and that of #4 was the highest among the boards prepared with PF resins, at 0.63 N/mm^2 . The IB strengths of boards #5–#7 were 0.68 , 0.73 , and 0.67 N/mm^2 , respectively. The value of board #7 was somewhat reduced; however, it was still high compared to the boards prepared using PF resins, as well as the reference board. In other words, the IB strength also increased in accordance to the increase of the number assigned to the board. For board #7, however, the IB strength decreased more than in the #6 because the order of resin synthesis was changed. Board #6 displayed the highest value at 0.73 N/mm^2 , which was 2.1 times higher than that of the reference board, while

board #7 showed a value of 0.63 N/mm^2 , which was 2.0 times higher than that of the reference board.

4. Conclusion

The aim of this experiment was to assist in reduction of the environmental load through the manufacture of particle boards, particularly by manufacturing non-emission formaldehyde particle board through inherent formaldehyde removal using NAF resin. To reduce the formaldehyde emission, the order of synthesis of the resin used in the particle boards was changed. Characteristics of the prepared resins were then determined by DSC, TGA, and FT-IR, and the results showed conformity. Particle boards prepared with resins which utilized various methods of synthesis displayed varying formaldehyde emission rates and values of the mechanical properties. In particular, #7 showed the lowest emission rate of formaldehyde, and it also had the second highest value of IB strength and the lowest value of bonding strength; however, these values were 1.4 times and 2.0 times those of the reference board, respectively. In terms of formaldehyde emission rates, while the goal was not achieved, the E0 rating (under 0.5 mg/L) was satisfied. In addition, the mechanical properties of the particle boards met the criteria for the 13.0 type particle board specified in KS F 3104: particle boards. The requirements for 13.0 type particle board are as follows: (1) bonding strength over 13.0 N/mm^2 in both the length and width directions and (2) IB strength over 0.20 N/mm^2 . The board made with PUF resin #7 displayed the formaldehyde emission rate of 0.4 mg/L , and had the mechanical properties of 13.04 N/mm^2 bonding strength and IB strength of 0.67 N/mm^2 . Therefore, particle boards made with PUF resin #7 were identified as having sufficient efficacy performance as a trial product.

Disclosure statement

No potential conflict of interest was reported by the authors.

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