

In: Moslemi, Al, ed. Inorganic bonded wood and fiber composite materials: Proceedings of the 2d international inorganic bonded wood and fiber composite materials conference; 1990 October 14-17; Moscow, ID. Madison, WI: Forest Products Research Society; 1991: 18-27.

## Technologies for rapid production of mineral-bonded wood composite boards

Marull H. Simatupang  
 Norbert Seddig  
 Christoph Habighorst  
 Robert L. Geimer

### Abstract

Existing technologies that reduce the production time of gypsum, magnesia, and portland cement-bonded wood composite boards are reviewed. Experimental data that verify these technologies are given, and new concepts are presented. Adding an ammonium lignosulfonate retarder, an ammonium sulfate accelerator, and gypsum dihydrate to provide a crystallization nuclei resulted in an open time of 4 minutes and a setting time of 2 minutes for boards made with **b**-gypsum hemihydrate. Steam injection during pressing of magnesia-bonded particleboards reduced press time to 15 seconds per millimeter of board thickness. Adding carbon dioxide as a gas injection or a carbonate allowed the production of rapid-setting cement-bonded particleboards. A fast-hardening cement mixture that is not retarded by soluble carbohydrates and tannins can be obtained by mixing portland cement, high alumina cement, and **b**-gypsum hemihydrate.

Wood composites manufactured with inorganic binders may contain 30 to 90 percent by weight of binder components. The three most important inorganic binders used in such composites (gypsum, magnesia cement, and portland cement) are generally crystalline in nature.

The setting, hardening, or hydration of inorganic binders is a slow process when compared to boards bonded with

thermosetting resins. Hydration temperature curves illustrating the time-related process of hydration are shown for **b**-gypsum hemihydrate, magnesium chloride cement, magnesium sulfate cement, and portland cement in Figure 1. The course of hydration is distinguished by two stages. The first stage or "open time" begins with the addition of water and ends when the binder starts to set. During this period, the cement paste is still plastic and can be formed. Mixing and forming of the furnish must be performed within the open time. Prior to the start of the second stage or "setting" period, pressing of the green board should commence. Pressing should continue until the maximum hydration temperature is attained. The time from mixing the water with the binder until the maximum hydration temperature is designated as total hydration time.

In the manufacture of rapid-setting boards, the course of hydration is altered. Generally, the setting time is shortened. However, the open time must be sufficiently long enough to enable proper mixing and forming of the furnish. Retarders and accelerators are often used to prolong the open time or shorten the setting time; retarders and accelerators may be added individually or in combination. In general, additives influence the hydration reaction by modifying the crystallization process. A retarder hampers the solution of the binder, thereby delaying hydration. An

The authors are from the Federal Research Center for Forestry and Forest Products, Institute of Wood Chemistry and Chemical Technology of Wood Hamburg, Germany (Simatupang); Faculty of Natural Sciences, University of Hamburg, Hamburg, Germany (Seddig Habighorst); and USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin (Geimer).

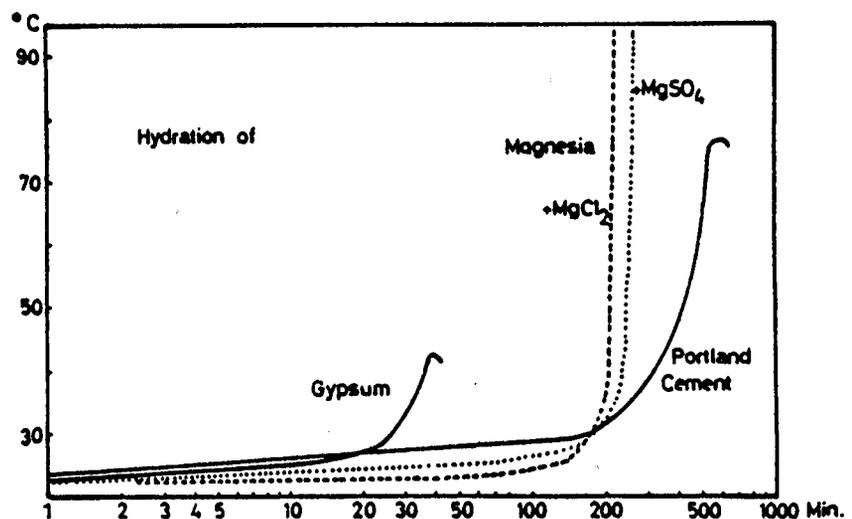


Figure 1. - Hydration temperature curve of gypsum, magnesium oxysulfate cement, magnesium oxychloride cement, and portland cement.

accelerator may enhance hydration by: 1) forming a supersaturated solution leading to early crystallization; 2) producing crystallization nuclei, such as occurs when gypsum dihydrate is added to  $\beta$ -gypsum hemihydrate; 3) increasing temperature, thus promoting the solubility of the binder and 4) direct reaction with a binder component, such as occurs when carbon dioxide ( $\text{CO}_2$ ) is added to portland cement.

This paper reviews existing technologies that reduce the production time of gypsum, magnesia, and portland cement-bonded wood composite boards. It also reports results of our research carried out in Hamburg, Germany, and Madison, Wisconsin, that verify these technologies and introduce new concepts.

### Gypsum-bonded composite boards

Gypsum wallboard is produced in large quantities throughout the world for use as sheathing material in interior applications. These boards are made from foamed  $\beta$ -gypsum hemihydrate (plaster of paris) and derive their bending strength from a thin layer of paper overlay. Addition of additives regulate the setting behavior and permit production on a continuous basis using a belt conveyor (36). Generally, lignosul-

fonate is added as plasticizer to reduce the water requirement. Potassium sulfate, water-glass solution, or gypsum dihydrate are commonly used accelerators. Total cure time is approximately 15 minutes at a production line speed of about 30 meters per minute.

The strength-enhancing advantages of mixing wood with an inorganic binder were foreseen as early as 1880 when a German patent was issued that described a light-weight, gypsum-bonded wood wool board (17). Wood composite boards must be pressed or clamped until the gypsum has hardened sufficiently to withstand the springback forces exerted by the wood particles or fiber. Gypsum particleboards are made commercially in a discontinuous process requiring 2 hours of clamping time (26). The long press time is needed because of the retarders used to obtain the required open time. Continuous production of gypsum-bonded particleboard on a pilot-plant scale has been reported by Bucking (2), but the 6-minute press time is still too long for profitable commercial operation.

Gypsum fiberboard has less Springback than gypsum-bonded particleboard and is produced commercially on a continuous basis. Cure times are ap-

proximately 15 minutes, similar to pure gypsum wallboard, but production line speed is limited to approximately 10 meters per minute. Any attempt to increase the setting rate of gypsum must consider the time needed to mix and form the composite ingredients. Several methods including the encapsulation of gypsum dihydrate with starch (3) have been used to mask the effect of an accelerator to allow ample open time.

Bucking (2) reported a method specific to the production of three-layer gypsum-bonded particleboards. The required make water for the entire board was deposited in the middle layer. In addition to air-dried fine wood particles and gypsum hemihydrate, the furnish for the outer layers contained gypsum dihydrate as a nuclei builder. During pressing, moisture diffused from the middle layer to both outer layers, activating the setting of the gypsum hemihydrate. A retarder can be added to regulate the setting time of the gypsum hemihydrate in the middle layer. According to Bucking, the setting time of gypsum is generally 2.5 times the open time. Since the open time was regulated to 24 minutes, the setting or pressing time was 6 min. In another variation of this method, water was added as granulated ice (1).

In our laboratory, we examined combinations of ammonium lignosulfonate as a retarder and ammonium sulfate as a latent accelerator for the production of gypsum particleboards. Three methods to mask the acceleration effect were applied: 1) encapsulation of ammonium sulfate with polyvinyl-propylene (PVP); 2) absorption of ammonium sulfate in fine wood particles, and 3) absorption of ammonium sulfate in a super absorber. In all cases, an ammonium lignosulfonate retarder solution was incorporated into the furnish by mixing with the wood particles. In the first method, encapsulation of ammonium sulfate with the water soluble PVP proved to be very effective in masking the acceleration effect. The masking effect lasted approximately 5 to 6 minutes, which is too long to be used in a continuous process but may have application in production with a single-opening discontinuous press.

In the second method, we treated a small portion of fine wood particles with an ammonium sulfate solution and

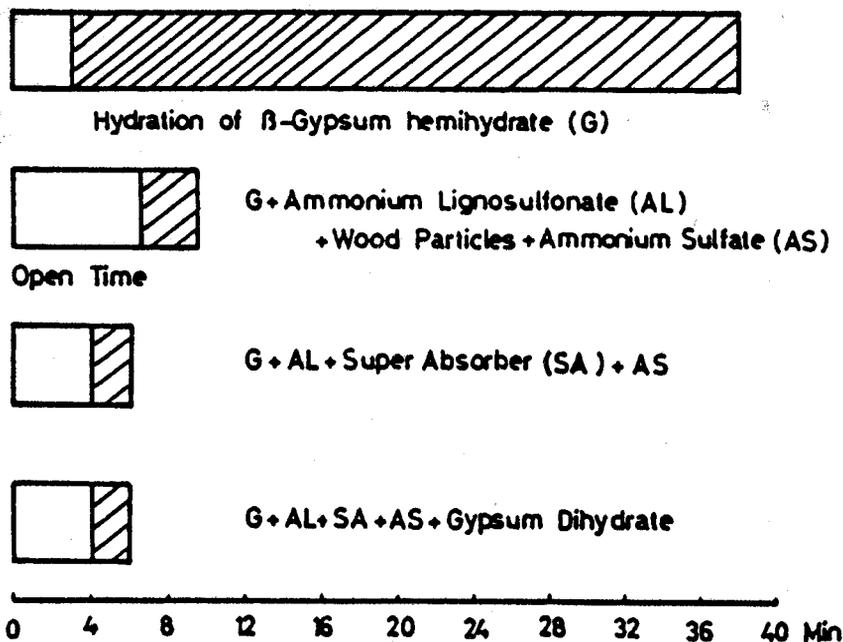


Figure 2. —Schematic of total hydration time of regulated gypsum using various additives. Plain area represents open time; shaded area represents setting time.

subsequently dried at 4°C. The ammonium sulfate was deposited as crystals in the wood particles. The treated wood particles were then mixed with the remainder of the furnish and used to make gypsum-bonded particleboards. The results of bending strength tests showed the feasibility of using a combination of a retarder and a latent accelerator. The ammonium lignosulfonate prolonged the open time of the binder from the usual 3 to 6.5 minutes. The ammonium sulfate proved to be a good accelerator. The setting time of the boards was 3 minutes.

In the third method, the ammonium sulfate accelerator was absorbed in a super absorber (Liquasorb HC 9797). One gram of the super absorber can absorb up to 34 grams of a salt solution. Super absorbers are commercially used in the manufacture of baby diapers and as a moisture retainer in sandy soils. The swollen super absorber is destroyed by light pressure, releasing the absorbed solution. The combination of ammonium lignosulfonate in the furnish and ammonium sulfate in the super absorber enabled an open time of 4 minutes and a setting time of slightly less than 3 minutes. If additional gypsum dihydrate is added to the furnish, a setting time of 2 minutes can be achieved. Our results are summarized in Figure 2.

#### **Magnesia cement-bonded composite boards**

Magnesia cement-bonded excelsior boards were the first inorganic-bonded composite manufactured on a continuous production line. The binder sets within minutes under the influence of higher temperatures (31). Magnesia cement-bonded particleboards were first described by Simatupang and Schwarz (32). Industrial production commencing in 1980 has been described by Loiri (13).

Magnesia cement is classified by Kurodsky and others (10) as chemical-bond cement. The major compound of magnesia cement is magnesium oxide. The minor component, about 10 to 15 percent based on magnesium oxide, is an acid radical, generally in the form of a magnesium salt or an ammonium salt. To prepare magnesia cement, a solution of the minor component is mixed with the magnesium oxide. However, magnesia cement can also be prepared

by reacting magnesium hydroxide with carbon dioxide and heating the formed reaction product.

Magnesium oxide is often referred to as magnesia. Magnesium oxide may be either caustic-calcined magnesia or dead-burned magnesia. Dead-burned magnesia is also called heavy magnesia. The choice of the magnesia form to make magnesia cement is predicated by the chemical composition of the minor component. Caustic-calcined magnesia is obtained by calcining or light burning of magnesium carbonate, magnesium hydroxide, or dolomite. Dolomite is a naturally occurring mineral containing equal amounts of magnesium carbonate and calcium carbonate. If dolomite is calcined below 825°C, only magnesium carbonate is decarbonized, leaving the calcium carbonate intact. Such material is designated as half-calcined dolomite and is also suitable to prepare magnesia cement. Heavy magnesia is obtained by high-temperature treatment (about 1650°C) of either magnesium carbonate or magnesium hydroxide. Caustic-calcined magnesia is suitable to prepare magnesia cement with the addition of magnesium sulfate, magnesium chloride, or magnesium nitrate. Heavy magnesia can be used in combination with a water-soluble ammonium polyphosphate.

Magnesia cement prepared from caustic-calcined magnesia and magnesium sulfate is designated as magnesium oxysulfate cement. The chemical composition of the set cement is determined by the temperature during setting (6). Setting time decreases with increasing temperature. Magnesium oxysulfate cement is used in the production of Heraklith boards and Tectum boards (magnesia cement-bonded wood wool boards) (14).

Magnesia cement prepared from caustic-calcined magnesia and magnesium chloride is designated as magnesium oxychloride cement. Magnesium oxychloride cement is faster setting than magnesium oxysulfate cement (Fig. 1). Magnesium oxychloride-bonded particleboards have been industrially manufactured in Finland (13). Magnesium oxynitrate cement, prepared by the addition of caustic-calcined magnesia and a solution of magnesium nitrate, is a relatively new cement that, as yet, has no industrial application in the produc-

tion of inorganic-bonded wood composites. The setting time is about the same as magnesium oxychloride cement (7).

Cement prepared from caustic-calcined magnesia and carbonic acid is designated as magnesium oxycarbonate cement. It is prepared by obtaining magnesium carbonate trihydrate from the treatment of a magnesium hydroxide slurry with exhaust gas from burners or engines that contain carbon dioxide. The precipitated magnesium carbonate trihydrate is filtered off, mixed with the fiber material, and formed into a mat. When the mat is heated above 80°C, magnesium oxycarbonate is formed along with by-products of carbon dioxide and water (8). Magnesium oxycarbonate cellulose fiberboards, sometimes containing glass-fiber reinforcement, are currently produced in Japan. Another method to make magnesium oxycarbonate cement includes heating a mixture of gel-like basic magnesium carbonate, magnesium hydroxide, pulp, and water. This method produces a lightweight board with good strength properties (34).

Paszner (21,22) utilized heavy magnesia with a water-soluble ammonium polyphosphate to produce magnesium oxyphosphate-bonded wood composites. The setting rate was in the range of 2 to 5 minutes. The open time was too short to perform proper mixing and mat forming. Therefore, dolomite powder was added to regulate the hydration process. Magnesium oxyphosphate-bonded wood composites have better properties than other magnesia cement-bonded composites. However, the process has not been industrially applied, probably as a result of the high cost of heavy magnesia. We have made exploratory experiments to improve the manufacturing process. Instead of using expensive, heavy magnesia, we succeeded in utilizing either caustic-calcined magnesia or half-calcined dolomite. A sparingly soluble ammonium polyphosphate was used as the minor component of the magnesia cement. The 12-mm-thick magnesia-bonded boards were pressed at 120°C for 15 minutes (30). A comparison of these particleboard properties bonded with three kinds of magnesia cement is given in Table 1. The modulus of rupture (MOR) of the three kinds of particleboards show little differences. However, magnesia oxyphosphate-

bonded particleboards swell less and are more water resistant.

As previously stated, elevated temperatures shorten the press time of particleboards bonded with either magnesium sulfate or magnesium chloride. A specific press time of 50 seconds per millimeter of board thickness was reported (13). This time is still too long compared to that of thermosetting resin-bonded wood particleboards. Simatupang (29) showed that the net press time may be short-ended, if the preheating and the decompression times are reduced. At a press temperature of 110° to 120°C, the specific press time was only 30 seconds per millimeter. However, boards with optimum strength properties were produced at a press temperature of 160°C. The specific press time was longer, 60 seconds per millimeter of board thickness, because additional time was necessary to de-gas the board. We succeeded in reducing the specific press time for magnesium oxy-sulfate-bonded particleboards to 15 seconds per millimeter by introducing saturated steam into the center of the board during pressing, similar to that used in thermosetting resin-bonded wood composites (5,35). Using three kinds of caustic-calcined magnesia as the major binder components, we found that less reactive grades resulted in boards with higher MOR values

(Table2). This was true for both conventional and steam-injected boards. Conventional pressing produced boards with higher strength properly values than did the steam-injected boards as a result of an undesirable precure in the steamed boards leading to lower face strength properties.

#### Portland cement-bonded composite boards

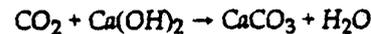
In the conventional manufacture of portland cement-bonded particleboards, the setting time is reduced by the addition of additives or admixtures and by increasing the temperature of the clamped boards. The admixtures, formerly designated as mineralizing agents, are usually one of the following: aluminum sulfate (in combination with either calcium hydroxide (lime water) or water-glass solution), calcium chloride, magnesium chloride, aluminum chloride, calcium formate, or calcium acetate. Nearly every manufacturing plant has its own recipe or secret formulation. The optimum temperature during setting varies from 40° to 80°C and is influenced by the chemical composition of the wood, the type of cement, and the use of admixtures (11). Minimum clamping time is 6 to 8 hours.

The addition of carbon dioxide and the modification of portland cement are two methods currently being used to

reduce the setting time of cement-bonded boards to a few minutes.

#### Addition of carbon dioxide

When carbon dioxide is added to cement according to the following equations, calcium carbonate is formed:



Calcium carbonate provides the initial strength necessary for early release of the board from the press. The carbon dioxide can be obtained from the decomposition of a carbonate or induced as a gas. We examined both variations.

*Addition of carbonates.* - The addition of carbonates is widely practiced in concrete technology. The amount of carbonate, up to about 3 percent by weight, based on cement, is generally added as a powder. The water dissolves the carbonate, and the heat of solution increases the temperature and catalyzes the reaction. In the manufacture of experimental cement-bonded particleboards, as much as 7.5 to 15 percent of either potassium carbonate, sodium carbonate, or ammonium carbonate is added to compensate for the spring-back force of the wood particles. In the presence of powdery carbonate, the open time of the cement is between 2 and 5 minutes.

Portland cement-bonded particleboard made with ammonium carbonate has the advantage of being void of additional cations. These cations, produced when potassium or sodium carbonate is used, may decrease the long-term durability of cellulose. However, boards made with ammonium carbonate emit ammonia that may be a health hazard. Our limited experience with sodium carbonates showed that boards with acceptable properties could be made if the sodium carbonate is free of sodium hydrogen carbonate. Sodium

Table 1. - Properties of magnesia-bonded particleboards manufactured with various acid radicals.

Acid radical	Density oven-dry (kg/m <sup>3</sup> )	MOR	MOE (MPa)	Internal bond	Thickness swell		Water absorption after 24 hours
					After 2 hours	After 24 hours	
Magnesium chloride	1,050 to 1,150	12 to 14	3,000	0.3 to 0.6	2 to 5	6	47
Magnesium sulfate	1,050 to 1,150	10 to 13	3,000 to 3,900	0.7 to 1.1	5	10	50
Ammonium polysphosphate	1.060	11.7	--	0.6	2.5	44	28

Table 2. - Influence of magnesia activity and pressing mode on the properties of magnesia-bonded particleboards.

Binder type	Total hydration time	Maximum temperature (°C)	Press mode	Density oven-dry (kg/m <sup>3</sup> )	MOE	MOE	Internal bond
Martin Marietta 35	51.0	99.5	Conventional	1,090	18.5	6,330	0.9
	51.0	99.5	Steam inject	1,090	16.5	5,600	0.7
Martin Marietta 50	13.4	124.8	Conventional	1,080	13.6	3,140	0.7
	13.4	124.8	Steam inject	1,040	7.2	2,140	0.3
TH	12.2	118.2	Conventional	1,000	10.7	2,630	0.5
	122	118.2	Steam inject	1,010	6.4	2,400	0.4

hydrogen carbonate causes an undesirable presetting of the cement and is unfortunately present in most commercially produced sodium carbonates.

Our results show that the best additive to produce rapid-setting portland cement-bonded boards is potassium carbonate. However, this compound is the most expensive of the three carbon-

ates tested. One method we used was a mixture of potassium carbonate and potassium hydrogen carbonate. By varying the amount of the potassium hydrogen carbonate, the setting time and the pH of the paste can be regulated (Fig. 3). The effect of press temperature on the properties of boards manufactured with potassium carbonate and po-

tassium hydrogen carbonate is tabulated in Table 3. Press temperatures of 70°C produce the highest strength boards. The setting time was only 5.8 minutes. However, the actual press time must be approximately twice as long to de-gas the board and prevent blows. Preheating cannot be used with the combination of potassium carbonate and potassium hydrogen carbonate accelerators because pre-cure takes place during the open time.

Another method using potassium carbonate and water-glass solution will prolong the open time at ambient conditions. In this case, the press time can be further reduced if the furnish is preheated. We used a kitchen microwave to preheat the formed furnish. The mat was immediately pressed after heating. The combination of preheat temperature and actual press time affected the board density and resulting physical properties (Table 4). Acceptable properties can be achieved in a 5 minute press time when the mat is preheated to 75°C. The board was 12 mm thick; therefore, the specific press time was 25 seconds per millimeter; setting time was 2 minutes.

The properties of boards made with the addition of potassium carbonate and water-glass solution are compared to conventionally made boards (Fig. 4). Three kinds of cements, portland (PZ 35F), tuff (Tuff), and high furnace (HFS), were used as the binder. Samples were tested after 28 days and 64 weeks of indoor conditioning and following 32 and 64 weeks of exterior exposure. The results show that conventionally made boards (8-hr. press time) have better bending strength properties than those made of potassium carbonate and water-glass solution. The properties of conventionally made boards also prevail after exterior exposure.

*Carbon dioxide injection.* - Carbon dioxide was reported to be used in some manufacturing plants to accelerate the setting of cement in the industrial manufacture of wood wool boards and Durisol elements (24). Exhaust gases from boilers and engines were the source of the carbon dioxide. These materials are coarse porous, allowing the gas easy access to the cement paste. Since the thickness of the cement paste in wood wool boards is about 0.3 mm (28), the reaction is nearly complete.

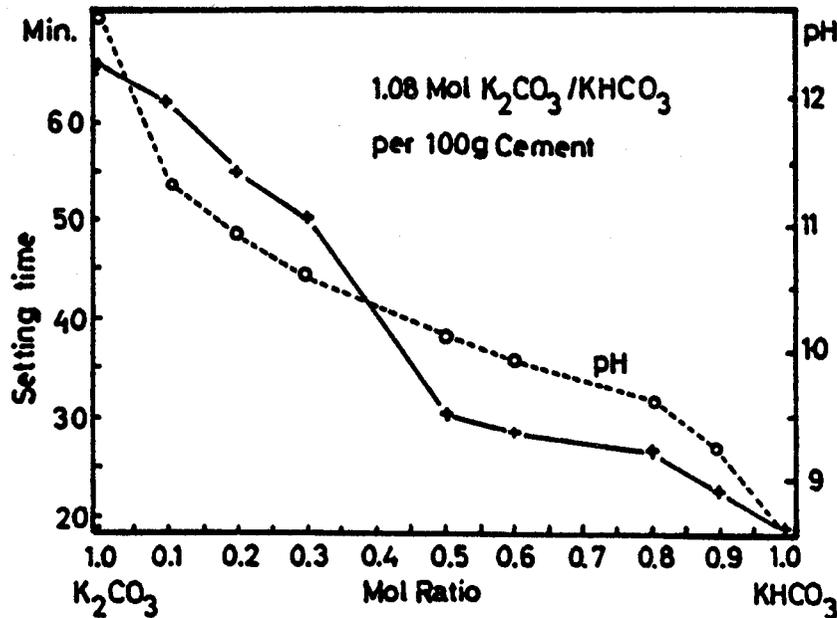


Figure 3. - Setting time and pH of cement paste containing various ratios of potassium carbonate and potassium hydrogen carbonate.

Table 3. - Influence of press temperature on the setting time of cement-bonded particleboard with carbonate addition.

Press temperature (°C)	setting time (min.)	Density oven-dry <sup>a</sup> (kg/m <sup>3</sup> )	Modulus of rupture (MPa)	Modulus of elasticity (MPa)
60	6.5	1,130	8.1	2,430
70	5.8	1,130	10.3	3,210
80	5.8	1,150	8.6	2,450
95	5.6	1,180	8.5	2,540

Table 4. - Influence of microwave preheating temperature on the properties of cement-bonded wood particleboards with potassium carbonate and water.

Reheating (°C)	Press time (min.)	Density oven-dry <sup>a</sup> (kg/m <sup>3</sup> )	Modulus of rupture (MPa)	Modulus of elasticity (MPa)
50	10	1,200	9.5	2,710
75	10	1,210	9.7	3,510
75	5	1,170	9.1	2,700
85	10	1,230	9.9	4,200
85	7	1,200	7.9	2,680
85	5	1,010	3.7	710
95	10	unstable board		

<sup>a</sup> Cement PZ 35F + 13 percent potassium carbonate + 1.5 percent water-glass solution. Press temperature 85°C; spruce wood particles.

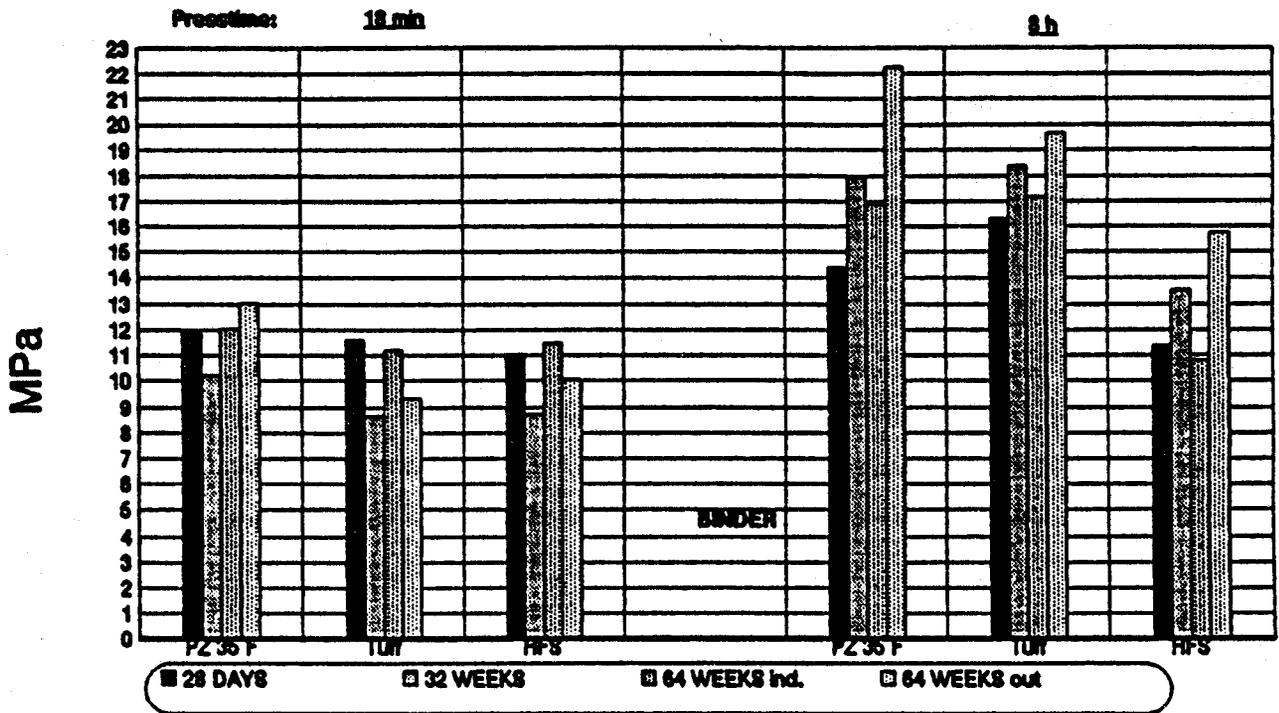


Figure 4. – Modulus of rupture of conventionally pressed and rapid-setting cement-bonded particleboards fabricated with three kinds of cement, after conditioning indoors for 28 days and 64 weeks and after outdoor exposure for 32 and 64 weeks. Conventionally pressed boards: press time 8 hours; accelerator 3 percent calcium chloride. Rapid setting boards: press time 18 minutes, accelerator 13 percent potassium carbonate plus 1.5 percent water-glass solution.

According to a British patent, carbon dioxide is used to make calcium carbonate-bonded fiber boards with good mechanical properties (16). The green boards, made on a Hatschek machine, were heated to 60°C and subsequently treated with carbon dioxide.

A Japanese patent describes the application of carbon dioxide to accelerate the hardening of cement-bonded fiber boards. In this process, the green boards are also heated to lower the moisture content and improve the porosity (15). According to a new process developed in Hungary (9,25), carbon dioxide is injected during the pressing of portland cement-bonded particleboards. The process resembles the injection of saturated steam during the pressing of phenol formaldehyde-bonded particleboards (5).

We have studied the basic reaction between cement and carbon dioxide using the apparatus shown in Figure 5. This device monitors the setting of cement paste under the influence of carbon dioxide by measuring the electrical

conductivity. The apparatus consists of a pressure vessel connected to a carbon dioxide source and a vacuum pump. A slightly modified spark plug is screwed into the pressure vessel. Cement paste (0.8 mm thick) is applied between the two electrodes of the spark plug. When a vacuum is applied, the electrical conductivity decreases as a result of moisture evaporation. When the specific electrical conductivity falls to 0.3 ms cm<sup>-1</sup>, carbon dioxide is introduced. Typical conductivity at a carbon dioxide pressure of 7 bar is shown in Figure 6. As soon as the carbon dioxide is introduced, the cement paste at the surface rapidly carbonizes and the electrical conductivity sharply decreases. Carbon dioxide pressure does not influence this initial reaction. The electrical conductivity comes to a stop and rises slightly again, probably resulting from the formation of calcium hydrogen carbonate. Carbon dioxide then diffuses into the cement paste and the electrical conductivity decreases steadily to a plateau. The inflexion point in the last part of the

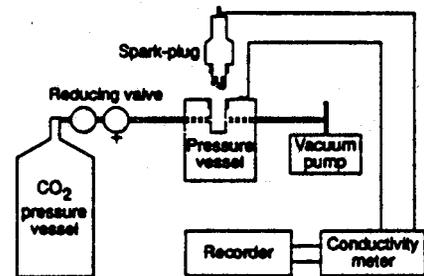


Figure 5. – Schematic of apparatus to monitor setting of cement paste by measuring electrical conductivity at various carbon dioxide pressures.

curve is considered to be the setting time of the cement paste. With increasing carbon dioxide pressure, the carbonization or setting time is shorter.

We made experimental boards using a press platen that enabled the injection of carbon dioxide during pressing (Fig. 7). A heating element was used in the carbon dioxide pressure supply line to prevent the gas from freezing. Gas was introduced only from the upper platen. A metal screen was placed on the bot-

tom platen. A seal was used to ensure a gas-tight compartment in the hydraulic press and to prevent loss of carbon dioxide. Experience has shown that with steam injection of particleboards, gas should be introduced before the furnish

is pressed to the ultimate board thickness. However, sufficient density must be attained prior to gas introduction to prevent the mat from being blown away by the gas. When we used this procedure to press cement-bonded particle-

boards, good distribution of the injected carbon dioxide was obtained. However, bending strength values decreased, as a result of presetting the cement on the surface of the wood particles. If carbon dioxide was introduced after the furnish was pressed to ultimate thickness, distribution of injected carbon dioxide was poor, as evidenced by the air pockets. However, where air pockets were absent, the bending strength of the boards was good. To avoid presetting and still maintain good distribution, we introduced nitrogen during the early stages of dosing. Subsequently after pressing to the stops, carbon dioxide was introduced.

In another variation, we applied vacuum after pressing, followed by the introduction of carbon dioxide. Before opening the press, vacuum was again applied to reduce the partial pressure and remove water vapor. Temperatures of 115°C were observed in the center of pressed boards. A comparison of board properties pressed according to various methods is shown in Table 5. Boards with good bending strength can be obtained by pressing to ultimate thickness, followed by vacuum treatment and injection of carbon dioxide.

The influence of the carbon dioxide pressure the properties of the boards is shown in Table 6. At pressures under 5 bar, no boards with acceptable properties can be produced. At pressures of 5 to 9 bar, boards show comparable properties and hydration times. However, the hydration temperature almost doubles when pressures increase from 5 to 9 bars. The reason for these large differences is unknown.

Glucose and hydrolyzable tannin compounds, when present at concentrations of 0.25 percent, severely retard the setting of portland cement paste and show less detrimental effect in the presence of carbon dioxide (Table 7). Up to

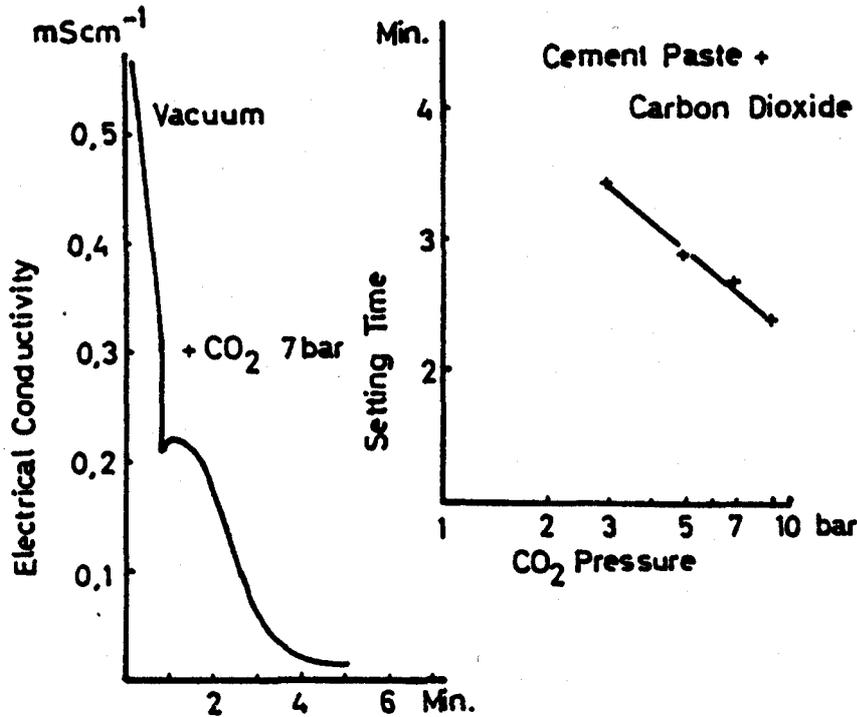


Figure 6. – Electrical conductivity of cement paste at a CO<sub>2</sub> pressure of 7 bar. Influence of carbon dioxide pressure on the setting rate of cement paste.

Table 5. – Influence of mode of carbon dioxide injection on cement-bonded particleboards.

Mode of injection <sup>a</sup>	Setting time (min)	Maximum hydration temperature (°C)	Density oven-dry (kg/m <sup>3</sup> )	Modulus of rupture (MPa)	Carbon dioxide distribution
Preinjection					
Carbon dioxide	1.2	95	1,050	8.0	Satisfactory
Nitrogen	1.5	79	1,090	12.5	Satisfactory
After closing					
Direct	1.1	82	1,090	12.7	Unsatisfactory
After vacuum	1.2	88	1,125	12.5	Good

<sup>a</sup> Carbon dioxide pressure 7 bar; injection from upper platen. Mat enclosed by a seal, provided with a valve to push out trapped air.

Table 6. – Influence of carbon dioxide pressure on cement-bonded particleboards.

Carbon dioxide pressure (bar)	Setting time (min.)	Maximum hydration temperature (°C)	Density oven-dry (kg/m <sup>3</sup> )	Modulus of rupture (MPa)
1.5	26	61.0	-- <sup>a</sup>	--
3	1.8	66.6	--	--
5	1.2	67.1	1,130	13.8
7	1.2	88.4	1,130	12.5
9	1.1	102.4	1,140	12.6

<sup>a</sup> Unsatisfactory board.

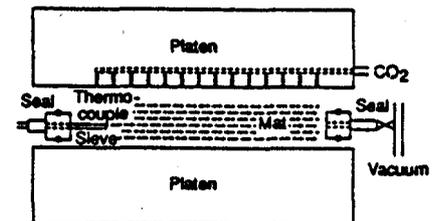


Figure 7. – Schematic of press platen with seal for carbon dioxide injection.

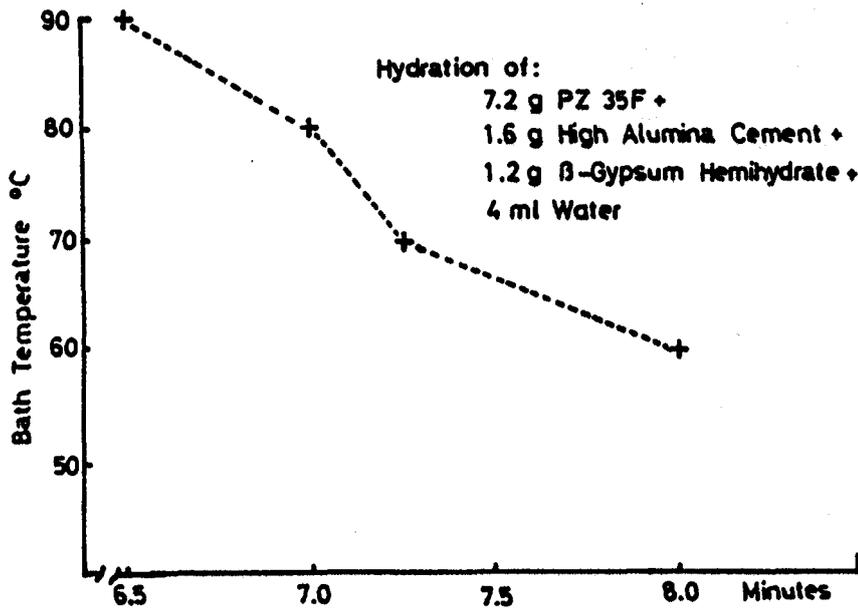


Figure 8. – Total hydration time of a rapid-setting cement mixture.

Table 7. – Influence of glucose or tannin on the setting of cement paste in the presence of carbon dioxide at 7 bar.

Compound	Cement concentration (%)	Setting time	
		Minutes	Relative (%)
Glucose	0.00	4.3	100.0
	0.11	4.3	100.0
	0.23	4.4	102.0
	0.45	4.5	104.7
	0.90	5.0	116.3
	2.25	6.0	139.5
Hydrolyzable tannin	0.00	4.3	100.0
	0.13	3.9	90.7
	0.25	3.7	86.0
	0.50	3.8	88.4
	1.00	4.0	93.0
	2.50	5.5	127.9

Table 8. – Influence of glucose or tannin on the setting time and maximum hydration temperature of a rapid-setting cement mixture.<sup>a</sup>

Compound	Cement concentration	Setting time		Temperature
		Minutes	Relative	
Glucose	0.00	7.0	100.0	83.5
	0.23	4.4	62.9	85
	0.45	4.2	60.0	85
	0.55	4.4	62.8	84
	1.10	5.5	78.6	83
Hydrolyzable tannin	0.00	7.0	100.0	83.5
	0.30	7.0	100.0	87
	0.50	6.4	91.4	87
	0.60	7.5	107.1	86
	1.20	8.0	114.3	85

<sup>a</sup> Cement mixture: 7.2 g PZ 35F + 1.6 g high alumina cement + 1.2 plaster and 4 ml water.

about 0.5 percent of these compounds, based on cement weight, can be tolerated by the gas-injected cement paste.

To determine if conventional equipment can be retrofitted for the introduction of carbon dioxide gas, we used the sealed frame shown in Figure 7 in combination with conventional press platens. Screens were placed on both sides of the mat. After pressing to ultimate thickness, vacuum was applied followed by the introduction of carbon dioxide. Using this arrangement, the setting times were about 1 minute longer when compared with boards pressed with the perforated platens. Properties of the boards made according to both methods are comparable with each other.

#### Modification of portland cement

The use of rapid-setting cement to manufacture wood wool board similar to Heraklith magnesia-bonded boards was described by Pletzer others (23). The cement was a portland cement containing 1 to 30 percent by weight of calcium-halogen aluminate. Another process described in the patent literature enables the production of rapid-hardening wood wool boards using a jet cement and ferric chloride accelerator (33). The press temperature was between 90°C and 120°C. Another type of rapid-setting cement was described by Shigekura (28). This binder, a mixture of  $\beta$ -gypsum hemihydrate and portland cement, sets within 20 minutes.

Okamura and others (19) used ettringite and a hydraulic binder (portland cement) to produce wall or ceiling boards. The ettringite was obtained by reacting aluminum hydroxide sludge with gypsum and calcium oxide. The Sekisui Chemical Co. (27) proposed the use of a mixture of portland cement, high alumina cement, gypsum calcium hydroxide, and eventually sodium citrate to produce particleboards. The United Gypsum Corp. has a patent to manufacture boards with organic aggregates with a binder composed of portland cement, high alumina cement, gypsum, and calcium hydroxide (4). Odler (18) reported the continuous manufacture of a cement-bonded particleboard utilizing a rapid-setting cement, and Paulisan (20) submitted a patent for the production of rapid-setting cement-bonded particleboards. The rapid setting of the binders report-

ed by Sekisui, united Gypsum Corp., Odler, and Paulisan may be a result of the formation of ettringite during the setting reaction.

Little data have been published in scientific journals that describes ettringite enhanced, rapid-setting cement-bonded particleboards. According to Kurdowski and others (10), most expansive cements (cement that expands after setting, in contrast to portland cements that shrink) are based on ettringite. Such cements are generally mixture of portland cement and high alumina cement with the addition of a third component (e.g., gypsum). The setting may be controlled by adding calcium components. We have made some examination with such ternary mixtures. The binder sets within 8 minutes at temperatures above 60°C (Fig. 8). The hydration is not retarded by glucose or hydrolyzable tannins. Both compounds, at concentrations at or over 0.5 percent, showed slightly accelerating effects (Tables 8). A small number of experimental boards were also made, confirming reported data (18,20) that such ternary cements are suitable to produce rapid-setting cement particleboards. Board properties such as weather resistance, thickness swell, water absorption, durability against fungi and insects, and absence of efflorescence can be modified by proper adjustment of the proportion of portland cement, high alumina cement, gypsum hemihydrate, and calcium hydroxide.

### Conclusions

Known methods to reduce the production time of gypsum, magnesia, and portland cement-bonded wood composite boards are reviewed. Experimental data that verify these techniques are given, and new concepts are presented. In many cases, reduction of setting times by the addition of accelerators must be accompanied by the addition of retarders to provide adequate open time to mix and form the furnish.

Inorganic-bonded fiberboard experiences much less springback after pressing than particleboards. The 15 minute setting time of gypsum has permitted the development of continuous production methods for manufacturing gypsum-bonded fiberboard. Laboratory experiments indicate that setting

times as short as 2 minutes may be obtained using ammonium sulfate as an accelerator and gypsum dihydrate to enhance crystallization. However, adequate open time can only be obtained using a technique such as encapsulation (temporary immobilizing the accelerator in wood particles), or a super absorber.

Magnesia cement-bonded wood composites prepared by mixing magnesium oxide with either magnesium sulfate or magnesium chloride experience hydration times of approximately 150 minutes. Much faster rates are obtained by mixing the magnesium oxide with ammonium polyphosphate. All the magnesia boards set faster when heated. Injection of steam into a magnesium oxysulfate-bonded board reduced specific prep times to 15 seconds per millimeter. The technique tends to reduce board strength resulting from a pre-cure effect. We also found that the slower reacting magnesia types were best suited for steam injection pressing.

Injection of carbon dioxide gas into a portland cement-bonded board produces calcium carbonate that provides sufficient initial strength to allow the pressure to be removed in a few minutes rather than hours. This process is being commercially applied in Hungary. The reaction is very fast and requires special techniques, such as preinjection with an inert gas or application of a vacuum to prevent pre-cure. The carbon dioxide can also be obtained by adding a carbonate to the furnish. This compound emits the carbon dioxide gas upon heating. Cement-bonded boards made with carbon dioxide tolerate much higher concentrations of glucose and tannins than do conventional boards.

Some success has been experienced in bonding particleboards using a mixture of portland cement, high alumina cement, and b-gypsum hemihydrate. The binder sets within 8 minutes at temperatures over 60°C and is not affected by soluble carbohydrates or tannins.

### Literature cited

1. Bison-Werke, Baehre und Greten GmbH & Co. KG. 1987. Verfahren zum Aufbereiten von Papier, Schneid-, Hobelspaenen oder dergleichen und zum Herstellen eines Gips-Faserstoff-Gemisches. Offenlegungsschrift DPA 3708875.0.
2. Bucking, G. 1983. Die Herstellung gipsgebundener Spanplatten im Endlosverfahren. Holz Roh-Werkst. 41:427-430.
3. Burkard, E.A. and D.A. Kossuth. 1975. Gypsum Set Accelerator. U.S. Patent 3,920,465.
4. Galer, R.E. and P.C. Web. 1984. Non-expansive, rapid setting cement. U.S. Patent 4,488,909.
5. Geimer, R. 1983. Method of pressing reconstituted lignocellulosic material. U.S. Patent 4,393,019. Assignee, USA represented by the Secretary of Agriculture, Washington, D.C.
6. Henning, O., A. Kuhl, A. Oelschlaeger, and O. Philipp. 1989. Technologie der Bindebaustoffe. Band 1. Eigenschaften, Rohstoffe, Anwendung. 2. Aufl. VEB Verlag fbr Bauwesen, Berlin.
7. Holcombe, C.E. 1978. New magnesium hydroxynitrate binder. J. Mater. Sci. 13:2736-2738.
8. Kirk, R.E. and D.F. Othmer. 1978. Encyclopedia of chemical technology, 3rd ed. John Wiley & Sons, N.Y. Vol. 14.
9. Konkola, J. 1989. Universal building material — entirely building material. In: Proc. Fiber and particleboards bonded with inorganic binders. A.A. Moslemi, ed. Forest Prod. Res. Soc., Madison, Wis. p. 83.
10. Kurdowsky, W., C.M. George, and F.P. Sorrentino. 1986. In: Proc. 8th Inter. Congress on the Chemistry of Cement. Rio de Janeiro, Brasil. 1:304-312.
11. Lange, H. and M.H. Simatupang. 1985. Herstellung von zementgebundenen Fichten- und Pappelspanplatten: Wechselwirkungen von fünf Einflussfaktoren und ihre Wirkung auf die Plattenfestigkeiten. Holz Roh-Werkst. 43:229-236.
12. Liquasorb HC 9797. BASF, D-6700 Ludwigshafen.
13. Loiri, V. 1989. Plant experience in the manufacture of magnesite and slag particleboards. In: Proc. Fiber and particleboards bonded with inorganic binders. A.A. Moslemi, ed. Forest Prod. Res. Soc., Madison, Wis. pp. 110-112.
14. Maloney, T.M. 1977. Modern particleboard and dry-process fiberboard manufacturing. Miller Freeman Publications, Inc., San Francisco, Calif.
15. Masatake, K. and T. Kawaguchi. 1986. Manufacture of curing based boards by carbonation. Japanese Patent 63/55 174 A2. CA 109(6):42675.
16. Moorehead, D.R. and M. Davis. 1979. Method of producing a building element. Great Britain Patent 2,0270,85B.
17. Moraeth, E. 1966. Herstellung und eigenschaften mineralgebundener holzwerkstoffe. In: Sandermann, W. and G. Schmitz, eds. Chemie und chemische technologie mineralgebunde-

- ner holzwerkstoffe. Internationale Arbeitstagung, Hamburg, Germany.
18. Odler, I. 1986. Neuartige umweltfreundliche Werkstoffe und Technologien. Hannover Messe 1986. Inst. f. Nichtmetallische Werkstoffe, Zehntnerstrasse 2A, D-3920 Clausthal-Zellerfeld.
  19. Okamura, T., H. Irifune, and H. Yoshida Kogyo K.K. 1982. Aluminium hydroxide-based building materials and method for manufacturing same. Great Britain Patent 2,083,088.
  20. Paulisan, P. 1989. Verfahren zur Herstellung plattenfoermiger Verbundstoffe. European Patent 89107567.3.
  21. Paszner, L. 1982. Method of adhering mineral deposits in wood fragments surfaces. U.S. Patent 4,339,405.
  22. \_\_\_\_\_. 1986. Private communication.
  23. Pletzer, F., H. Strasser, and H. Tschernuth. 1976. Method of production of cement-bonded molded articles particularly light weight fiber boards. U.S. Patent 3,981,950.
  24. Sandermann, W. 1963. Chemische Holzverwertung. Muenchen. BLV Verlagsgesellschaft.
  25. Schmidt, E. and K. Karpati. 1986. Verfahren zum raschen Binden von Zement ei Fasermaterialien enthaltenden und mit Zement geklebten Platten und Profilen. European Patent 0189127 Bl.
  26. Schwarz, H.B. 1989. Experience in the manufacture of gypsum-bonded boards. *In: Proc. Fiber and particleboards bonded with inorganic binders.* Moslemi, A.A., ed. Forest Prod. Res. Soc., Madison, Wis. pp. 108-109.
  27. Sekisui Chemical Co. 1981. Wood chip cement products. JP 56/140059. Chemical Abstract 96(10):73740.
  28. Shigekura, Y. 1989. Wood fiberboards bonded with inorganic binders in Japan. *In: Proc. Fiber and particleboards bonded with inorganic binders.* A.A. Moslemi, ed. Forest Prod. Res. Soc., Madison, Wis. pp. 141-150.
  29. Simatupang, M.H. 1988. Eignung einiger Magnesiasorten zur Herstellung von Spanplatten. Holz Roh-Werkst. 46:223-229.
  30. \_\_\_\_\_. and R.L. Geimer. 1990. Inorganic binder for wood composites: feasibility and limitations. *In: Proc. 1990 Adhesive symposium (in preparation).*
  31. \_\_\_\_\_. H. Lange, A. Kasim, and N. Seddig. 1989. Influence of wood species on the setting of cement and gypsum. *In: Proc. Fiber and particleboards bonded with inorganic binders.* A.A. Moslemi, ed. Forest Prod. Res. Soc., Madison, Wis. pp. 33-47.
  32. \_\_\_\_\_. and H.G. Schwarz. 1975. Magnesit als Bindemittel fuer Spanplatten. Stuttgart, Germany: Holz-Zentralblatt. 87:1129-1130.
  33. Toyama Prefecture. 1980. Rapid hardening of wood wool-mixed products. Japanese Patent 14,827/80. CA 93(26):244,348.
  34. Ube Slate Kogyo Co. 1980. Lightweight magnesium carbonate plates. Japanese Patent 80 85446. CA 93(24):224,785.
  35. Walter, K. 1989. Dampfpresanlage zur Herstellung von MDF mit hochverdichteten Daeckschichten. Holz Roh-Werkst. 47:117-120.
  36. Wirsching, F. 1976. Gips. *In: Ullmann encyclopaedie der technischen chemie, 4. Aufl., Bd. 12.* Weinheim: Verlag Chemie 289-313.