

PROPERTY ENHANCEMENT OF WOOD COMPOSITES USING GAS INJECTION

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ABSTRACT

Investigations were conducted at the Forest Products Laboratory to extend the technology of steam injection pressing to the injection of other gases. Trimethylborate was injected into phenolic- and isocyanate-bonded flakeboards during

pressing with the object of improving fire resistance. The deposition of borates improved fire resistance, but reduced board strength. Methylformate injected into phenolic-bonded boards prior to a steam injection treatment permitted the reduction of press times. Approximately 90% of

ultimate shear strength and 66% of ultimate bending strength was obtained in 190 seconds. Carbon dioxide introduced into cement-bonded boards permitted the reduction of clamp time from eight to twenty-four hours to four minutes. The carbon dioxide treatment reduces the cure-inhibiting effect of wood and promises to improve the long-term performance of cement-bonded wood composites. Details of problems and process variable interactions are discussed and suggestions are given for further research efforts.

INTRODUCTION

Historically, one of the earliest commercial uses of gas to enhance the pressing of wood composites occurred in 1914 in an Austrian plant that produced inorganic-bonded wood excelsior boards. The process used steam, injected into the mat during the pressing operation, to promote a rapid cure of a magnesium oxide binder (Maloney 1977). Research needed to adapt this process to thermosetting adhesives was conducted during the 1960s, 70s, and 80s (Corbin and Hall 1966; Geimer 1983; Taylor and Reid 1985). This work resulted in the construction of several plants throughout the world during the 1980s. These plants utilize injected steam to cure urea-, phenolic-, and isocyanate-bonded wood composites. The basic advantage of this system is rapid transfer of heat and consequently, a significant reduction in the time necessary to press thick panels. Further adaptation of the steam injection system to deliver a variety of gases has intrigued researchers. Theoretically, it is possible to introduce fire retardants, dimensional stabilizers, catalysts, insect repellents, preservatives, and other property and/or performance enhancers using injection technology.

The purpose of this paper is to present the results of several studies, conducted over the past few years at the Forest Products Laboratory in Madison, Wisconsin, which investigated the introduction of three different gases into wood composites. The gases under consideration were methyl borate (MeB), methyl formate (MeF), and carbon dioxide (CO₂).

Methyl Borate as a Fire Retardant

Methyl borate can be used to enhance the fire performance of cellulosic materials. Investigations at the USDA Southern Regional Research Center (Knoepfler et al. 1975) resulted in the issuance of a patent describing the greatly improved fire resistance of cotton mattresses obtained by treating the material with MeB gas (Knoepfler et al. 1977). We wished to determine if this process could be adapted to the treatment of thermosetting resin-bonded wood composites. Although the presence of borates on wood flakes has been shown to be detrimental to adhesive bond formation (Laufenberg et al. 1986), we conjectured that the detrimental effect might be minimal if treatment was simultaneous with steam injection. Details of the investigation are presented under the section on methyl borate.

Methyl Formate as a Resin Catalyst

At least three, room temperature curing, gas catalyzed resin systems have been developed commercially to replace oven-cured adhesives used in bonding metal casting sand molds. One system trademarked ISOCURE utilizes dimethylethylamine (C₂H₅N(CH₃)₂) gas to rapidly set an isocyanate resin. Another resin system, INSTA-DRAW, uses sulfur dioxide (SO₂) to rapidly cure a furan resin. BETASET is a system that uses methyl formate (HCOOCH₃) to advance the cure of a phenolformaldehyde resin. All three systems have the potential for use in the wood composite industry. Because of the problems associated with the steam injection curing of phenolic resin (Geimer et al. 1991), our efforts were concentrated on BETASET. This system served as the basis for a recent patent (Hickson 1990), which describes its usefulness in manufacturing wood composites. Experimental details are given in the section on methyl formate.

Carbon Dioxide as an Accelerator for Cement-Bonded Wood Composites

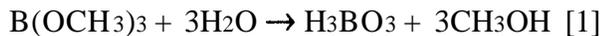
Carbon dioxide (CO₂) is used to promote the initial set of a cement-bonded wood composite. Injection of CO₂ gas reduces the press time of cement-bonded particleboard from eight hours to less than four minutes. This system was tried

on a commercial scale in Hungary (Anonymous 1992), but the inability to obtain consistent quality forced the plant to close after one year. In the past few years, we have performed a number of investigations to determine the possibilities and limitations of the CO₂ system. Results of some exploratory studies are presented in the section on carbon dioxide.

Investigations of this nature are necessarily slow, being heavily dependent on equipment development, process awareness, preliminary hazard studies, safety preparations, and scheduled exploratory time. The research described here, with both methyl borate and methyl formate, was preliminary in nature and designed to determine the feasibility of these treatments. The description of the research conducted with carbon dioxide provides a highlighted summary of detailed studies in the area of cure acceleration of cement-bonded wood composites.

METHYL BORATE

On a weight basis, the equation



shows that when 1 g of MeB reacts with 0.5 g of water, approximately 0.6 g of boric acid and 0.9 g of methanol are formed. Boric acid has been shown to impart fungal resistance to wood composites at a loading of 0.5%, oven-dry (OD) wood basis (Williams and Parresol 1991). The loading level must be increased to approximately 7.5% in order for the boric acid to serve as a fire retardant and to insure that boards meet the class I flame-spread standards of ASTM E-84 (LeVan and Tran 1990; ASTM 1991a). If all the water in a wood-composite mat were available for this reaction, then a mat moisture content of approximately 6.2% would be necessary to provide a loading of 7.5% boric acid. The concern in this exploration was to introduce the gas in such a manner that it had time to react with the water and be deposited on the wood without being carried through the mat and lost as a vapor. By using steam injection pressing (SIP), we hoped to provide the reaction with ample heat and moisture resulting in rapid formation of the boric

acid. Exploratory trials indicated that any boric acid formed by the interaction of steam and MeB was not condensed as crystals until the gas mixture cooled.

MeB Procedure

At atmospheric pressures, methyl borate becomes a gas at 69°C. A laboratory-scale prototype gas generator was developed from a 304 mm tall by 150 mm diameter steel canister that had been provided with heating coils (Figure 1). Liquid MeB was preloaded into the canister and heated to 88°C. The vaporized liquid was then forced into a manifold, which fed a perforated platen, by pressurizing the canister with nitrogen at a pressure of 550 kPa. The platens (Geimer 1983), which were attached to a computer-controlled press, were heated to 177°C. The edges of the boards were exposed to atmosphere during pressing; i.e., the press was not equipped with a sealed chamber. Trials were made with various combinations and sequences of gas and steam introduction. Steam pressures were limited to either 440 or 550 kPa. Sixty-two experimental boards were fabricated, including 15 conventional and 14 SIP control boards made without gas injection. The 350 by 350 by 13 mm boards were made from 38 by 12.7 by 0.63 mm disk-cut aspen flakes and pressed to a specific gravity (SG) of 0.70 (OD weight basis). Boards were made using either 5% phenolic or 3% isocyanate resin (OD wood basis) as the adhesive. Target mat moisture content was 8% (OD mat basis).

Gas flow was not monitored. The generator was precharged with 150 g of liquid MeB in all cases, which was sufficient to provide 9.5% boric acid retention at 100% efficiency. Preliminary trials indicated that the gas could be transferred to the platens in a very short time when exhausting to the atmosphere. The manifold pressures were, of course, dependent on the density of the mat, as dictated by the nature of the pressing schedule. Checks made on the canister following pressing failed to show any unused MeB. One shortcoming of the trials was the lack of control on gas flow. Although the press schedules varied the length and mode of the gas introduction

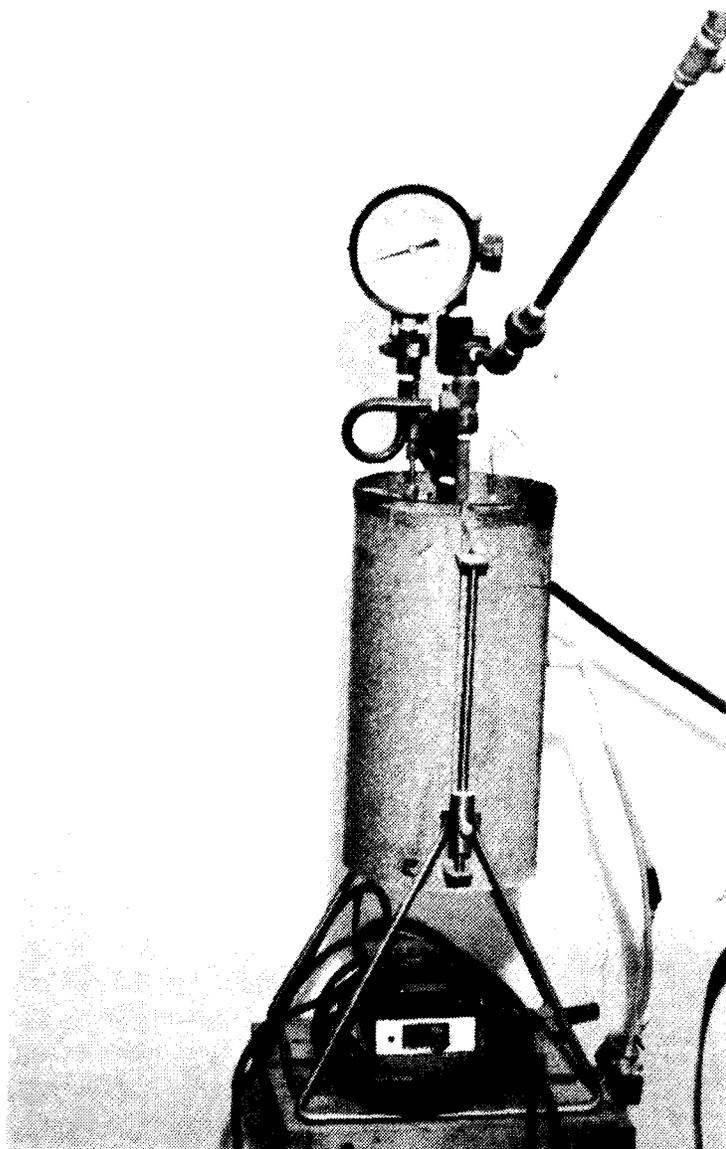


Figure 1.—*Canister used to generate methyl borate gas*

sequences (gas alone or gas and steam together), there was no way of assuring that gas flow was linear throughout these periods.

Following pressing, the boards were weighed and measured for thickness. Three 50 mm diameter plugs were then cut from each hot panel, measured for specific gravity, and tested for shear properties. These shear values provided a major influence in selecting press schedules for ensuing boards. The boards were then equilibrated at 27°C, 65% relative humidity. Eight samples from each board were tested for compression shear strength using the Minnesota

shear tester as outlined in ASTM D-1037 sections 143P152 (ASTM 1993). Three samples from each board were tested for bending modulus of elasticity (MOE) and modulus of rupture (MOR) according to ASTM D-1037 procedures (ASTM 1993). Heat release rate (HRR), a test to determine fire resistance, was performed on a 125 by 125 mm specimen from each board. This test is a modified form (Tran 1990) of the Ohio State University test as outlined in ASTM E 906-83 (ASTM 1991b). A small sample from each board was analyzed for boron content by means of inductively coupled plasma emission spectrometry.

MeB Results and Discussion

Pressing information and test data for a selected number of boards are given in Table 1. Shear and internal bond (IB) properties of the phenolic-bonded SIP control boards (type B) were only 30 and 60%, respectively, of those properties measured for conventionally pressed control boards (type A). Bending properties were also lower. This reduction in strength resulting from SIP was shown previously in phenolic-bonded boards made with aspen disk-cut flakes (Geimer 1991).

Introduction of MeB further reduced the IB and MOR properties. Press schedules calling for the introduction of steam prior to the introduction of MeB gas (type C boards) produced results similar to those programs that scheduled the gas to be simultaneously introduced with the steam (type D boards). Introduction of the MeB prior to introduction of the steam (type E boards) had the most detrimental effect on strength properties of phenolic-bonded boards. The most significant result observed with this series of phenolic-bonded boards was the fact that some boric acid was deposited in the board as indicated by reductions in HRR. Inductively coupled plasma emission spectrometry of type E boards indicated that the maximum boric acid deposition was 1.57% based on OD wood.

Mechanical properties of the isocyanate-bonded SIP controls (type G) were lower than those of the isocyanate-bonded conventional controls (type F). The IB was lower by 25% and shear and MOR properties were lower by 15%. This is not unreasonable considering the 75% reduction in press time. Not foreseen was the adverse effect of the MeB gas on mechanical properties. The IB and shear values of isocyanate-bonded boards injected with gas prior to steaming were as low as or below those measured for the phenolic-bonded gas-injected boards. Boards pressed with a seven second simultaneous steam and gas injection period (type I boards) suffered a loss in bending properties in addition to a reduction in IB and shear strength.

A series of boards were made using a three second MeB injection period followed by simultaneous MeB and steam injection prior to closure. By decreasing press closure rate, the period of simultaneous MeB and steam injection could be lengthened. Boards made with the first two press schedules (J and K) using five and eight seconds of simultaneous gas and steam were either too poorly bonded to test or had extremely poor mechanical properties. This was attributed to an insufficient supply of heat to cure the resin. Maximum board temperatures were low and were reached rather late in the press cycle. Board strength and fire resistance improved slightly as the steam and gas period lengthened (type L through O boards). The best (lowest) HRR level attained was 83.6 kW/m². This represents a 43% reduction of HRR and denotes a Class 2 fire rating. An HRR value of approximately 40 is needed to achieve a Class 1 rating. Boric acid levels of 1.8 to 2.4% were indicated for those boards having HRR values in the 80s and 90s. Three boards (type P) were made using a press program that incorporated a long (eight second) steam period prior to closure and delayed entry of the gas until after press closure. These boards showed lower HRR values, but retained excellent board properties.

The results of these trials indicate that fire-retarding properties can be modified by using a gas injection system to deposit chemicals into the mat during pressing. Of equal significance is the observation that modification of the environment surrounding the adhesive can significantly affect its bonding capabilities. Unfortunately, in the case of MeB, the environmental change was detrimental to board properties.

METHYL FORMATE

BETASET 9512 is an ester, curable, alkaline, phenolic resole resin, characterized by a phenolic resin content of 500A, a viscosity of 150 cps, and a pH of 12.1. The resin was formulated for the foundry industry to provide extremely fast cures of sand cores at room temperature. Unlike other organic cold-box systems, the injected MeF gas does not act strictly as a catalyst, but

Table 1.—Composition of methyl borate boards^a

Board Type	Press Schedule Segment ^b	Board Replications	Press Time (seconds)	Press Temp (°C)	Maximum Temp/Time ^c (°C/seconds)	Gas (g)	Gas Temp (°C)	Steam Press (kPa)	SG ^d	Plug Shear (MPa)	IB (MPa)	MOR (MPa)	MOE (MPa)	HRR (kW/m ²)	Boric Acid ^e (%)
Phenol Formaldehyde															
A	Conventional control	8	600	190	121/600	--	--	--	0.754	3.06	0.789	38.75	5,896	142.93	0.00
B	S2-CCL-S3	6	420	190	140/38	--	--	550	0.752	1.10	0.474	31.73	4,952	139.23	--
C	S4-G7-CL	2	360	190	130/26	150	65-90	550-410	0.775	1.18	0.448	27.40	4,950	112.3	1.02
D	S/G7-CL	2	360	190	126/31	150	88	550-410	0.8	1.67	0.363	27.80	5,000	116.10	--
E	G3-S/G4-CL-S2	6	360	190	125/360	150	88	410	0.788	1.46	0.178	23.70	5,541	114.00	1.57
Isocyanate															
F	Conventional control	7	660	177	148/605	--	--	--	0.729	3.56	0.934	45.16	5,819	148.89	--
G	S2-CL-S3	8	160	177-190	150/32	--	--	720/550	0.738	3.06	0.714	37.69	4,916	139.82	--
H	S3-S/G8-CL-S4	5	240	190	143/37	150	65	410	0.754	0.22	0.381	35.30	5,520	117.7	2.01
I	S/G7-CL	4	220	190	130/30	150	88	410	0.752	1.92	0.142	17.40	3,735	114.4	1.14
J	G3-S/G5-CL-S2	4	220	190	110/211	150	88	410	--	--	--	--	--	95.8	--
K	G3-S/G8-CL-S4	1	240	190	110/111	150	88	410	0.744	--	0.02	16.60	5,325	124	1.19
L	G3-S/G10-CL-S4	1	240	190	151/38	150	88	410	0.739	1.53	0.115	32.80	5,467	96.2	2.28
M	G3-S/G11-CL-S4	1	240	190	124/30	150	88	410	0.768	1.05	0.132	32.10	4,951	115.7	--
N	G3-S/G15-CL-S4	1	240	190	149/32	150	88	410	0.744	1.57	0.106	29.30	5,323	83.6	1.84
O	G3-S/G15-CL-S4	3	350	190	147/60	150	88	410	0.789	1.62	0.166	31.40	5,558	95.4	1.94
P	S8-CL-V3-G80	3	240	190	153/28	150	65	410	0.752	4.04	0.771	39.10	5,131	114.8	--

^a IB is internal bond; MOR, modulus of rupture; MOE, modulus of elasticity; HRR, heat release rate.

^b Sequence and length of pertinent press segments. S2 is steam for 2 s; G7, gas for 7s; S/G5, simultaneous steam and gas for 5 s; V3, vent for 3 s; CL, press closure.

^c First value is maximum core temperature. Second value is time to reach maximum temperature.

^d Obtained from IB specimens.

^e Boron values as measured by inductively coupled plasma spectrometry in parts per million; expressed as boric acid equivalent in percent of oven-dry wood.

becomes part of the bond, releasing formic acid and methanol in the process.

Using the small gas generator described in the previous section, preliminary studies were conducted to determine the feasibility of adapting the Betaset process to wood composites. Information acquired from the construction of sixteen panels prompted the establishment of a cooperative effort with Acme/Borden, the company that supplies resin and services to the foundry business, and with their parent company, Borden Inc. Acme/Borden provided a portable gas generator, the methyl formate, a special phenolic resin formulated for the Betaset process, and counsel on the normal use of the system. Borden supplied the necessary expertise link between foundry and wood-composite technology. The two day investigation resulted in the production of eighteen boards.

MeF Procedure

Eighteen 762 by 660 by 13 mm boards were fabricated and pressed to a target SG of 0.64, OD weight basis. With the exception of two boards constructed with 60 by 12 by 0.76 mm aspen disk-cut flakes, all boards were constructed using 0.76 mm thick aspen ring-cut flakes obtained from 20 mm chips. The furnish was screened to eliminate the fines passing a 1.6 mm screen and sprayed with Betaset 9512 resin to a target retention of 5% resin solids, OD wood basis. Moisture content into the press was targeted at 8.0%. The MeF gas generator (Figure 2) was calibrated to supply a 60/40% mixture of MeF and air, respectively, at a delivery rate of either 6.5 or 11.5 g of MeF per second. The boards were placed in the press described earlier and pressed to a target thickness of 13 mm and a SG of 0.64, OD basis. A press perimeter sealing chamber was not used. Press variables included platen temperature, total press time, gas duration, gas amount, gas injection sequence, steam duration, steam flow, and steam injection sequence. Steam pressure was limited to a maximum of 400 kPa. Upon removal from the press, the boards were weighed and measured for thickness. Three 50 mm diameter plugs were then cut from each hot panel,

measured for specific gravity and tested for shear strength. The shear values were a principal factor in selecting press schedules for ensuing boards. Following equilibration at 27°C, 65% relative humidity, eight samples from each board were tested for Minnesota shear properties and three samples from each board were tested for bending MOE and MOR according to ASTM D-1037 (1993) procedures.

MeF Results and Discussion

The gas generator used in this experiment is capable of warming the air and MeF mixture to between 35°C and 55°C. This temperature is sufficient to vaporize the MeF, which has a boiling point of 32°C at atmospheric pressure, and to keep it in the gaseous form when it contacts the cooler sand. A notable characteristic of the foundry operation is the relatively low pressure (below 40 kPa) used to deliver the gas. Low pressures permit the MeF to be vaporized at low operating temperatures and are possible because the gas easily penetrates the permeable sand castings. The individual sand grains, unlike wood particles, are not compressed during mold formation and permeability remains the same throughout the entire gassing procedure. Temperatures are much higher in the wood composite pressing process because of the need to plasticize the wood composite mat and enhance the heat transfer rate. A gas generator designed for use with wood composites would provide higher operating temperatures and pressures.

Much of the experimental work was directed at discovering press schedules that permitted effective gas distribution. Selected boards are described in Table 2; the relative importance of the major pressing variables is indicated. For comparison, test values are given for a board (type A) pressed in the conventional fashion for 10 minutes. Results from several boards made without steam injection, but exposed to MeF gas following press closure (type B boards) indicated that although the gas was affecting resin cure rates, additional heat would be necessary if press times were to be reduced substantially.

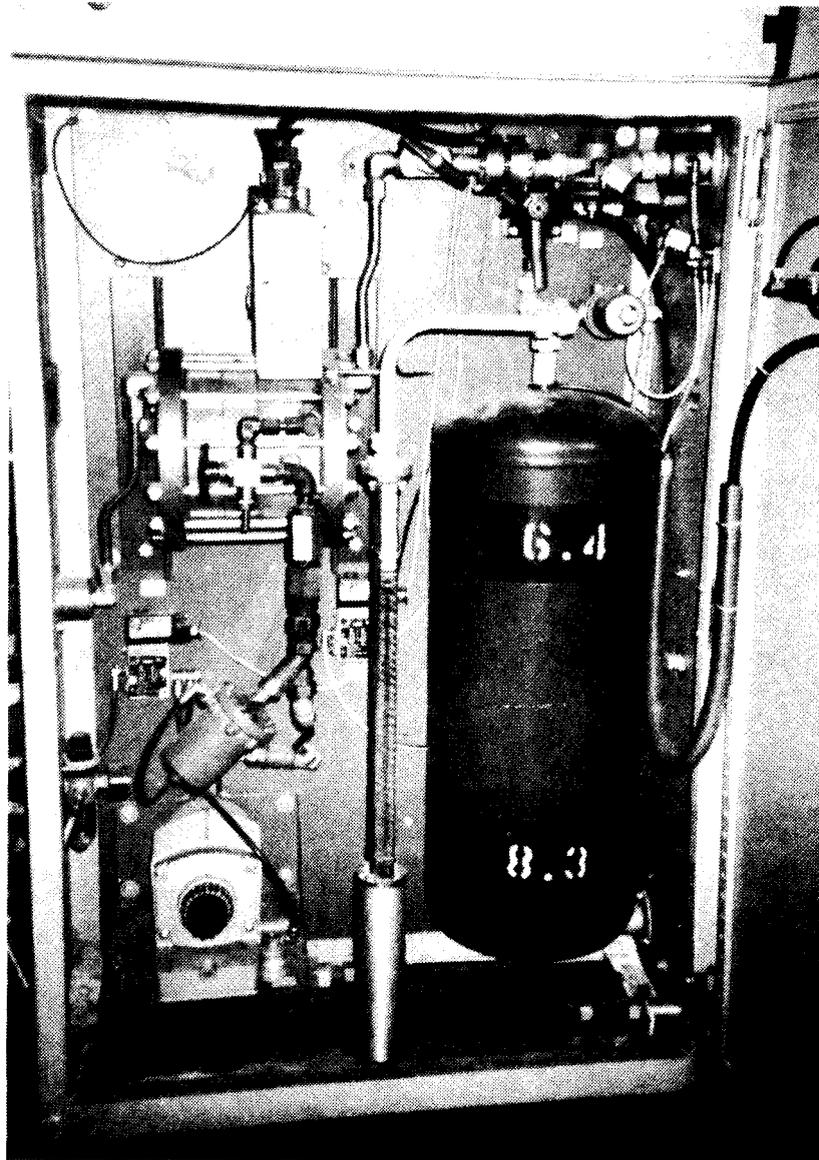


Figure 2.—*Methyl formate gas generator*

A question of major concern was whether introduction of gas and/or steam prior to press closure would cause precure problems. After trying several press schedules, including steam injection followed by gas injection (type C), it was concluded that preclosure injection of MeF did not adversely affect bond formation. Consequently, gas was introduced first, at a mat SG of 0.25. The press was then closed further and a 4 second steaming period was initiated when the mat reached a SG of 0.38. Finally, the press was closed to target thickness. This schedule (type D)

permitted the manufacture of a board in 105 seconds, having a plug shear value of 0.7 MPa. When total press time was increased to 195 seconds (type F boards), the average plug shear strength increased to 1.5 MPa or 58% of that measured for the conventionally pressed board.

Comparison of these out-of-press plug shear values with the value obtained for the drier conventional board must be tempered by the fact that strength will increase as the board dries out. The Minnesota Shear strength of the 195 second gas-injected board (type F) was 87% of that of

Table 2.—Composition of methyl formate boards

Board Type ^a	Press Schedule Segments ^b	Press Time (seconds)	Press Temp (°C)	Maximum Temp/Time ^c (°C/seconds)	Gas Time ^d	Gas Use (g)	Board SG	Plug Shear (MPa)	Minnesota Shear (MPa)	Bending MOR (MPa)	Bending MOE (MPa)
A	Conventional control	600+	150	--	0	0	0.690	2.6	4.9	24	3,377
B	CL-GV40	180	150	93/180	30	195	0.582	0.7	1.9	9	1,650
C	S6-V 1-GV10-CL-GV35	165	150	104/165	10	116	0.600	--	1.2	9	1,841
D	GV12-S4-CL	105	150	98/105	10	116	0.608	0.7	2.3	8	1,591
E	GV12-S4-CL	135	150	114/39	10	116	0.629	0.9	2.4	11	1,826
F	GV12-S4-CL	195	150	117/39	10	116	0.676	1.5	4.2	14	2,226
G	S4-CL	185	150	111/32	0	0	--	--	--	--	--
H	GV4-S4-CL	185	150	113/32	5	58	0.659	1.2	3.0	15	2,443
I	GV17-S4-CL	202	150	111/49	20	232	0.653	0.7	2.3	11	1,890
J	GV4-S4-CL	185	205	119/33	5	58	0.687	1.3	3.5	14	2,187
K	GV15-S7-CL-S5	140	150	141/153	10	116	0.591	1.3	4.0	16	2,761
L	GV25-SV7-CL-S60	148	150	151/112	20	230	0.738	1.1	3.2	13	2,585
M	GV12-S5-CLL-S30	191	205	110/53	10	116	0.721	1.6	4.4	16	2,571
N	GV12-S5-CL-S30	195	205	142/45	10	116	0.648	--	--	--	--

^a All board types fabricated with ring-cut flakes except type N (fabricated with disk-cut flakes). Each board type refers to a single board.

^b Press segment descriptors are the same as in Table 1. In addition, GV12 is gas top and vent bottom platen for 12 seconds; SV7 is steam top and vent bottom platen for 7 seconds.

^c First value is maximum temperature in core. Second value is time to reach maximum temperature.

^d Gas flow rate was 11.6 g/second except for board type B, which was 6.5 g/second.

the 600 second conventional board (type A) when both samples were equilibrated to the same moisture content. Likewise, the difference in the vertical density gradients of SIP and conventional pressed boards will affect their mechanical properties. Reduced face layer SG, a characteristic of most steam-injected boards, is conducive to lower bending properties.

A series of boards (type G through I) was produced to determine the effect of the gas usage. Delamination of the board made with a 4 second steam period, but no MeF gas injection (type G) verified that MeF was essential to the reduction of press time. A board (type H) with a plug shear value of 1.2 MPa was made in 185 seconds using only 58 g of MeF. In this case, the ratio of MeF to resin solids was 0.3:1 as compared to the ratio of 0.4:1 often used in curing sand castings. Increasing the platen temperature from 150°C to 205°C (type J board) enhanced shear values, but did not improve final bending properties. Lengthening the duration of steaming time both before and after closing (type L and M boards) increased board temperatures and further improved properties.

Boards made using aspen disk-cut flakes (type N board) in place of ring-cut flakes delaminated upon release of press pressure. Although the MeF gas has been shown to accelerate resin cure, it still remains to be determined if this system can be adapted to the fabrication of a structural board using large flakes. It is somewhat disappointing that a system that permits the cure of sand castings in less than 10 seconds does not produce the same fast response when used to manufacture wood composites. However, it is entirely possible that the extent of cure necessary to hold the sand in place is much less than that needed to oppose the springback forces inherent in a compressed wood composite. Further investigations along these lines would benefit from the use of a gas generator capable of producing vapor pressures high enough to permit simultaneous introduction of MeF and steam.

CARBON DIOXIDE

When unhydrated cement and water are exposed to carbon dioxide (CO₂), the stiffness of the mixture rapidly increases. This early “set” is attributed to the reaction of CO₂ with calcium hydroxide (Ca(OH)₂) to form calcium carbonate (CaCO₃) and water.



By replacing a small portion (5%) of the cement with Ca(OH)₂, the reaction rate is increased. Press clamping times of 8 to 24 hours, necessary to prevent springback (out of press thickness swell) and to permit handling of conventionally manufactured cement-bonded wood composites, are reduced to less than 4 minutes when CO₂ is injected into the mat. Mat temperatures of over 100°C are reached during the pressing stage as a result of the exothermic nature of the reaction. Upon removal from the press, the boards have attained between 25 to 75% of their final bending stiffness and are set aside for another 28 days to attain near maximum strength.

This gas pressing technique has the further advantage of reducing normal hydration inhibitions caused by those wood species that contain an abundance of sugars and hemicellulose. Research has shown that bending properties of high density (SG greater than 1.0) CO₂-injected boards are increased by as much as 100% over those attained with conventional pressing methods (Ceimer et al. 1992). In addition, the treatment improves durability of the boards and reduces efflorescence (blushing) caused by migration of Ca(OH)₂ to the surface (Souza et al. 1994).

Research Parameters

Uniform gas distribution, critical to consistent quality, is easier to obtain in low density boards (0.50 SG) than in high density boards. This attribute prompted the cooperative research between the Forest Products Research and Development Institute (FPRDI), College Laguna, Philippines, which had developed 50 mm thick, low density, cement-bonded wood excelsior boards for use in the construction of low cost housing (Pablo et al. 1990). In application, both

faces of the boards are coated with a mortar mix after their erection as a structural wall. On the supposition that a board that incorporated smooth surface layers might eliminate or reduce the need for on-site finishing, the study was extended to investigate three-layer configurations. The following discussion relates to an extensive preliminary investigation (encompassing the fabrication of more than 150 boards) to determine the general parameters of manufacturing three-layer cement-bonded wood composite boards with the CO₂ gas injection System. Among the variables studied were wood species; excelsior dimensions; flake, fiber, particle, and splinter geometry; water to cement ratios (water: C); wood to cement ratios (wood: C); board SG; face to core proportions; and a variety of blending and pressing techniques.

Observations

A major difference in the mat characteristics of an inorganic-bonded board as compared to a

thermosetting resin-bonded board is the amount of binder used. Whereas flakeboard and particleboard contain between 2 and 10% resin (OD wood weight basis), the amount of Portland cement (SG of 3) used ranges between 130 and 400%. This implies that low-density boards must be constructed with an excelsior-like particle that permits numerous relatively large void spaces between the particles (Figure 3). The fundamental composition of low-density boards is similar to that of fiberglass ceiling tile; in both cases, the intermeshing fibers are coated with a stiffening agent,

Blending techniques used for high-density cement-bonded particleboards are not necessarily suitable for the low-density panels made from excelsior (Shigekura 1988). Compared to the 0.2-0.4 water: C ratio normally used in high-density particleboards, a water:C of 0.5- 1.0 is necessary to obtain uniform cement coverage of excelsior strands (Figure 4). The higher water:C

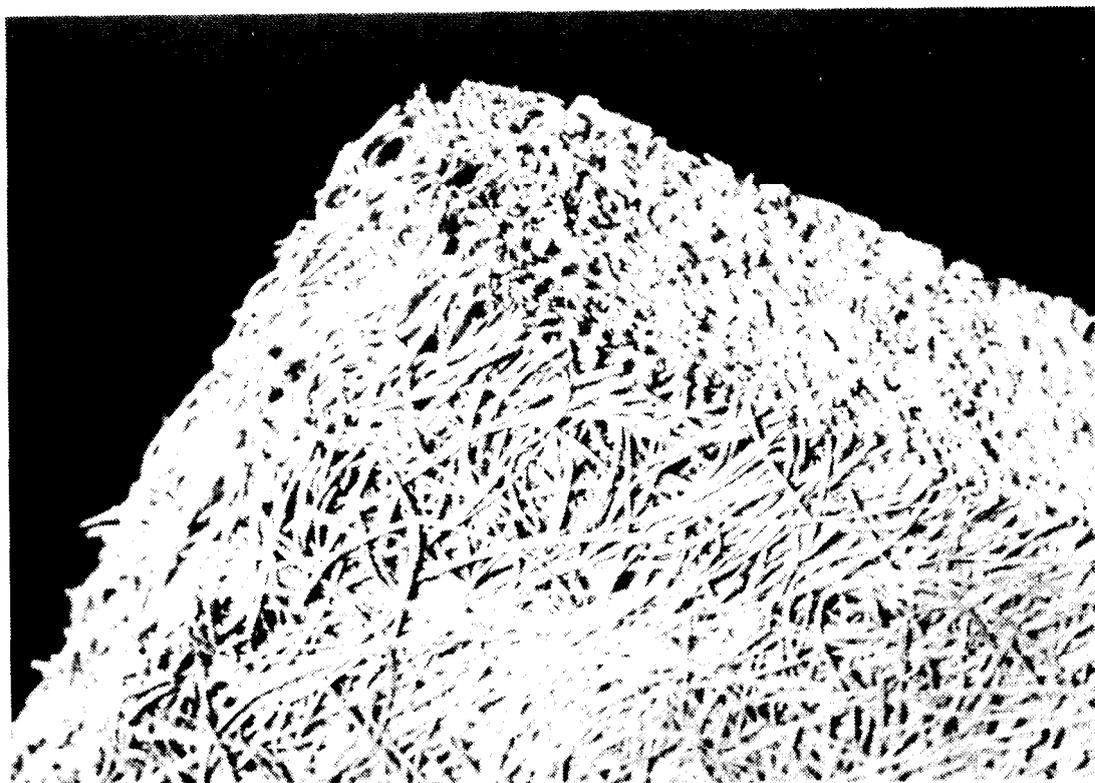


Figure 3.—Low density (0.45 specific gravity), cement-bonded board made with type 732 excelsior strands

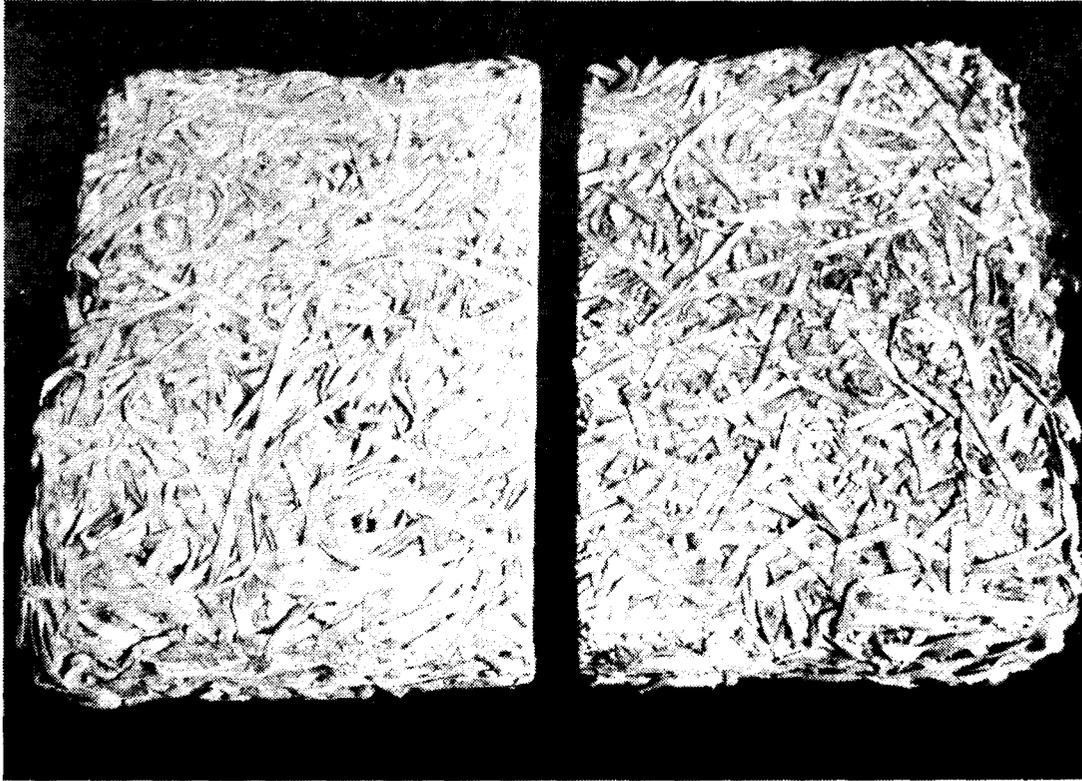


Figure 4.—*Addition of cement and water improves distribution of cement. Board on right, which shows poor cement distribution, was fabricated with SG of 0.45, wood:C ratio of 0.75, and water:C ratio of 0.6. Board on left, which shows good cement distribution, was fabricated with SG of 0.45, wood:C ratio of 0.65, and water:C ratio of 0.8. Both boards were made with type 604 excelsior*

ratio also ensures that adequate water is supplied for the hydration process. During the pressing of high-density boards, water is forced from the cell lumens as the wood is crushed. This water is not available for cement hydration in a low-density board because much less wood compaction occurs during pressing. On the other hand, very high water:C ratios (above 1.0) create an unbalanced board as the cement slurry migrates to the bottom of the mat prior to completion of the hydration process (Figure 5).

Spraying a slurry of water, cement, and $\text{Ca}(\text{OH})_2$ provided good coverage, but limited the minimum amount of water used. The slurry spraying technique was not easily adaptable to changes in particle configuration. The blending method that proved to be most adaptable to laboratory exploration studies involved spraying a solution of $\text{Ca}(\text{OH})_2$ and water onto the flakes

and then slowly adding cement that had been sifted through a fine screen. A variety of excelsior Portland cement blending techniques were tried using a single shaft, 0.85 m^3 , pin-type blender to tumble the excelsior. Changes in the blender pin configuration were necessitated by changes in excelsior geometry.

Two sizes of aspen excelsior were used for the study. The narrow strand (732) had a length of 457 mm, thickness of 0.5 mm, and width of 0.8 mm. The type 732 strand is similar to that used in boards produced by the two existing U.S. commercial inorganic-bonded-excelsior plants. The wide strand (604) had a length of 457 mm, thickness of 0.46 mm, and width of 6.4 mm, closely resembling the strand used by FPRDI. The type 604 excelsior was used to manufacture the majority of the homogeneous boards and the core layer of the three-layer boards. The type 604

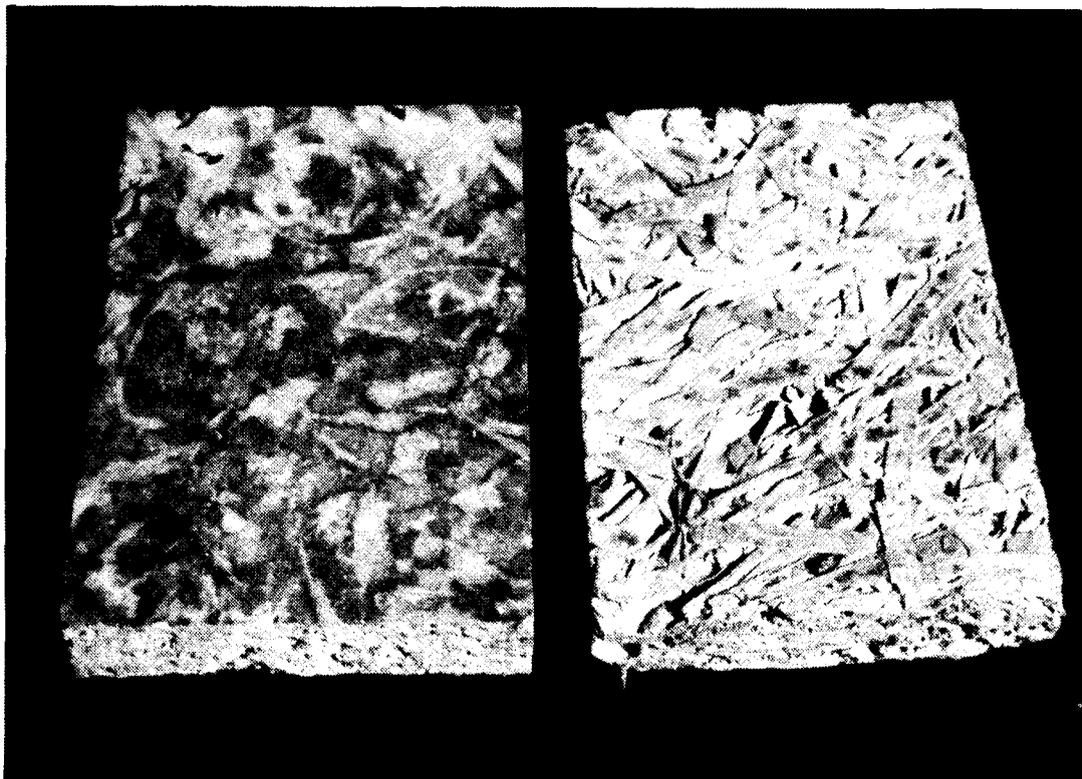


Figure 5.—Surfaces of cement-bonded excelsior board showing migration of cement slurry, from top face (right) to bottom face (left)

strands had a tendency to ball up in the pin blender, making it difficult to achieve uniform blending and forming. The type 732 (0.5-mm-thick) excelsior also tended to ball up, but it permitted a more uniform coverage on the individual strands that were coated with cement. Ultimately, the strand length was reduced to approximately 150 mm.

Uniform gas penetration was obtained easily with both excelsior types. With an open-sided press, efficiency is reduced as a result of gas loss through the board edges to the surrounding atmosphere. In the majority of our studies, a sealing frame was attached to the upper platen to create a closed chamber at press closure. In most exploratory press programs, press closure was followed by a short period during which CO₂ gas was injected into the top face while a vacuum was pulled on the bottom face. This eliminated entrapped air, which could block the CO₂ reaction. The chamber was then maintained at se-

lected levels of CO₂ gas pressure between 400 and 700 kPa for 80 to 210 s. Then, the gas remaining in the manifold and platens was allowed to react with the cement for designated periods of 90 to 300 seconds before being vented to the atmosphere. Varying the (1) gas pressure, (2) duration of gas application, and (3) time allowed for depletion of the gas in the system permitted the trade-off between minimum cure time and gas usage.

The high moisture needed for blending excelsior reduces the effectiveness of the CO₂ treatment (Geimer et al. 1992). Results on the relationships between initial moisture content, out-of-press stiffness, and final board strength are in preparation (Pablo and Geimer). Initial analysis of that data indicated that a water:C ratio of 0.8 permits the formation of satisfactory out-of-press board stiffness and acceptable final cured board properties. A minimum press time of 3.5 minutes was determined for the homoge-

neous type 604 excelsior boards. Further reduction of time resulted in the appearance of blisters and soft spots when boards were removed from the press. As mentioned previously, the out-of-press or "green" stiffness was between 25 and 75% of the final board stiffness. Because initial compaction forces are relatively small, the green stiffness requirements in a commercial operation will be dictated by the board handling equipment rather than the residual springback of the board.

Weight gain during pressing, a measure of gas consumption, averaged approximately 10% of the weight of the unhydrated cement and $\text{Ca}(\text{OH})_2$ mixture. This indicated that reactions, beyond that afforded by the 5% addition of $\text{Ca}(\text{OH})_2$, were occurring. Since only 0.594 parts of CO_2 are required for every one part of $\text{Ca}(\text{OH})_2$, a weight gain of approximately 3% is sufficient to consume the added $\text{Ca}(\text{OH})_2$. Actual gas usage, monitored with a flow meter during only the latter stages of the study, varied between 25 and 45% of the OD weight of the cement, indicating an efficiency of approximately 30%. Efficiency values (weight-gain: CO_2 -used ratio) must be tempered with adjustments made for moisture evaporation during the press cycle, moisture flash-off occurring after the press is opened, and excess gas vented to the atmosphere.

A variety of furnishes were examined as candidates for surface layers of a three-layer construction. These included thin strands of excelsior, virgin and recycled newsprint fiber, fine and coarse particles, planer shavings, coir (coco-nut husk fiber), and kenaf. The ideal surface is a thin, smooth layer having a high SG. However, compaction of the wood in the face layer is limited by the relatively low pressures needed to compress the entire mat. Face layer SG is therefore highly dependent on the amount of cement used in the face. When using fibers, very little cohesiveness of the hydrated mixture was obtained when working within the ranges of 0.35-0.45 wood:C and 0.6-0.8 water:C. Decreasing wood or increasing water beyond these limits in an attempt to develop a denser layer led to the creation of craters and voids in the top surface

caused by migration or sifting of the surface material into the core voids.

Material made from both coarse and fine particles produced a relatively smooth surface at workable ratios of water:C and wood:C. However, under these conditions, the top layer exhibited considerable problems with core show-through (Figure 6). Bulky materials like planer shavings and thin excelsior strands were able to span the core voids and worked quite well as face material (Figure 7). Acceptable cohesiveness between planer shavings pressed to 0.45 SG was obtained with a water:C of 0.8 and a wood:C of 0.35 or less.

In summary, with proper selection of particle geometry and adjustments to blending and forming techniques, the construction of a three-layer low-density cement-bonded wood composite is feasible. No major problems were encountered with the CO_2 gas injection process and acceptable boards were produced in less than 4 minutes.

CONCLUSIONS

The feasibility of using steam injection perforated platens as a delivery system for other gases has been investigated. The exploratory studies described in this report demonstrated the ability to deposit property-enhancing chemicals, modify internal mat environments, and introduce reactive binder components during the pressing of wood composites.

Results indicated that the introduction of methyl borate (MeB) gas can improve the fire resistance of panels, but only at the expense of lowering strength properties. This observation was made with both phenolic- and isocyanate-bonded boards.

Experiments with methyl formate (MeF) indicated that this gas has the potential to decrease press times for wood composites bonded with phenolic resin. Unlike the curing of sand castings, the curing of wood composites requires additional heat to achieve adequate adhesive bond strength. Used in conjunction with steam

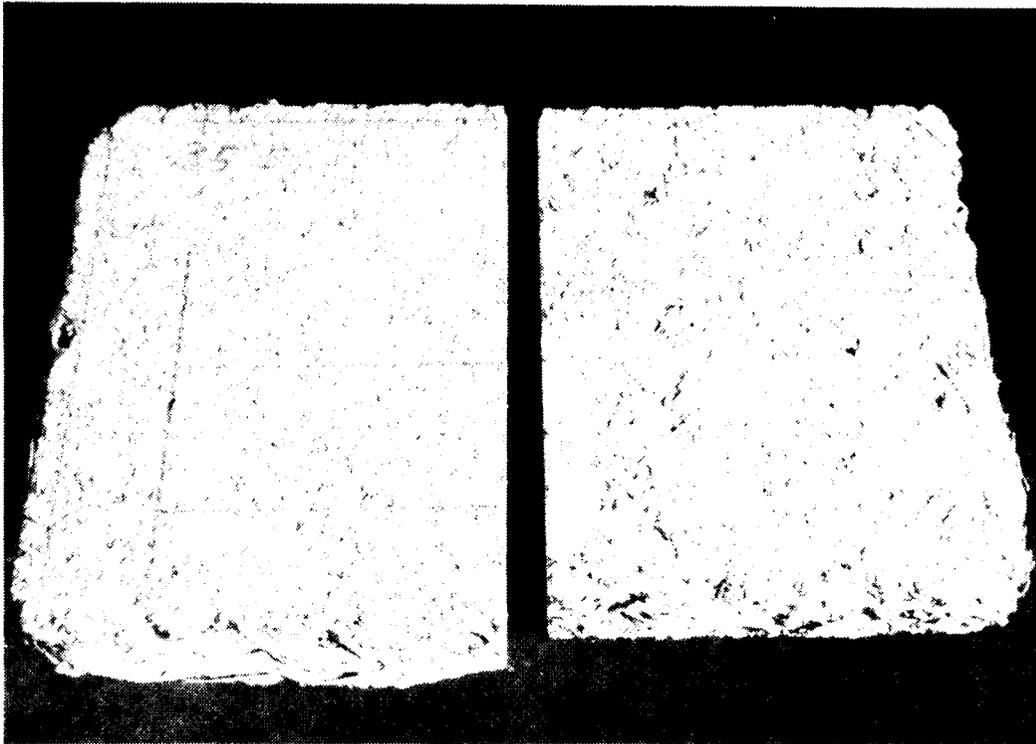


Figure 6.—Top (right) and bottom (left) surfaces of three-layer cement-bonded board showing core show-through resulting from sifting of top face particles into core

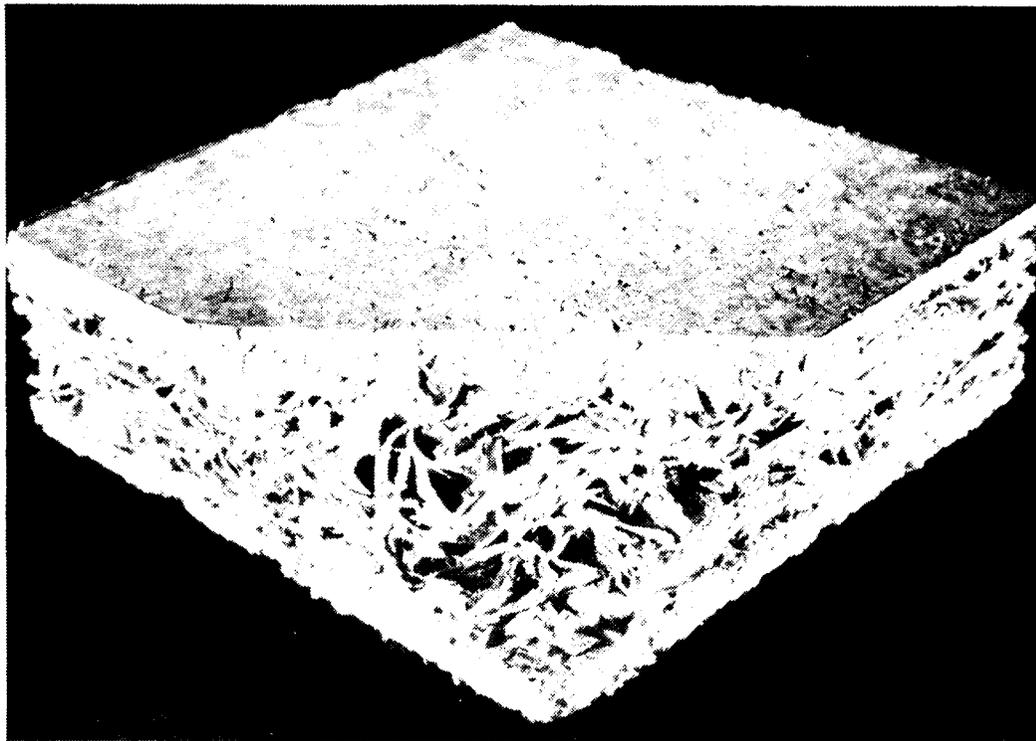


Figure 7.—Three-layer cement-bonded board fabricated with type 604 excelsior core and faces made of planer shavings

injection, the addition of MeF enabled boards to be pressed in less than three minutes.

Studies with cement-bonded boards showed that the introduction of CO₂ gas can reduce the press time of low density boards to less than four minutes. The blending equipment must be modified to achieve acceptable cement distribution on the excelsior strands. Judicious selection of materials permits the fabrication of three-layer low-density boards.

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NOTES

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