

A SEALED PRESSING SYSTEM AND ITS USE TO EXPLORE THE AMMONIA PLASTICIZATION OF NATURAL FIBER MATS

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(Received September 2003)

ABSTRACT

The effectiveness of gaseous ammonia as a softening agent for natural fiber and particle composite mats has been examined. A miniature digitally controlled sealed pressing system with provision for chemical injection and removal through permeable graphite platens was developed for this and other purposes. The pressing system is described, along with its use to examine the softening action of anhydrous ammonia on 97-mm-diameter thermomechanical pulp (TMP) mats. This was done principally in terms of treatment time (0 to 900 s) and partial pressure (0.0345 to 0.7444 MPa). Ammonia applied at moderate partial pressures (0.42 MPa) was found to rapidly penetrate and soften (within 4 s) mats compressed to a density of 525 kg m⁻³ at near-room temperatures (29°C). The results suggest that judicious injection of ammonia in sealed pressing arrangements may provide an attractive alternative to our current dependency on heat- and moisture-induced softening in conventional composite pressing methods—both in terms of production speed and providing the ability to optimally sequence rheological and adhesion mechanisms.

Keywords: Natural fiber mats, ammonia plasticization, sealed pressing, gas injection.

INTRODUCTION

Wood-based composite products have internal structures and consequent properties that are greatly influenced by the nature of the pressing processes used in their manufacture. In most current pressing methods, unsteady-state heat and moisture transfer and phase change results in spatial gradients of temperature and sorbed moisture. These gradients, in turn, lead to differential densification in the consolidation direction (Thoemen and Humphrey 2003). Such time-based establishment of structure is due to the hygro-thermo-viscoelastic nature of wood cell-

wall material (Ren 1992). Resultant cross-sectional density and associated property profiles are all-important since they affect the flexural and other important bulk properties of products.

As a result of the above mechanisms, residual stress and inter-fiber bonding differ throughout composites' microstructure as consolidation progresses. The magnitude of gradients in these parameters affects production speed and product stability. Adhesion tends to develop prematurely in the rapidly heated surface zones of panels—only to be damaged as consolidation and associated microstructural rearrangement continues

thereafter (Humphrey 1994). Further, pressing speed is limited by the rate of heat and moisture transfer to the core layers of panels. Many types of conventionally pressed panel are also subject to high irreversible swelling in service. This is due to the spring-back of compressed and deformed wood elements (relaxation of residual microstresses) and the consequential triggering of pooled cascades of adhesive bond breakage at the microstructural level. Such effects can be initiated even by modest increases in localized moisture content.

Manufacturers of conventional wood-based composite materials (mainly panels) have only limited control over the sequencing of thermodynamic and rheological mechanisms operative during pressing—even with the somewhat increased flexibility offered by continuous pressing technology (Thoemen and Humphrey 2003). A long-term goal of the research of which the present work is a part is to develop proactive approaches to affecting the sequence of mechanisms that occur within natural fiber and particle mats during their consolidation. The above-mentioned difficulties may thereby be ameliorated and control of the spatial distribution of product properties affected. One requisite of this strategy is the sequencing of rheological and adhesion mechanisms so that largely stress-relaxed microstructures may be established *prior* to the initiation of adhesion. The sealed pressing system reported here enables the necessary manipulation of thermodynamic and chemical environments and applied stress to be achieved.

The present work addresses the use of anhydrous ammonia as an agent for controlled softening in the sealed pressing system (Chowdhury 1999).

LITERATURE

Bearing in mind its known solvent properties, Schuerch (1952) suggested that ammonia should penetrate the lignin fraction of wood cell walls. Using Sitka spruce (*Picea sitchensis*) and birch (*Betula alleghaniensis*) strips, Stamm (1955) and Schuerch (1963) subsequently demonstrated that ammonia can indeed do so and that it also causes

plasticization. Bariska et al. (1969) and Davidson and Baumgardt (1970) suggested that plasticization occurs when the relative vapor pressure of the gaseous ammonia is close to saturation, and Davidson (1968) maintained that the presence of moisture (about 10–12%) in wood enhances the rate of ammonia sorption from the gas phase.

Following earlier pioneering X-ray diffraction studies of Barry et al. (1936), Kalninsh et al. (1967) reported that ammonia breaks hydrogen bonds within both amorphous and crystalline regions of the carbohydrate portion of the cell wall. The latter pointed out that ammonia molecules react strongly by an acid-base reaction with cellulose hydroxyl groups and that, though too small to dissolve the cellulose, their partial penetration into the crystallites increases the distances between the chains. This opening up of the structure does, Kalninsh et al. maintained, allow polymers to flow past one another. After removal of ammonia by diffusion and evaporation, new hydrogen bonds form at new locations and, in this way, new cross-linked structures occur. Kalninsh et al. further reported that during ammonia exposure a portion of the carbohydrate-to-lignin bonds are broken and free active groups of lignin become available; upon ammonia removal such bonds re-condense.

No report of the softening action of gaseous ammonia on natural fiber composite mats has been found in the literature. The low rates of bulk plasticization of solid wood observed in the above-reported studies may well have led researchers to the conclusion that treatment speeds would be too low to be industrially useful in composites manufacture. It was hypothesized at the outset of the present study that the long response times reported for solid wood may be the result of diffusion-limited mechanisms rather than being limited by the kinetics of the chemical reactions themselves.

In light of the above discussion, the objectives of this study were: 1) to develop a sealed pressing system with provision for load and position control and the dynamic injection and removal of reactive fluids through permeable pressing surfaces, and 2) to employ the pressing system to

evaluate the effect of gaseous ammonia treatment time and treatment vapor pressure on the softening of preformed fiber networks.

EXPERIMENTAL METHODS

The sealed pressing system

Function: The sealed pressing system (shown schematically as Fig. 1) enables the following principal functions to be performed:

- Dynamic computer-controlled application of uniaxial compressive stress and platen position
- Sequential injection and removal of vapor-phase chemical reactants through the pressing surfaces
- Sliding low-friction peripheral sealing to contain treatment chemicals
- Accurate platen and gas supply heating
- Automated supply and extraction of three or more treatment gases with closed-loop control of pressure with changing press-space volume
- Internal fluid pressure and temperature sensing of material conditions

In addition to its use in the present ammonia softening study, the system is designed to be used for the measurement of diverse mat properties including biaxial fluid permeability, thermal conductivity, and hygro-thermo-viscoelasticity—all as functions of density (level of consolidation), chemical environment, moisture content, and temperature. Such information is necessary input data for deterministic simulation models that will be used to aid in developing new composite formation methods. The pressing system may also be employed to create diverse miniature composite samples with both uniformly and nonuniformly distributed microstructures.

Mechanical design.—The upper and lower portions of the pressing system (Fig. 2) are identical. Most of their components are made from alloys of high heat diffusivity in order to maximize the rate of internal heat distribution, though stainless steel was employed for all gas-exposed surfaces. The system has the capacity to be run at temperatures ranging between ambient and about 200°C.

The main functions of the *porous pressing platen inserts* are: (i) to distribute the chemical reactants across the platen-sample interfaces, and (ii) to act as compression surfaces. The in-

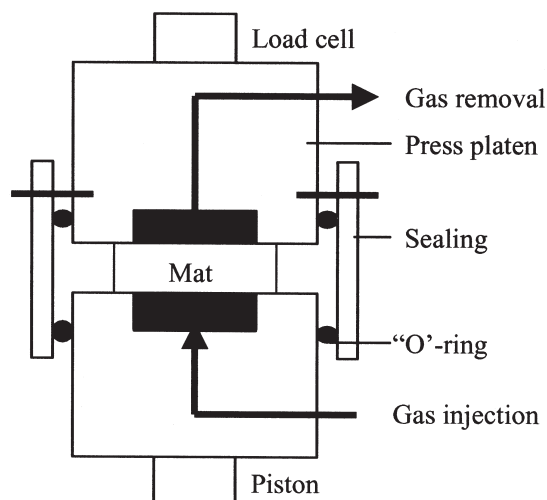


FIG. 1. Conceptual schematic of the sealed pressing approach. (Multiple gas supply and removal lines have been omitted for clarity.)

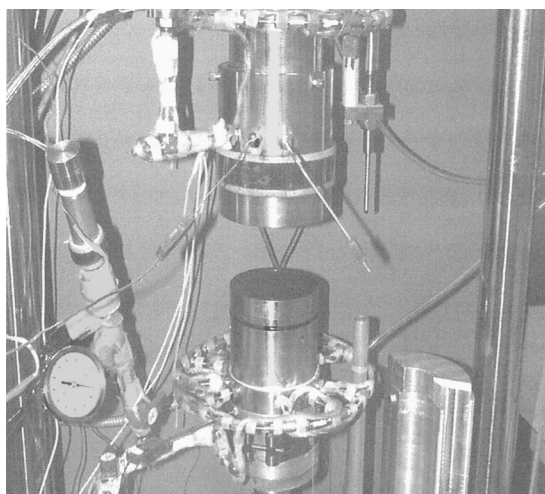


FIG. 2. The pressing heads and an overview of the system mounted on the servo-hydraulic loading frame.

serts are made of graphite of 25% porosity (on an accessible-volume basis). Material properties, including transverse permeability ($1.5 \times 10^{-12} \text{ m}^2$), compressive strength (2.5 GPa), bulk modulus (4.8 GPa), and heat stability, were considered when selecting the material for the porous plates and their thickness. The low thermal conductivity of the porous graphite ($2.2 \text{ W m}^{-1} \text{ K}^{-1}$) impedes conventional heat transfer from the pressing heads into mats (though pressing at elevated temperatures was not necessary in the present ammonia softening study). Warrens within the pressing heads were therefore designed to act as heat exchangers for gases injected through the graphite into mats. The graphite's low heat capacity ($1.8 \times 10^{-3} \text{ J kg}^{-1} \text{ }^\circ\text{C}^{-1}$) aids in affecting such transfer. The Cambridge Materials Selector protocol (Ashby et al. 1995) was employed in the design process.

The stainless steel *backing plates* that support the graphite platen inserts (Fig. 2) contain nine systematically arranged hollow sections that meet nine axial holes from the *gas supply manifold* beneath. The hollow sections in the backing plates act as reservoirs to insure effective distribution of reactant in the mats as well as aiding in the above-mentioned heat exchange. The plates provide the necessary support of the graphite to enable pressures of up to 12 MPa to be transferred without breakage or significant distortion. Further, the gas supply pathways in the backing plates are segregated from one another to provide for zoned injection and removal of chemicals in future applications of the system where in-plane as well as cross-sectional property gradients will be affected by judicious gas injection.

The stainless steel *collar* provides peripheral sealing necessary for one to control the thermodynamic and chemical environments inside the pressing chamber. A band-heater is mounted circumferentially around the collar to counter radial temperature gradients in the samples. High temperature and chemically resistant O-ring seals embedded in the pressing heads affect low-friction sealing with the collar. The static and dynamic frictional drag between the sliding (lower) seal and the collar were calibrated as a function of internal gas pressure. Corrections were then

applied to force values applied by the servo-hydraulic loading frame to mats.

The upper portion of the pressing system is connected to a 75 kN capacity load transducer with glass epoxy *thermal barrier* interposed. This is attached to the top cross-beam of the *servo-hydraulic testing machine* (SHTM), and loads may be monitored and controlled within $\pm 2 \text{ N}$. The lower press head is connected to the hydraulic piston of the SHTM via an *air-cooled isolating column*. A displacement transducer (LVDT) of 150-mm range is mounted externally to enable the position of the press to be monitored and controlled within $\pm 0.1 \text{ mm}$. The system is designed to accommodate in excess of 50 kN of force, which results in a maximum platen pressure of 8.5 MPa (for a disk of 97-mm dia).

Figure 3 schematically represents the *external gas supply and removal system*. The system consists of the following main components: (i)

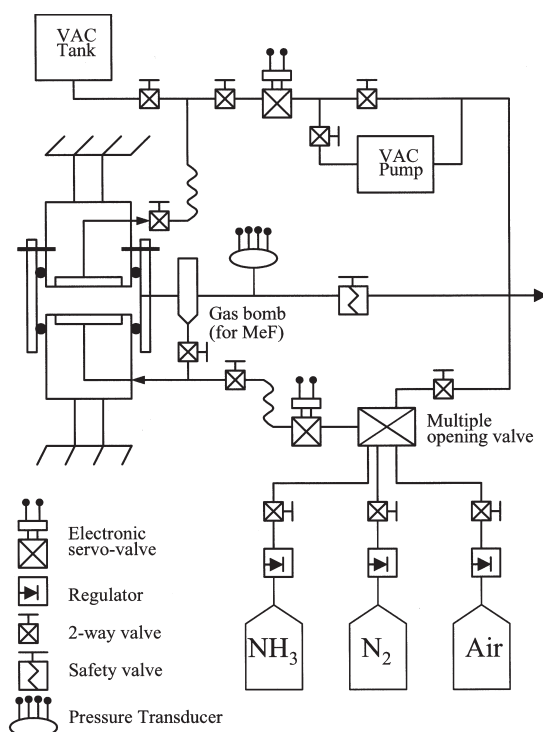


FIG. 3. A schematic of the external gas supply and removal system.

paired proportionally opening electronic servo-valves and feedback logic, which apply and remove gas from the pressing chamber based on error values from the pressure transducer located on the chamber; (ii) a range of manually controllable 2-way and 3-way valves used to configure the system for a wide range of gas sequences and flow paths; (iii) a manifold enabling up to four gas supply sources to be connected sequentially; (iv) a vacuum pump, which may be used to extract air from the pressing chamber or vacuum tank; and (iv) a vacuum tank, which may be used to rapidly extract treatment chemicals from the pressing chamber during treatment cycles. In order to avoid localized condensation of treatment chemicals, all external supply lines are temperature controlled.

Wood fibers and mat formation

Wood fibers, consisting of 60–70% Douglas-fir (*Pseudotsuga menziesii*) and 30–40% hemlock (*Tsuga heterophylla*), were generated in an industrial thermo-mechanical defibrator running at 125 °C, 0.86 MPa vapor pressure, and 50-s residence time. Fibers were extracted from the defibrator so as not to contain any adhesive or wax. Having been extracted at a moisture content (MC) of 70–75%, the fibers were dried in a moving air environment at 50 °C and 10% relative humidity (RH) and then conditioned in a standard room at 19 ± 1 °C and $60 \pm 2\%$ RH until constant weight was reached. The MC of the conditioned fibers was 10% ($\pm 0.5\%$). In order to separate agglomerations before mat forming, fibers were agitated inside a specially made cylindrical vessel with variable speed impeller.

Circular mats of 97-mm diameter were formed by air-depositing previously dried and separated fibers into a specially designed cylindrical disk former and then gently prepressed at 10 kN with a piston. A metered quantity of conditioned fibers was used for each mat. This quantity was selected so that a target density (oven-dry weight basis) of 525 kg m⁻³ was affected after compressing to a thickness of 5.00 mm in the sealed pressing system. This study

was limited to a single target density in order to establish the sealed pressing technique and verify that ammonia softening is viable.

Gaseous ammonia treatment

Ammonia pre-treatment effects on mat compaction.—Preliminary tests were conducted in which mats were treated with ammonia at 0.62 MPa and 19°C for a range of times prior to being compressed. This was done in order to explore the time necessary to usefully plasticize fully accessible (highly porous) fiber networks. Ammonia was injected into mats once the pressing system had closed to a thickness of 16 mm (corresponding to a mat density of 100 kg m⁻³). Ammonia treatment times were 0 (control), 10, 30, 60, 100, 200, 300, 450, 600, 750, and 900 s. Following treatment, the samples were pressed at 8.4 MPa pressure for 120 s in load control mode, and the thickness response was monitored. The closing and opening rate of the system was 1 mm s⁻¹. During the pressing periods, time (s), load (N), mat thickness (mm), and partial pressure of applied ammonia (MPa) were recorded digitally at a cycle frequency of 10 Hz.

In order to simplify gauging the effect of ammonia pre-treatment time on compaction, density values at one point of time during pressing (after 30 s of compaction at constant pressure) were extracted from the data. A graph of treatment time versus derived density was then plotted.

Vapor pressure effects on stress relaxation of pre-compressed mats.—Mats were compressed to a thickness of 5.00 mm (corresponding to a target density of 525 Kg/m³ on an oven-dry weight basis) at a closing rate of 1 mm s⁻¹. Ammonia gas of pre-selected vapor pressure was passed into the compressed mat 60 s after press closure. The partial pressures of ammonia employed were 0.0345, 0.0621, 0.1034, 0.1103, 0.1379, 0.1585, 0.1723, 0.2068, 0.2757, 0.3102, 0.4274, 0.5652, and 0.7444 MPa. All experiments were carried out at room temperature (19°C), and force (N), mat thickness (mm), and ammonia pressure (MPa) were recorded digitally throughout each cycle at a sampling frequency of 10 Hz.

Force data were converted to pressure acting on the mat by first compensating for the effects of the partial pressure of ammonia and frictional drag of the collar's O-ring seal, and then dividing by mat area. No correction was made for the Poisson's ratio effect during mat compaction; however, increases in diameter from beginning to end of the pressing cycles were small (typically about 1.5%), and intermediate conditions during pressing cycles could not be accurately ascertained.

Graphs of corrected counterpressure versus elapsed pressing time were plotted for each of the treatment conditions employed. The reduction in counterpressure due to injection was extracted for each data set and expressed as a percentage of the counterpressure just before ammonia injection.

RESULTS AND DISCUSSION

Ammonia pre-treatment time effects

In the preliminary tests, variously pre-treated mats were compressed under load control (a constant pressure of 8.4 MPa), and their densification with time was recorded. Two typical plots, one for a mat pre-treated with 0.42 MPa ammonia for 10 s and the other non-treated, are shown as Fig. 4. As would be expected, the mats behaved viscoelastically. Their densities tended to level out at ap-

proximately 1050 kg m^{-3} and 820 kg m^{-3} for the treated and non-treated samples respectively; corresponding densification rates after 30 s were $0.98 \text{ kg m}^{-3} \text{ s}^{-1}$ and $1.15 \text{ kg m}^{-3} \text{ s}^{-1}$.

Figure 5 shows the effect of ammonia pre-treatment time on the density attained 30 s after the platens reached the target corrected pressing pressure. Each point corresponds to one treatment cycle. Two important findings are apparent: (i) there is a big difference in density between the control (zero treatment time) without ammonia pre-treatment (820 kg m^{-3}) and all of the ammonia treated mats, and (ii) treatment time (as long as above zero) is not a very important factor in affecting ammonia softening of fibers. There is not a statistically significant difference among non-zero treatment times at the 5% exclusion level (the mean was 970 kg m^{-3}). These preliminary results suggest that small treatment times may be sufficient to produce significant and useful levels of softening in fiber networks—at least under the concentration and temperature conditions used here.

Ammonia vapor pressure effects on stress relaxation

Experiments were carried out to explore both the rate at which ammonia penetrates and softens fiber mats *after* they have been compressed to a target density and the effect of gas pressure on such behavior. Figure 6 shows a typical plot of

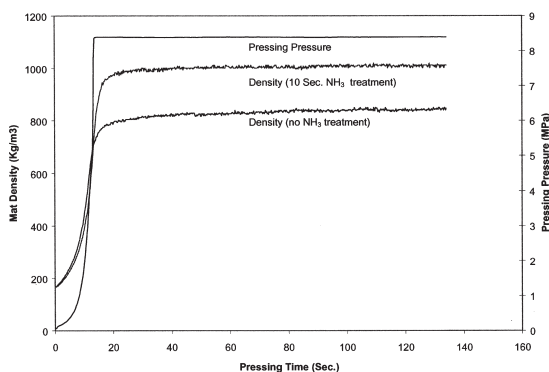


FIG. 4. Two example plots of the variation of mat density with elapsed compression time at a constant pressure of 8.2 MPa. (a) pre-treated with ammonia at 0.42 MPa for 10 s, and (b) with no exposure to ammonia.

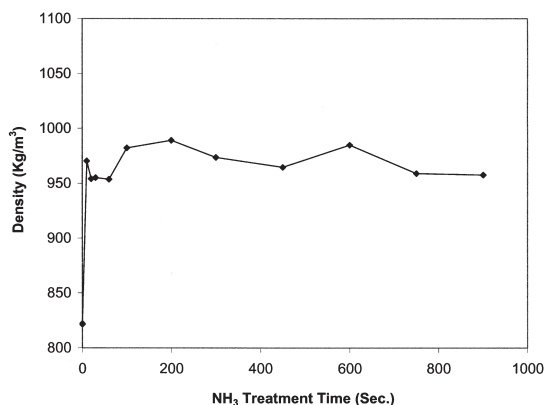


FIG. 5. Effect of ammonia pre-treatment time on the density attained after compression for 30 s at 8.4 MPa.

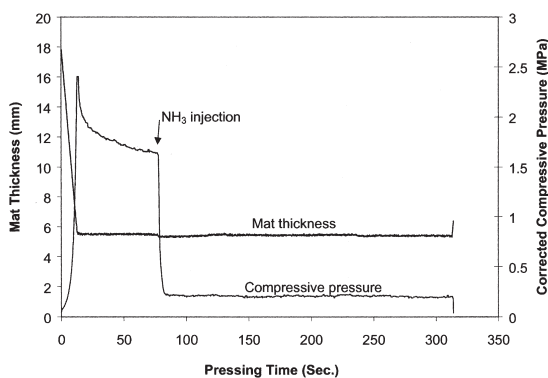


FIG. 6. Counterpressure versus time during pressing at a fixed position of 5.00 mm (525 kg m^{-3}) with ammonia of 0.42 MPa partial pressure injection affected 60 s after press closure.

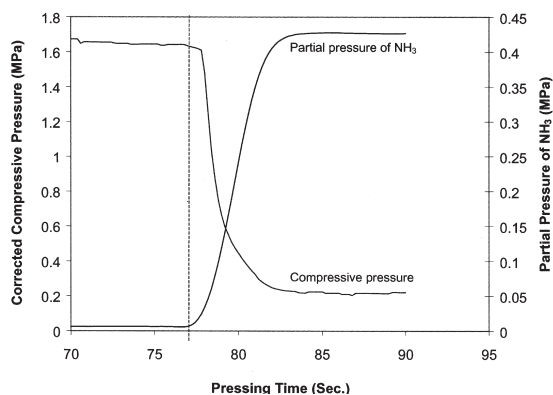


FIG. 7. Counterpressure and ammonia pressure fluctuations during ammonia application (corresponding to the data of Fig. 6).

counterpressure versus time in which ammonia vapor was injected at a pressure of 0.42 MPa 60s after press closure to a fixed position. Press closing proceeded at a rate of 1 mm sec^{-1} until the target bulk density of 525 kg m^{-3} was achieved. During this time the counterpressure rose exponentially to a peak value of 2.49 MPa. Subsequent stress relaxation initially progressed at a rapid rate (approximately 160 kPa s^{-1}) and then decayed to 5 kPa s^{-1} after 60 s. Ammonia gas was then injected and counterpressure plummeted at a rate of 1.6 MPa s^{-1} . The interaction of ammonia pressure and counterpressure during the injection period (initiated 60 s after closure) is shown as Fig. 7.

The rate of ammonia-induced decrease in counterpressure may be related to ammonia pressure increase around the sample (Fig. 7) by plotting the quotient of counterpressure divided by ammonia pressure against time elapsed after ammonia introduction. The shape of the resulting plot (Fig. 8) supports the assertion that ammonia pressure increase and counterpressure reduction closely track each other and that there is little time delay in the material's response to the application of gas. Further, it is evident that once ammonia pressure has stabilized in the system, there is very little subsequent change in counterpressure. This rapid response could occur only if penetration of ammonia to the core of the sample (complete penetration) is very rapid and

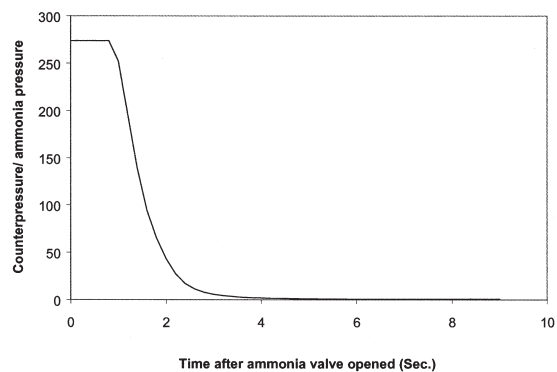


FIG. 8. Quotient of counterpressure over ammonia pressure versus elapsed time derived from the data of Fig. 7.

if there is little delay in the action of ammonia on the cell wall.

These findings appear to contradict the suggestion of Schuerch (1964) that there are time-dependent chemical transformations associated with ammonia-wood interactions. Alternatively, if slow reactions do occur, then they appear not to be significantly linked to softening. Expanded use of FTIR and NMR may, in future work, be employed to better link within-wall mechanisms to the physical effects observed here. The speed of the effect also suggests that the permeability of compressed mats (to a density of 525 kg m^{-3} this case) is not limiting. Measurements of the permeability of similar mats in related studies

(Haselein 1998; Bolton and Humphrey 1991) suggest that they had values in the order of 10^{11} m² at a density of 525 kg m⁻³. This compares with values in the order of 10^{16} m² for solid wood (Douglas-fir heartwood) in the transverse direction (Bolton and Humphrey 1991).

One of the main reasons that the initial experiments with ammonia were conducted on unconsolidated (loose) mats was that it was expected that penetration would then be unrestricted by resistance to flow. Evidently, this appears not to be a limitation.

The amount of ammonia consumed by the fiber network during treatment could not be directly measured due to its volatility once the press was opened. For purposes of experimental duplication by others, it may however suffice to specify the concentration (partial pressure) and temperature of gas applied since the behavior observed suggests that gas concentrations normalize quite readily within the pore structure. The addition of mass flow measurement instrumentation to the dosing system is planned for future work; this will enable mass balance to be evaluated.

Figure 9 indicates the effect of ammonia vapor pressure on its softening action. This effect has been represented as the reduction of counterpressure expressed as a percentage of that prevailing immediately before ammonia injection. The plot implies that modest vapor pressures of ammonia (in the order of 0.16 MPa) might be sufficient to produce useful levels of softening. Indeed, increasing concentrations above about 0.2 MPa appears to have little beneficial effect.

CONCLUSIONS

A sealed pressing system with provision for sequential injection and removal of reactive gases has been developed and tested. This offers great flexibility, and control of the conditions under which fiber and particulate mats may be treated and compressed. Permeable graphite has been shown to be a viable material for transferring both stress and fluids to fiber mats. Ceramic materials may, however, prove superior for fu-

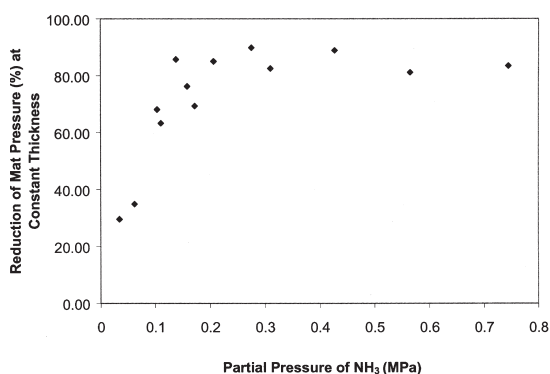


FIG. 9. Effect of ammonia partial pressure on percentage reduction in counterpressure upon ammonia injection.

ture development of laboratory and eventual industrial applications, not least because it may be formed with contoured surfaces and is highly resilient. The sealed pressing apparatus may prove useful for developing new treatment methods, for forming new composite samples, and for collecting certain material property data as a function of mat density and pre-treatment. The latter include hygro-thermo-viscoelasticity, thermal conductivity, and gas permeability—all mat properties that affect the formation mechanisms of composite products during their consolidation under industrial conditions, and therefore necessary input data for computer simulation models of pressing operations.

The sealed pressing system has been used to explore the softening action of gaseous ammonia on natural fiber networks in terms of treatment time and treatment vapor pressure. Preliminary tests on loose mats (100 kg m⁻³ density) suggested that fibers are rapidly softened by ammonia. The aim of the main part of the experiment was to examine the reduction of internal residual stress that occurs in compressed fiber mats due to ammonia injection. This was conducted in order to assess whether ammonia injection with the sealed pressing system may be used to achieve useful softening levels at acceptable rates. Reduction of internal stress was inferred by monitoring the reduction of force necessary to hold the mat at a constant density upon ammonia application. Results show that moderate

vapor pressures of ammonia (in the order of 0.16 MPa) can rapidly penetrate and soften mats compressed to a density of 525 kg m^{-3} at near-room temperature (29°C .) Full (in excess of 99%) of the potential relaxing effect was seen to be completed in 3.5 s of ammonia application.

The development of mechanistic viscoelastic models of diverse mat types under diverse ammonia treatment conditions was beyond the scope of the work reported here. Non-linear five-element models will, however, be developed in future studies since they will be necessary to provide material property input data for deterministic simulation modeling of novel sealed pressing approaches to be established. The effect of temperature on ammonia softening may also be the subject of future investigations. This may, however, be of limited importance in most practical applications since rapid pressing of composites at room temperature offers considerable advantages over the present dependency on the use of heat and moisture to affect necessary softening in composite manufacturing operations.

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