

DECOMPRESSION DRYING OF *PINUS RADIATA* SAPWOOD CHIPS

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ABSTRACT

Gas bubble nucleation and expansion in supersaturated aqueous solution, by decompression of the applied gas pressure, can remove free water from the cell lumens of a permeable wood structure. Carbon dioxide and nitrogen gas were used to evaluate the relative effects of the process variables pressure, temperature, and absorption time on the decompression drying of radiata pine sapwood chips. Water loss with carbon dioxide gas increased with pressure and absorption time, and varied with temperature. The volume of gas bubbles generated on decompression appeared to be an important criterion for water loss. The water loss also increased with repeated cycles of gas absorption and decompression drying, which suggests the proportion of air/water vapor present in the wood was also important in determining the amount of water loss. Water loss with nitrogen gas was determined by the number of cycles of decompression drying, rather than the period of nitrogen gas absorption. The very low solubility of nitrogen gas in water suggests the water loss occurred due to the compression and expansion of pre-existing air bubbles.

The nucleation of bubbles of a solute gas in supersaturated aqueous solutions provides a rapid means of drying sapwood chips. The supersaturated state is achieved by lowering the applied gas pressure, usually by some tens or hundreds of atmospheres, to atmospheric pressure. The number of bubbles and their rate of production increase with increasing supersaturation of gas dissolved in the water (1). The bubbles that form can effectively force the free water from the cell lumens within a permeable wood structure.

An assessment was made of the relative effects of a number of process variables on decompression drying. The variables included temperature, pressure, time, and the choice of solute gas. Temperature affects the rate of gas diffusion into water, pressure affects the concentration of dissolved gas in water, and time affects the amount of gas dissolved at a given temperature and pressure. The gases used were carbon dioxide and nitrogen. Both have similar diffusion coef-

ficients in water, but carbon dioxide gas has a much higher solubility in water than nitrogen gas (Table 1). High gas solubilities in water can be expected to improve the effectiveness of decompression drying by increasing the supersaturation of the gas in the water at decompression, thereby increasing the number and rate of gas bubble formation in the wood. The way in which the process variables affected the decompression drying response of the two gases was used to assess the effect of gas solubility on decompression drying.

METHODS AND MATERIALS

Temperature and pressure both have a strong effect on the solubility of carbon dioxide gas in water. A three-way facto-

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rial analysis of variance design was used to assess the effects of temperature, pressure, and absorption time on carbon dioxide gas decompression drying. A combination of six temperatures (15°, 20°, 25°, 30°, 35°, and 40°C), three gas pressures (1.50, 4.89, and 6.89 MPa), and three absorption times (5, 15, and 45 min.) were used with carbon dioxide gas. The gas pressure of 6.89 MPa could only be attained at temperatures of 30°, 35°, and 40°C.

The very low solubility of nitrogen gas in water at a pressure of 5.065 MPa (Table 1) suggests that the process variable pressure, which determines the concentration of dissolved gas in the water, was of little importance in determining the decompression drying ability of nitrogen gas compared with the variables temperature and absorption time. A two-way factorial analysis of variance design was used to assess the effects of temperature and time on nitrogen gas decompression drying. A combination of three temperatures (20°, 30°, and 40°C) and three absorption times (5, 15, and 45 min.) were used with a nitrogen gas pressure of 4.89 MPa.

Decompression drying with carbon dioxide and nitrogen gas may also be affected by the extent to which the wood chips are saturated with water. The expansion of dissolved supersaturated carbon dioxide or nitrogen gas into pre-existing gas/water vapor bubbles within

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Forest Prod. J. 49(1):67-72.

TABLE 1. — Carbon dioxide and nitrogen gas diffusion coefficients and solubilities in water.

| Gas | Diffusion coefficients ^a ($\times 10^{-9} \text{ m}^2 \text{ s}^{-1}$) | Solubilities ^b | |
|----------------|--|---|-------|
| | | 25°C (cm^3 of gas at STP/g of H_2O) | 50°C |
| Carbon dioxide | 1.99 | 27.23 | 17.25 |
| Nitrogen | 2.19 | 0.67 | 0.53 |

^a Measured at 25°C and 0.1013 MPa (2).^b Measured at 5.065 MPa (3,4).

TABLE 2. — Sapwood chip water loss with carbon dioxide gas.

| Source of variation | Degrees of freedom | Mean water loss | | Variance of water loss | |
|---|--------------------|-----------------|---------------------|------------------------|----------------------|
| | | MS | F | MS | F |
| Temperature | 5 | 239.16 | 8.75 *** | 0.127 | 13.99 ** |
| Pressure | 1 | 3483.41 | 127.49 ** | 0.558 | 61.20 ** |
| Time | 4 | 1852.01 | 67.78 ** | 0.007 | 0.74 ns ^b |
| Temperature \times pressure | 5 | 78.63 | 2.88 * ^c | 0.079 | 8.65 ** |
| Temperature \times time | 20 | 33.51 | 1.23 ns | 0.011 | 1.18 ns |
| Pressure \times time | 4 | 68.28 | 2.50 ns | 0.035 | 3.84 * |
| Temperature \times pressure \times time | 20 | 27.32 | | 0.009 | |

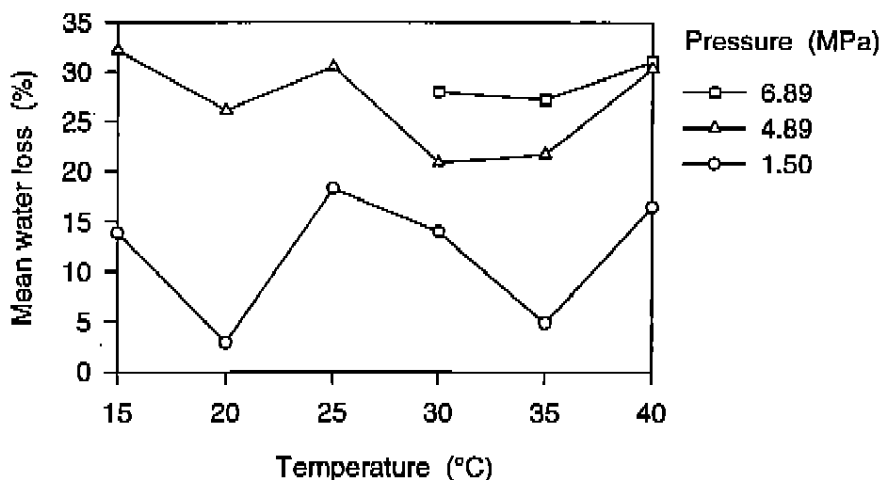
^a ** = $p < 0.01$ ^b ns = $p > 0.05$ ^c * = $p < 0.05$ 

Figure 1. — Mean water loss from sapwood chips with carbon dioxide gas. The values plotted are the means for the five absorption times.

the wood chip could improve the process of decompression drying. Repeated cycles of decompression drying were used to test the idea. Two cyclic decompression drying time series were used, one involving nine repeated cycles of 5 minutes absorption time and the other three repeated cycles of 15 minutes absorption time, both giving a total gas absorption time of 45 minutes.

Carbon dioxide and nitrogen gas decompression drying were compared using a three-way factorial analysis of variance for the combination of three

temperatures (20°, 30°, and 40°C) and three absorption times (5, 15, and 45 min.) at a pressure of 4.89 MPa. The variables in the analysis were temperature, time, and carbon dioxide versus nitrogen (CO_2 vs. N_2).

WOOD CHIP PREPARATION

Flat-sawn boards (100 mm by 25 mm) of *Pinus radiata* sapwood were dressed, ripped, and cross-cut to yield chips of dimensions 8 mm by 25 mm by 25 mm with a flat-sawn orientation. The chip moisture content ranged from 95 to 199

percent, with saturations of 93 to 99 percent, and basic densities of 372 to 611 kg/m^3 . The large range of values for chip moisture content and basic density reflect the large differences in the proportions of earlywood and latewood in the chips.

DECOMPRESSION DRYING PROCEDURE

The equipment consisted of two pressure cylinders connected in series to a carbon dioxide or nitrogen gas supply cylinder, all contained inside an air-conditioned room. The large reservoir cylinder was pressurized to the operating pressure, and its temperature was allowed to re-equilibrate to the operating temperature. There were 100 chips randomly selected for each combination of temperature, pressure, and time; 50 to measure decompression drying and 50 as controls to correct for evaporative water loss, particularly at elevated temperatures. The chips were individually weighed and placed in the small decompression drying cylinder or a metal container (for the controls). The decompression drying cylinder was pressurized from the reservoir cylinder. The effect was very rapid pressurization with only a small drop in pressure. This was quickly adjusted with additional gas from the carbon dioxide or nitrogen gas supply cylinder. The temperature change associated with pressurization was small, and quickly re-equilibrated to the operating temperature. Decompression after the required absorption time was rapid, taking just a few seconds depending on the pressure. The wood chips were individually weighed and the water loss calculated as a percentage of the total water content of the chip.

RESULTS

CARBON DIOXIDE

Decompression drying of *Pinus radiata* sapwood chips with carbon dioxide gas was strongly affected by pressure and to a lesser extent by temperature and absorption time (Fig. 1, Table 2). There was a similar pattern of water loss with temperature at pressures of 1.50, 4.89, and 6.89 MPa. However, at 25° and 30°C there was a very marked increase in water loss at low pressure (1.50 MPa) relative to high pressure (4.89 MPa). This gave rise to a significant temperature \times pressure interaction, which suggests the effect of temperature depends on the pressure at which the water loss occurs. The pattern of water loss with temperature at

low pressure (1.50 MPa) was very consistent for the different absorption times (Fig. 2).

The variation of water loss from the chips was affected by pressure, temperature and time (Fig. 3, Table 2). The effect of temperature was most pronounced at low pressure (1.50 MPa) and at lower temperatures there was a substantial increase in the variation of water loss relative to high pressure (4.89 MPa). This accounts for the significant temperature \times pressure interaction and suggests that the effectiveness of decompression drying at low temperatures and pressures depends increasingly on the characteristics of the chips. If we examine the effect of absorption time on the variation of water loss at pressures of 1.50 and 4.89 MPa (Fig. 4), a reversal in ranking occurs with longer absorption times having greater variation of water loss at low pressure (1.50 MPa) and slightly lower variation at high pressure (4.89 MPa). This explains the significant pressure \times time interaction and illustrates how variable the water loss is at low pressure.

Repeated cycles of gas absorption and decompression drying (nine cycles of 5 min. absorption, three cycles of 15 min. absorption) were more effective in removing water from chips than a single cycle of 45 minutes absorption at pressures of 1.50, 4.89, and 6.89 MPa (Table 3). There was no significant difference in the effectiveness of nine cycles of 5 minutes absorption and three cycles of 15 minutes absorption.

The variation of water loss was significantly lower with repeated cycles of gas absorption and decompression drying (nine cycles of 5 min. absorption, three cycles of 15 min. absorption) than a single cycle of 45 minutes absorption at pressures of 4.89 and 6.89 MPa. There was no significant difference in the variation of water loss with nine cycles of 5 minutes absorption and three cycles of 15 minutes absorption.

NITROGEN

Water loss with nitrogen gas was determined by the number of cycles of decompression drying, rather than the period of nitrogen gas absorption at 4.89 MPa (Fig. 5, Table 4). There were substantial increases in water loss with three cycles of 15 minutes, and nine cycles of 5 minutes, gas absorption and decompression drying. Water loss with one cycle of gas absorption and decompression dry-

TABLE 3. — Cyclic versus non-cyclic sapwood chip water loss with carbon dioxide gas.

| Source of variation | Degrees of freedom | Mean water loss | | Variance of water loss | |
|---------------------------------------|--------------------|-----------------|----------|------------------------|----------------------|
| | | MS | F | MS | F |
| Pressure 1.50 MPa | | | | | |
| Temperature | 5 | 201.41 | 4.37 *** | 0.201 | 10.54 ** |
| Time | 4 | 636.73 | 13.80 ** | 0.027 | 1.44 ns ^b |
| 45 vs. 9 \times 5 and 3 \times 15 | 1 | 649.49 | 14.08 ** | 0.033 | 1.75 ns |
| 9 \times 5 vs. 3 \times 15 | 1 | 25.26 | 0.55 ns | 0.002 | 0.12 ns |
| Temperature \times time | 20 | 46.14 | | 0.019 | |
| Pressure 4.89 MPa | | | | | |
| Temperature | 5 | 116.39 | 7.92 ** | 0.006 | 6.94 ** |
| Time | 4 | 1283.56 | 87.37 ** | 0.014 | 17.30 ** |
| 45 vs. 9 \times 5 and 3 \times 15 | 1 | 995.82 | 67.79 ** | 0.017 | 19.96 ** |
| 9 \times 5 vs. 3 \times 15 | 1 | 0.34 | 0.02 ns | 0.002 | 2.00 ns |
| Temperature \times time | 20 | 14.69 | | 0.0008 | |
| Pressure 6.89 MPa | | | | | |
| Temperature | 2 | 21.05 | 2.32 ns | 0.003 | 4.41 ns |
| Time | 4 | 524.65 | 57.84 ** | 0.007 | 9.39 ** |
| 45 vs. 9 \times 5 and 3 \times 15 | 1 | 461.27 | 50.85 ** | 0.013 | 17.24 ** |
| 9 \times 5 vs. 3 \times 15 | 1 | 6.41 | 0.71 ns | 0.0002 | 0.26 ns |
| Temperature \times time | 8 | 9.07 | | 0.007 | |

*** = $p < 0.01$

^b ns = $p > 0.05$

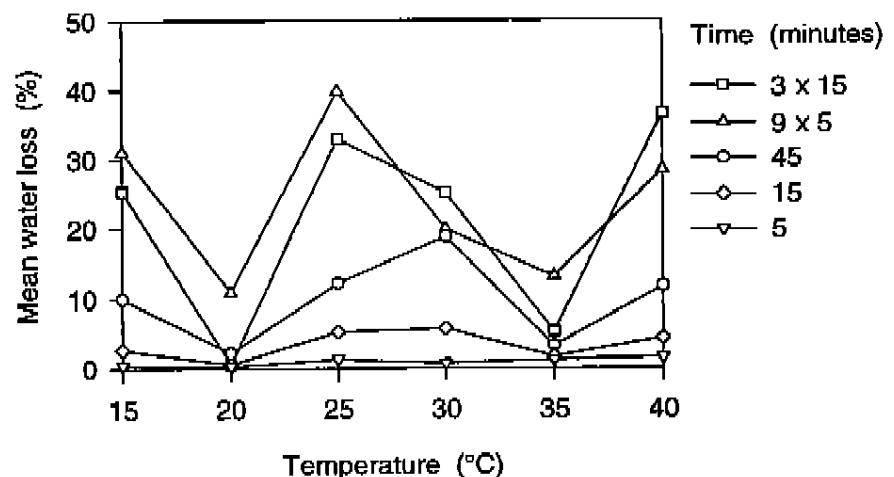


Figure 2. — Mean water loss from sapwood chips with carbon dioxide gas at 1.50 MPa.

ing was very low, even at absorption times of 45 minutes.

The number of cycles of decompression drying had no effect on the variation of water loss, which was low with nitrogen gas and increased only slightly at lower temperatures (Fig. 6, Table 4).

CARBON DIOXIDE VERSUS NITROGEN

Decompression drying of sapwood chips was more effective with carbon dioxide than with nitrogen gas at 4.89 MPa (Fig. 7, Table 5). There was only a very

small increase in water loss with absorption time for nitrogen gas, but a substantial increase in water loss with absorption time for carbon dioxide. This explains the significant time \times CO₂ vs. N₂ interaction, which can be attributed to the large solubility difference of the two gases in water (Table 1).

Chips dried with carbon dioxide at 4.89 MPa showed a similar water loss after three cycles of 15 minutes and nine cycles of 5 minutes, which suggests that at high gas solubilities in water both the

TABLE 4. — Sapwood chip water loss with nitrogen gas.

| Source of variation | Degrees of freedom | Mean water loss | | Variance of water loss | |
|---------------------|--------------------|-----------------|-----------------------|------------------------|---------------------|
| | | MS | F | MS | F |
| Temperature | 2 | 17.04 | 2.08 ns ^a | 0.023 | 6.30 * ^b |
| Time | 4 | 224.72 | 27.40 ** ^c | 0.004 | 1.22 ns |
| Temperature × time | 8 | 8.20 | | 0.004 | |

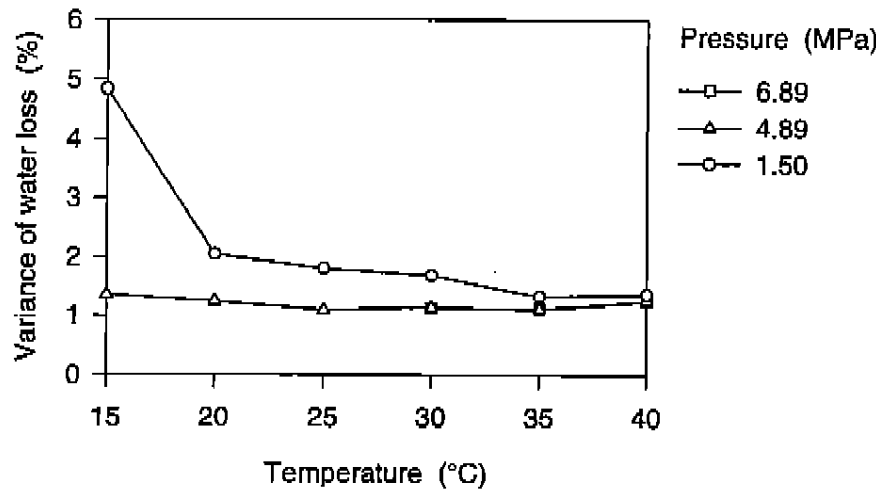
^a ns = $p > 0.05$ ^b * = $p < 0.05$ ^c ** = $p < 0.01$ 

Figure 3. — Variation of water loss from sapwood chips with carbon dioxide gas. The values plotted are the means for the five absorption times.

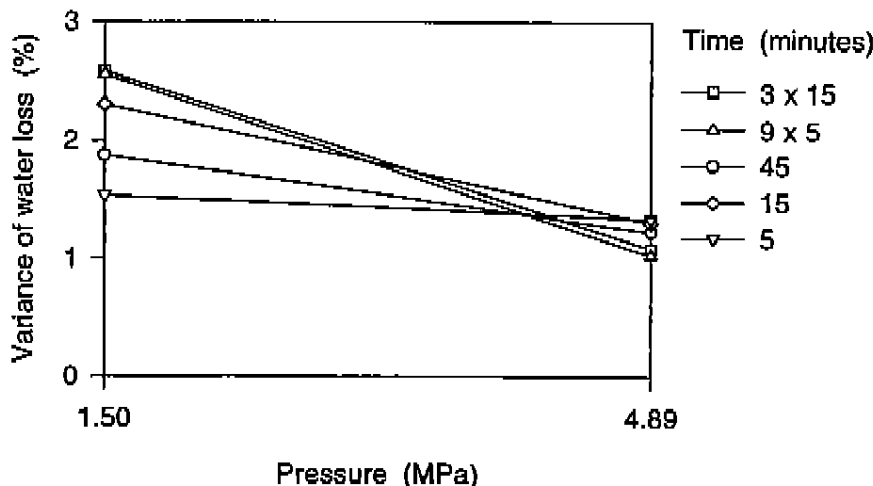


Figure 4. — Variation of water loss from sapwood chips with carbon dioxide gas. The values plotted are the means for the six temperatures.

length of gas absorption and the number of decompression drying cycles are important. Nitrogen gas, in contrast, showed

a far greater and surprisingly high water loss with nine cycles of 5 minutes compared with three cycles of 15 minutes gas absorption. The number of cycles of decompression drying, rather than the length of gas absorption, appeared to be more important, which suggests that

water loss with nitrogen gas was determined by factors other than the amount of gas absorbed. Examination of the chip water loss as a function of chip percent saturation, for repeated cycles of decompression drying with carbon dioxide gas at low solubility (Fig. 8), showed chip water loss was low above 90 percent saturation, but from 90 to 70 percent saturation the water loss increased substantially. This suggests that the increase in water loss can be attributed to the cumulative increase in the amount of air present in the chips, and it is this factor that appears to determine water loss at low gas solubilities.

The variation of water loss from chips was similar for carbon dioxide and nitrogen gas at 4.89 MPa. The variation was relatively constant with temperature for carbon dioxide but declined with increasing temperature from 20° to 40°C for nitrogen gas (Fig. 9). Although the temperature × CO₂ vs. N₂ interaction was significant (Table 5), the differences were very small.

DISCUSSION

For carbon dioxide gas, pressure had the most significant effect on water loss of the three variables analyzed: pressure, temperature, and time. This can be explained in terms of the large solubility differences of carbon dioxide gas in water between 1.50 and 4.89 MPa (6.6 and 22.2 cm³ of CO₂ at STP/g H₂O respectively¹). The considerably smaller solubility differences between 4.89 and 6.89 MPa (22.2 and 23.4 cm³ of CO₂ at STP/g H₂O respectively¹), help to explain the generally smaller increase in water loss at 6.89 MPa (Fig. 1).

While temperature had a significant effect on water loss (Table 2) the pattern of change was not consistent with solubility changes. The reduction in the solubility of carbon dioxide gas in water with increasing temperature at constant pressure might be expected to decrease the amount of water loss, and while there was a general decrease in water loss from 15° to 35°C at 4.89 MPa (Fig. 1) there were modest departures from it at 25° and 40°C. At 1.50 MPa, the departures were further accentuated (compared with 4.89 MPa), with very little water loss at 20° and 35°C, and high levels of water loss at 15°, 25°, 30°, and 40°C. The striking feature is that such large differences in water loss occur over temperature intervals as small as 5°C, and that they

¹ Solubility of carbon dioxide gas in water at 35°C (5).

occur consistently for each of the five absorption times (Fig. 2). Temperature anomalies occur in the properties of water near interfaces in capillaries and thin films where the superficial surface area is very small compared to the surface area available to the water molecule (6). The water properties near these interfaces can change abruptly at certain temperatures and over small temperature intervals, and such changes within the tracheids of wood could explain the interaction of pressure and temperature with decompression drying.

The effect of time was greater for gases of higher solubility in water. This was clearly demonstrated in the comparison between carbon dioxide and nitrogen gases (Fig. 7). The higher solubility of carbon dioxide gas in water produced a much greater increase of water loss with time than was obtained with nitrogen gas. It might be expected that at higher temperatures with lower solubilities and greater rates of gas absorption, the effects of increasing gas absorption time on water loss would diminish. The effect should show itself in a significant temperature \times time interaction, but in the analysis of variance the interaction was not significant (Table 2).

The proportion of air present in the wood was important for effective water loss at low gas solubilities in water (Fig. 8). The volume of gas generated by bubble nucleation at low gas solubility appears insufficient to expel the water. The loss of water is therefore thought to occur as a consequence of the compression of pre-existing air bubbles within the chips, creating a thin low-moisture zone at the chip surface. On decompression, the re-expansion of compressed air partially refills this zone and expels some water along with the gas. The very low solubility of nitrogen gas in water would suggest that water loss with nitrogen gas occurs by this process of air bubble compression and expansion. Once some of the initial water has been lost, it may be possible that compressed gas will get behind the wet line in the chip, via adjacent dry tracheids, and force water from the chip when it expands during decompression. This process could contribute to the increased water loss as the percent saturation of the chip decreases.

CONCLUSIONS

Decompression drying of *Pinus radiata* sapwood chips with carbon dioxide

TABLE 5. — Comparison of sapwood chip water loss with carbon dioxide and nitrogen gas.

| Source of variation | Degrees of freedom | Mean water loss | | Variance of water loss | |
|---|--------------------|-----------------|-----------|------------------------|----------------------|
| | | MS | F | MS | F |
| Temperature | 2 | 96.91 | 5.87 ** | 0.013 | 5.23 * |
| Time | 4 | 631.36 | 38.25 ** | 0.012 | 4.91 * |
| CO ₂ vs. N ₂ | 1 | 3043.56 | 184.39 ** | 0.004 | 1.59 ns ^c |
| Temperature \times time | 8 | 8.67 | 0.53 ns | 0.002 | 0.79 ns |
| Temperature \times CO ₂ vs. N ₂ | 2 | 33.03 | 2.00 ns | 0.012 | 4.99 * |
| Time \times CO ₂ vs. N ₂ | 4 | 153.25 | 9.29 ** | 0.002 | 0.86 ns |
| Temperature \times time \times CO ₂ vs. N ₂ | 8 | 16.51 | | 0.028 | |

^a * = $p < 0.05$

^b ** = $p < 0.01$

^c ns = $p > 0.05$

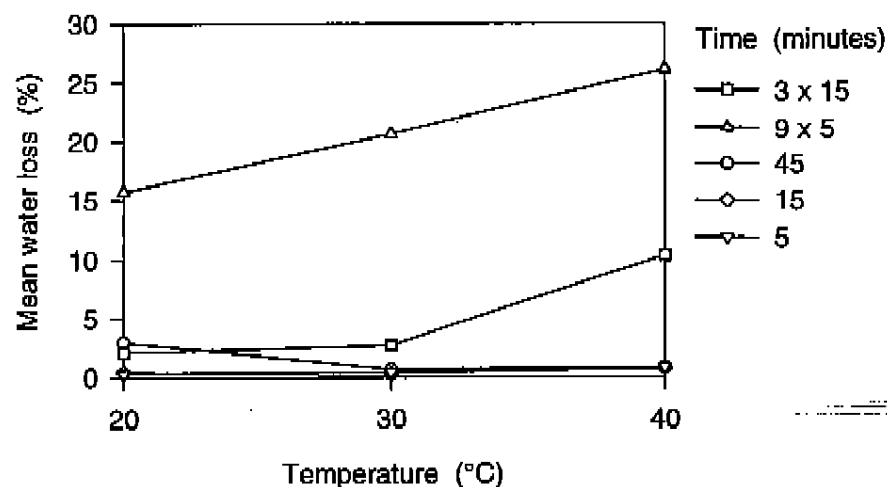


Figure 5. — Mean water loss from sapwood chips with nitrogen gas at 4.89 MPa.

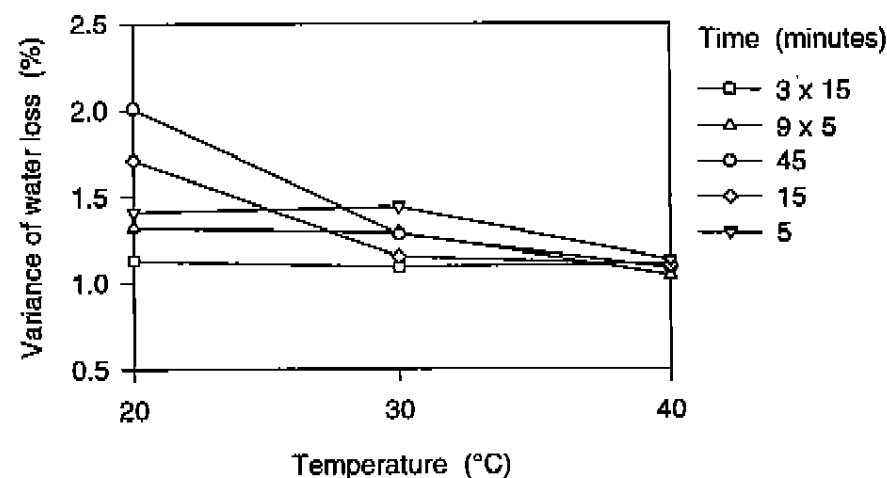


Figure 6. — Variation of water loss from sapwood chips with nitrogen gas at 4.89 MPa.

gas, at temperatures of 15° to 40°C, pressures of 1.50 to 6.89 MPa, and absorption times of 5 to 45 minutes was effective in removing a significant proportion of the

free water present. Water loss increased with pressure and absorption time, and varied with temperature. The effect of pressure and time can be attributed to the

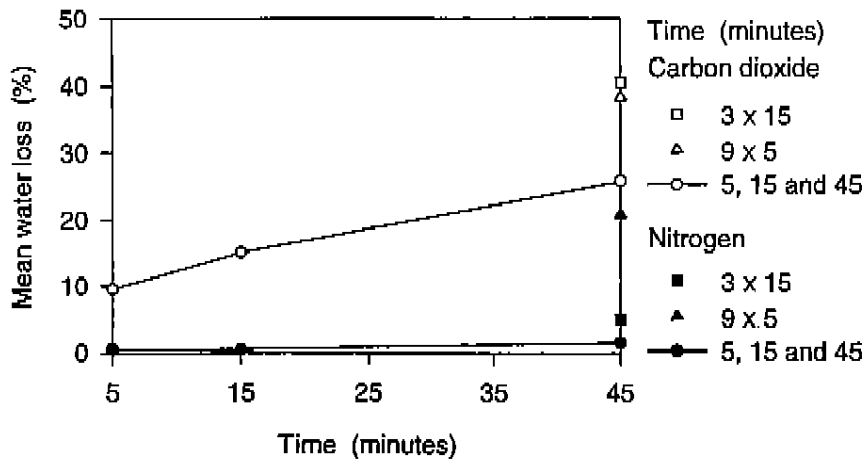


Figure 7. — Mean water loss from sapwood chips with carbon dioxide and nitrogen gas at 4.89 MPa. The values plotted are the means for the three temperatures.

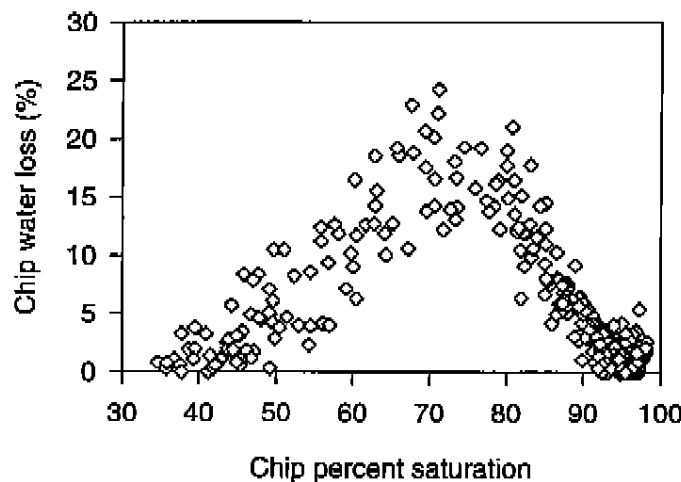


Figure 8. — Chip water loss with carbon dioxide gas at 15°C and 1.50 MPa as a function of chip percent saturation for each of nine repeated cycles of 5 minutes decompression drying.

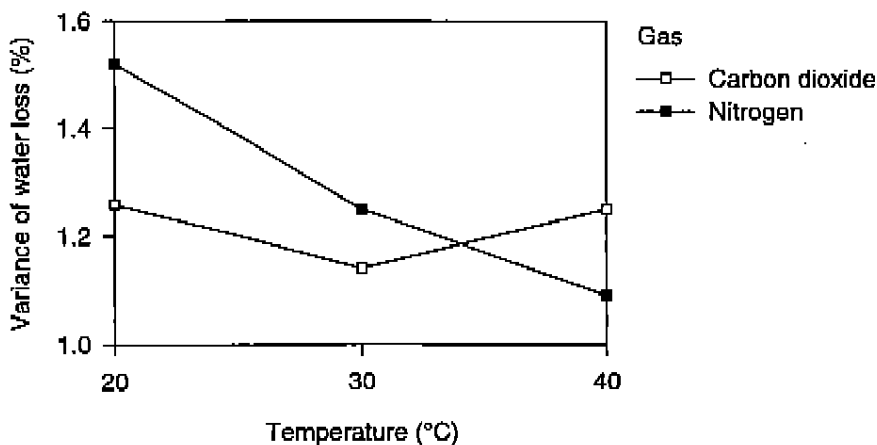


Figure 9. — Variation of water loss from sapwood chips with carbon dioxide and nitrogen gas at 4.89 MPa. The values plotted are the means for the five absorption times.

large increase in the solubility of carbon dioxide gas in water at higher pressures and the amount of carbon dioxide gas absorbed with time. The volume of the gas bubbles generated on decompression appeared to be an important criterion for water loss. The water loss also increased with repeated cycles of gas absorption and decompression drying. This suggests that the proportion of air/water vapor present in the wood was also important in determining the amount of water loss.

The variation of water loss with carbon dioxide gas increased with lower temperature and longer absorption time at a pressure of 1.50 MPa, but decreased slightly with longer absorption times at a pressure of 4.89 MPa. The variation of water loss was lower with repeated cycles of gas absorption and decompression drying at pressures of 4.89 and 6.89 MPa.

Water loss with nitrogen gas, at temperatures of 20° to 40°C, a pressure of 4.89 MPa, and absorption times of 5 to 45 minutes, was determined by the number of cycles of decompression drying, rather than the period of nitrogen gas absorption. The very low solubility of nitrogen gas in water suggests that the water loss occurred due to the compression and expansion of pre-existing air bubbles. Expansion of compressed carbon dioxide and nitrogen gas that had moved behind the wet line in the wood through adjacent dry tracheids may also have contributed to the increase in water loss with increased air volume.

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