Effect of fumigant dose, timber moisture content, end-grain sealing, and chamber load factor on sorption by sawn timber fumigated with ethanedinitrile

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Abstract Ethanedinitrile (EDN, syn. cyanogen, EDN Fumigas®) is a relatively new fumigant with the potential to disinfest quarantine pests from radiata pine (*Pinus radiata* D. Don) logs and sawn timber for export. This research evaluated the effects of dose (20 g/m³ or 50 g/m³), timber moisture content (green or kiln dried sawn timber), end-grain sealing (sealed or unsealed timber end-grain) and load factor (11% or 44%) on sorption of EDN by sawn timber at 15°C. This was quantified using headspace samples taken from 28-litre fumigation chambers. Chamber loading significantly influenced sorption, with higher loading resulting in greater sorption. Changes in the dose of EDN did not affect the sorption pattern. Increased moisture content and end-grain sealing both reduced sorption, but these effects were relatively small and the differences in sorption patterns caused by moisture content or end-grain sealing decreased over time.

Keywords EDN, fumigation, methyl bromide, *Pinus radiata* D. Don, quarantine treatments.

INTRODUCTION

Methyl bromide has been used in New Zealand as a quarantine and pre-shipment treatment for many years (Glasse 2013). The current New Zealand annual consumption of methyl bromide is 565 tonnes for the year ending June 2013 with the majority used for pre-shipment treatment of forestry products to meet quarantine requirements (STIMBR 2014). However, methyl bromide is an ozone-depleting compound and reduction of its use and the recapture and destruction of methyl bromide at the end of fumigations is strongly encouraged by governmental authorities (Glasse 2013). Moreover, the recapture of all methyl bromide remaining in the head space following fumigations will become mandatory in New

Zealand in 2020 (New Zealand Environmental Protection Authority 2011a).

Ethanedinitrile (EDN; C₂N₂, molecular weight 52.04) is a colourless, pungent gas with a boiling point of -21°C (Matheson Tri-Gas 2008). EDN was identified as an ozone-safe alternative to methyl bromide (CSIRO 2005) and its potential use as a disinfestation treatment for logs, timber and wood packing materials has been known since 1996 (CSIRO 1996). Recently, EDN Fumigas® was registered in Australia as a disinfestation treatment for logs and sawn timber (BOC 2012; Australian Pesticides and Veterinary Medicines Authority 2013) and EDN became available for testing in New Zealand (New

Zealand Environmental Protection Authority 2011b). To date, there is no information available on the toxicity of EDN to the pests of quarantine importance for New Zealand log and timber exports (Ren et al. 2005a, b; 2006).

An important characteristic of a useful fumigant is low sorption (Lorraine 2014). High sorption can reduce the concentration of fumigation doses to sublethal levels before a commodity has been disinfested (Bond 1984; van Someren Graver 2004; Darby 2008). Sinclair & Lindgren (1958) and Darby (2008) showed that sorption varied with the fumigant being evaluated and with changes in fumigation load factor.

Recent research on penetration and sorption of EDN on timber was reported by Ren et al. (2011) who used Douglas fir (*syn.* Oregon pine, *Pseudotsuga menziesii* (Mirb.) Franco) of low (7.8%) moisture content to show that the initial rate of sorption was significantly greater for EDN than for the other fumigants they tested (including methyl bromide) and that the penetration of some fumigants into the end-grain was greater than through the cross-grain. Additionally, Ren et al. (2011) showed that the quantitative data for a fumigant's sorption, penetration and efficacy could be combined to predict the efficacy of that fumigant at any specified depth in the wood. Similar studies are needed to elucidate the factors affecting EDN behaviour during fumigation of radiata pine (*Pinus radiata* D. Don.) timber at moisture content ranges found in timber exported from New Zealand.

The objective of this work was to measure the effects of fumigant dose, timber moisture content, end-grain sealing and load factor on EDN sorption by sawn radiata pine of standard dimensions exported to Australia. Of specific interest was EDN sorption by both green and dry timber, especially the effect of high moisture content of radiata pine on EDN sorption, because EDN is highly water soluble and known to dissociate rapidly in water (Matheson Tri-Gas 2008).

MATERIALS AND METHODS

Preparation of timber planks

All timber preparation took place in New Zealand. The logs were harvested on 2 September 2013, from Waitarere forest (Levin, latitude

-40.50°, longitude 175.21°). Three days after harvest, twenty 240 mm x 45 mm planks of timber (ca 26–64% moisture content) were milled from these logs at Mitchpine Products (Levin). Ten planks of green timber were covered with tarpaulins to maintain moisture content and stored at 4°C. The remaining ten planks were transferred to Eastown Timber Processors, Wanganui, for kiln-drying on the same day. The drying process took approximately 1 week to reach a moisture content of ca 14%. The green and dried planks were cut into 280 mm lengths (Figure 1), separately covered with tarpaulins, and stored at 4°C before fumigation.

Figure 1 Radiata pine planks with sealed end-grain using paraffin wax (left) and planks with unsealed end-grain (right).

Moisture content was determined using an oven-drying method adapted from standard test methods (ASTM Standard D4442, 2007). Ten planks each of green or kiln-dried planks were dried for 120 to 132 h using a Marford temperature controlled cabinet (GEO Wilton & CO. Ltd, Wellington) set at $103 \pm 2^\circ\text{C}$. The moisture content of timber was calculated on a percent wet weight basis using the formula:

$$\% \text{ moisture content} = 100 \frac{\text{wet weight} - \text{dry weight}}{\text{wet weight}}$$

One-half of the remaining planks were end-sealed by dipping each end to a depth of 2–4 cm into a container of warm paraffin wax 2–3 times to ensure that each plank end was thoroughly sealed. The wax was allowed to harden for 30–40 min before putting the planks back into the room at 4°C and covering them again with tarpaulins. Figure 1 shows planks with sealed and unsealed end-grain.

All planks were moved from storage at 4°C to another room at 15°C for 16 h before treatment began. Then, 28-litre fumigation chambers were loaded with either one or four planks and held at 15°C. The four planks were stacked (Figure 2) and each plank separated by steel nuts from the plank below or between the bottom of the chamber and the first piece of timber to allow air movement between the planks.

Treatments

Planks at moisture content ranges of 12–16% or 26–64% with sealed or unsealed end-grain were exposed to 20 or 50 g/m³ EDN in 28-litre fumigation chambers at 15°C for either 6 or 10 h. Sorption was monitored by measuring headspace

concentrations of EDN in each chamber over time. Each set of experimental parameters was replicated four times. Table 1 gives the EDN sorption test parameter matrix.

EDN delivery and monitoring

Before fumigation, either 250 ml or 625 ml of air was withdrawn from sealed 28-litre fumigation chambers to allow for the injection of 250 ml or 625 ml of pure EDN required to provide doses of 20 g/m³ or 50 g/m³, respectively. Pure EDN was transferred from a cylinder to each fumigation chamber using a 0.5 litre or a 1.0 litre gas-tight syringe connected to an EDN delivery system (BOC Australia, Sydney) as described by Roynon (2012).

Figure 2 Radiata pine plank stacking pattern before (left) and after loading (right) in a chamber prior to fumigation. Steel nuts were used as spacers between the bottom of the chamber and the first plank, and between the first and uppermost plank to separate them to allow air circulation.

Table 1 Treatments used to measure the effect of ethanedinitrile (EDN) dose, timber moisture content and chamber load factor on the sorption by sawn timber planks with and without sealed end-grain.

Treatment	EDN dose (g/m ³) ¹	Moisture content range (%)	Load factor (%) ²
1	20	12–16	11
2	20	26–64	11
3	50	12–16	11
4	50	26–64	11
5	20	12–16	44
6	20	26–64	44
7	50	12–16	44
8	50	26–64	44

¹1 g/m³ of EDN at 15°C is approximately 450 ppm.

²One plank (240 mm × 45 mm × 280 mm) ca 11% load factor and 4 planks ca 44% load factor in a 28-litre chamber. Chamber loading (load factor) is % volume occupied by timber (all fumigations were done at 15°C).

After the EDN was injected into each chamber, a circulating fan in each chamber was switched on immediately and kept on for the duration of each fumigation period to ensure thorough gas mixing. Headspace samples were collected at selected intervals up to 6 h or 10 h after treatment. The 10-h treatment duration used in the present study reflects a recent change in the Australian label for EDN, specifically EDN Fumigas® for the treatment of logs and timber using 50 g/m³ for 10 h (BOC 2014).

EDN concentration was quantified using an Agilent 7890A gas chromatograph instrument fitted with a flame ionisation detector and a 30 m × 0.53 mm GS-Q capillary column (Agilent Technologies Inc., Auckland, NZ). The oven and detector temperatures were maintained at 100°C and 300°C, respectively. The flow rates of helium, air and nitrogen carrier gases (BOC Gases NZ Ltd, Auckland) were 40, 400 and 20 ml/min, respectively. Gas samples were withdrawn using a 5 ml gas-tight syringe and a 3 ml sample was injected into a sample loop with a 1 ml subsample taken for analysis. The EDN concentration was measured on a part per million (ppm) basis, which was determined by a five-point calibration curve using dilutions of pure EDN in air. The EDN concentrations then were converted to g/m³ for reporting.

The initial headspace concentrations were estimated from the amount of EDN injected and the load factor, because EDN concentrations in the chambers could not be reliably measured until 5 min after gas injection when gas-mixing in the chambers by the fans was completed. Sorption studies often show higher scatter at the start of experiments (Darby 2008).

Statistical analysis

For each replicate of each treatment, a smooth curve was fitted to the EDN sorption data from which concentrations at any time could be estimated for statistical comparison. As the concentration generally showed an initial rapid drop followed by a more gradual decrease, an equation describing a two-phase exponential decay was fitted to each replicate:

$$C(t) = C_0(A\exp(-k_1t) + (1-A)\exp(-k_2t))$$

where $C(t)$ is the concentration at time t in hours,

C_0 is the initial concentration calculated from the injected amount of gas and the load factor, and A , k_1 and k_2 are parameters to be estimated separately for each replicate. Parameter k_1 is the exponential decay rate of the initial drop, k_2 is the exponential decay rate of the subsequent gradual decrease, and A specifies the relative weightings given to each of these processes.

Curve parameters were estimated by the method of least squares, using SAS PROC NLIN Version 9.2 of the SAS System for Windows (Copyright ©2008 SAS Institute Inc., Cary, NC, USA). All parameters were constrained to be positive, and k_1 was constrained to be less than 20 per hour because values greater than this suggest a rapid decay that could not be estimated with any accuracy.

Concentrations at times 1, 2, 4, 6 and 10 h were estimated for all replicates using the fitted curves. Two variables were analysed at each time point: the concentration $C(t)$, and the ratio of the concentration $C(t)$ to the initial concentration C_0 . The effects of dose, load factor, moisture content and end-grain sealing were estimated by regression analysis using SAS PROC GLM.

RESULTS AND DISCUSSION

Moisture content

The average moisture contents of dry and green timber were $13.7 \pm 0.4\%$ and $48.3 \pm 5\%$, respectively (Table 1). There was greater variation in the moisture content of green timber when compared with dry timber and the variation may be related to differences in moisture content between heartwood and sapwood (Harris 1954; Cown 1999).

Sorption of EDN by timber

A two-phase exponential function fitted the sorption data well, with an average mean square error of just 0.7 g/m³ across all times and test replications. Figure 3 shows the average of all fitted curves for treatments and all data used for fitting the curves. Studies on grain fumigation (reported by Darby 2008) show methyl bromide sorption also exhibited two phases, while phosphine only exhibited two phases on grain types that had a stronger sorption. This implies there are at least two rate-controlling factors occurring in the sorption process. Darby (2008)

suggested a mass transfer model suited to grain physiology. There are currently not enough data to speculate on the kinetic components associated with EDN sorption by wood.

Although all individual fitted parameters varied between replicates and treatments, the highest differences were between the two different loadings. With an 11% load factor, the relative importance of the more rapid decay process ($A = 0.20$) was much

smaller than with a 44% load factor ($A = 0.57$) (Table 2). After 1 h, EDN concentrations were typically about 89% of the initial concentration in treatments with 11% load factor but only about 38% of the initial concentration in treatments with a 44% load factor. On average, after 10 h the concentrations remaining in the 11% and 44% loading configurations were 61% and 20% respectively of the applied dose. Higher sorption

Figure 3 Fitted curves for the change in concentration of ethanedinitrile over time in 16 treatments of *Pinus radiata* sawn planks. Curves are the fitted average for all four replicates (black lines) and individual data (grey lines; cross = replicate 1, square = replicate 2, triangle = replicate 3, circle = replicate 4). Each graph is labelled with dose (20 or 50 g/m³), load factor (11 or 44%), moisture content (dry (13.7 ± 0.4%) or green (48.3 ± 5%)) and end-grain sealing (none (unsealed) or wax (sealed)). All fumigations were done at 15°C.

in the presence of higher load factors has been reported for other fumigants and other fumigated products. Sinclair & Lindgren (1952) showed that methyl bromide, ethylene dibromide, ethylene chlorobromide and hydrogen cyanide were more highly sorbed with higher load factors when oranges were fumigated. Darby (2008) reported highest sorption of methyl bromide by wheat occurred at the highest load factor tested. In the present work, the variation between treatments with different load factors was much greater than the differences between treatments with the same load factor for parameters A and k_2 (Table 2). This was not the case for parameter k_1 , which reflected the difficulty of estimating the slope for the initial rapid decay that occurred in all fumigations. The decay rate for the later gradual drop in concentration was about 3.6 times greater for fumigations using a 44% load factor ($k_2 = 0.136$) than for fumigations using an 11% load factor ($k_2 = 0.038$). These decay rates are close to those that could be expected based on the differences in surface area of timber available for EDN sorption being four times greater than the lower loading.

Ren et al. (2011) also showed that initial EDN headspace concentrations dropped rapidly to 50–60% of the original concentration in the first hour of using kiln-dried Douglas fir and a load factor of 20%.

Table 3 summarises effects of EDN dose, chamber load factor, timber moisture content, and end-grain sealing on EDN sorption by sawn timber. The interactions between these factors were not significant.

EDN dose and chamber load factor had a significant ($P < 0.001$) effect on EDN sorption at

Table 2 Average ($\pm$ SD) fitted parameters for ethanedinitrile headspace depletion by sawn timber at either 11% or 44% load factor.

Parameter	11% load factor	44% load factor
A	0.20 (± 0.03)	0.57 (± 0.06)
k_1	5.4 (± 2.7)	8.6 (± 2.5)
k_2	0.038 (± 0.005)	0.136 (± 0.015)

all times the concentration was monitored during treatment. The impact of the initial concentration dropped with time. For example, after 2 h the concentration due to a 50 g/m³ dose was on average greater than that of a 20 g/m³ dose by 22.2 g/m³, but after 10 h this difference dropped to only 12.6 g/m³. In contrast, over the 10 h fumigation, the effect of the load factor increased with time, e.g. after 2 h the concentration with a loading of 44% was on average less than that with 11% loading by 8.7 g/m³, but after 10 h this difference grew to 14.8 g/m³. Although dry timber appeared to cause a more rapid initial decrease of the EDN concentration, the difference in EDN concentration between dry and green timber became smaller and less significant over the total fumigation time. The effect of moisture content on fumigant sorption varies. Zahora & Morrell (1989) found that sorption of methylisothiocyanate in Douglas-fir heartwood was 5 times greater at a moisture content of 10% when compared with a moisture content of 70%. For wheat and maize fumigated with phosphine and methyl bromide, Vincent & Lindgren (1971) found highest sorption occurred at highest grain moisture content. When end-grain was sealed, less EDN was absorbed in the first hour

Table 3 Differences in headspace concentration (g/m³) due to ethanedinitrile (EDN) sorption by sawn timber after fumigation periods of 1, 2, 4, 6 or 10 h at 15°C under different EDN dose, chamber load factor, timber moisture content and end-grain sealing treatments.

Factor	Fumigation time (h)				
	1	2	4	6	10
Dose (50 g/m ³ versus 20 g/m ³)	25.1 *** ¹	22.2 ***	18.7 ***	16.2 ***	12.6 ***
Load factor (44% versus 11%)	-5.9 ***	-8.7 ***	-11.5 ***	-13.3 ***	-14.8 ***
Moisture content (green versus dry)	3.1 ***	2.8 ***	2.0 *	ns	ns
Sealed versus unsealed ends	1.6 *	ns	ns	ns	ns

¹ns = differences not significant or significant at * $P < 0.05$, ** $P < 0.01$ or *** $P < 0.001$.

Figure 4 Curves for the ratio of actual concentration to initial concentration (C/C_0) plotted against time, regardless of dose of ethanedinitrile applied to *Pinus radiata* sawn planks. Values are the average for loading (solid lines = 11%, dashed lines = 44%), moisture content (black = green, grey = dry) and sealed end (thick lines = unsealed, thin lines = sealed) treatments. The lines for sealed and unsealed end-grains overlap at 11% loading.

($P < 0.05$), but at later times differences between headspace concentrations with and without end-grain sealing were not significant.

Figure 4 shows the average fitted curves for the ratio of EDN headspace concentration to the initial concentration (C/C_0) regardless of the initial dose. Relative concentrations were, of course, 1.0 at the start, but from 1 h onwards the differences between relative concentrations in the 11% and 44% loading treatments were approximately those predicted by Table 4. Changes in the dose of EDN did not affect the sorption pattern. The effect of dose on this ratio was not significant at any of the times considered. This suggests that approximately the same shape of curve is followed regardless of the applied dose, so the time course of concentration change can be simply scaled up or down when different doses are applied.

Regardless of EDN dose, chamber loading of 44% led to a much more rapid initial drop in chamber EDN concentration than 11% loading, with the difference reaching 39% of the initial concentration after 1 h (Table 4). Drying the timber led to a more rapid initial drop in concentration, but differences in concentration due to moisture content then decreased with time. Headspace EDN concentrations also initially decreased more rapidly in fumigations of planks with unsealed end-grain than with sealed end-grain. These concentration differences then became smaller over the fumigation period. For planks with sealed end-grain, the total sealed surface area accounts for ca 13.5% of the total surface area of the plank. By comparing the curves of EDN concentrations of planks with sealed versus unsealed ends the average slopes of

Table 4 Differences in the ratio of ethanedinitrile (EDN) concentration to initial concentration in the headspace following sorption by sawn timber for fumigation periods of 1, 2, 4, 6 or 10 h at 15°C under different chamber load factor, timber moisture content and end-grain sealing treatments.

Factor	Fumigation time (h)				
	1	2	4	6	10
Load factor (44% versus 11%)	-0.39 *** ¹	-0.42 ***	-0.44 ***	-0.45 ***	-0.43 ***
Moisture content (green versus dry)	0.06 ***	0.06 ***	0.04 ***	0.03 **	ns
Sealed versus unsealed ends	0.04 **	0.03 *	ns	ns	ns

¹ns = differences not significant or significant at * $P < 0.05$, ** $P < 0.01$ or *** $P < 0.001$.

the curves over the first 30 min of the fumigation was ca 13.5% steeper for unsealed than sealed planks (Figure 4). The present data therefore provide no evidence that the sorption of EDN by the end-grain of sawn timber was significantly higher than that of other surfaces. In commercial practice, using much longer lengths of timber, the influence of end-grain will be even less than in this experiment.

CONCLUSIONS

The results of this study illustrate that headspace concentrations of EDN change in proportion to the applied dose. The sorption pattern of EDN by the sawn timber as measured by the concentration of gas in the headspace at any time compared with the initial concentration in the chambers was mainly influenced by the loading factor. On average, after 10 h the concentrations remaining in the 11% and 44% loading configurations were 61% and 20% respectively of the applied dose. The moisture content of the timber and sealing of the end-grain of planks had minimal effects on the sorption pattern.

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