



Review and Comparison of Test Methodologies Used to Evaluate the Leaching of Wood Preservatives

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The extent to which wood preservative components leach or otherwise migrate from treated wood during its life cycle is an important factor affecting serviceability and acceptability of treated products by consumers and regulatory agencies. Preservative emissions from treated wood during storage at the treating plant and in consumer storage areas may contaminate the soil and groundwater, and potentially require costly remediation. The extent of leaching in service significantly affects the service life of treated wood products and helps determine the suitability of different preservative systems for specific end uses. Leaching and other losses from treated products in service may also increase contaminant levels in soil, surface water, and groundwater near the treated products, resulting in localized environmental impacts affecting aquatic and soil organisms. Leaching of preservatives from wood removed from service and disposed of in landfills has the potential to contribute to groundwater contamination. The accumulations of preservative components in different environmental compartments at different stages of product life cycles are used in risk assessments of potential ecotoxicity and health effects of preservative use.

Preservative treatments are evaluated for their potential to lose efficacy or their propensity to contaminate their surroundings by a number of different standardized or non-standardized test methods, usually designed to address either efficacy or risk to the environment. Typically, the leaching characteristics are expressed in one of the following ways:

- % leaching of total preservative component during the test or by the end of the test. This provides a good assessment of the preservative persistence, i.e., ability of the component to remain in wood over a

long exposure time, and is indicative of its long-term efficacy to protect wood. This measure is not useful for predicting potential risks of preservative leaching to the environment, except as a relative measure of preservative stability in wood.

- Cumulative emission or flux of preservative component from the wood, expressed as mass per unit of exposed surface area, e.g., mg/m². Such values, in combination with the structure dimensions, exposure conditions, stability of the leached component, and the volume of the receiving water, soil, or sediment sink, allow estimation of the "Predicted Environmental Concentration" (PEC), which can be compared with the "Predicted No Effect Concentration" (PNEC) for risk assessment (Deroubaix et al. 2000). This provides a leaching estimate that can be applied to different products or specimen sizes to predict worst-case cumulative leaching, normalized to the exposed surface area of the structure. It is a measure of the cumulative environmental loading.
- Flux rate or loss per unit time, obtained by dividing the measured flux by the leaching interval. This is somewhat arbitrary, as the interval selected may be the time between successive leaching events or measurements, the duration of the actual leaching period, or an arbitrary time that a leaching exposure is meant to represent. For example, the proposed test protocol by the Organization for Economic Co-operative and Development (OECD) to represent aboveground leaching exposure specifies two one-hour soaking periods to represent one day of leaching exposure (OECD 2006). Flux rate provides a measure of potential acute ecotoxicity effects and identifies the interval where emission rates are highest.

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Leaching performance of treated wood in leaching tests or in service depends on a number of variables, including wood product or specimen size, dimensions and grain orientation, exposure time, properties of the leaching solution, exposure conditions, such as wetting and drying and UV exposure, and the development of wood checks or splits during exposure. Many of these factors are discussed in Chapter 7 of this book and in such review papers as Lebow (1996), Brooks (1997), Cooper (1994, 2003), Hingston et al. (2001), and Lebow et al. (2004). Factors specifically affecting leaching results of laboratory test methodologies will be reiterated here in the context of how they influence the interpretation of results from different testing methods.

In this chapter, different testing protocols are discussed and compared and advantages, disadvantages, potential problems, and other considerations discussed. In most cases, comparisons are made between methodologies, using results of comprehensive leaching comparisons of three copper-based wood-preservative systems: chromated copper arsenate (CCA-C), alkaline copper quaternary ammonium compound (ACQ-D), and a micronized copper system (MCQ) based on 2:1 copper carbonate and a quaternary ammonium compound, dimethyldidecyl ammonium chloride (DDAC) (Cooper and Ung 2008, 2009).

13.1 EVALUATION OF LEACHING TENDENCY SOON AFTER TREATMENT—PRESERVATIVE FIXATION

It is important to minimize leaching and other losses at the preservative treating plant, as well as during transport and storage of treated products before they are placed in service. This is usually accomplished by ensuring that pretreatment, treatment, and post-treatment processing ensure maximum cleanliness of the treated product and, where appropriate and possible, a high degree of preservative fixation. This can be achieved through guidelines and “Best Management Practices” (BMPs) that specify process requirements, occasionally complemented by simple quality-control tests at the treating plant. Examples of these are Lebow and Tipple (2001), the Western Wood Preservers Institute (WWPI) (1996) *Best Management Practices for the Use of Treated Wood in Aquatic Environments*, and the technical recommendation documents (Brudermann 1999) commissioned by Environment Canada and the Canadian wood-treating industry as guides to

minimize environmental and health impacts of treated wood.

For chromated copper arsenate (CCA), the fixation reactions of all preservative components are essentially complete when the hexavalent chromium in the wood has been reduced to the trivalent state and the potential for leaching of all CCA components is minimized (Cooper et al. 1996). Simple quality-control tests have been developed to indicate when most of the chromium has undergone its fixation reaction. These include the chromotropic acid test (Foster 1988) and tests based on the analysis of solution leached or expressed from treated wood borings, based on the color reaction of hexavalent chromium with diphenylcarbazide (Coggins and Hiscocks 1987, Cooper and Ung 1993). For the chromotropic acid test, treated borings are placed on absorbent paper and a drop of the chromotropic acid solution added. The absence of a purple color reaction is an indication that more than 99% of the chromium has been reduced. The advantages of this test method are its high sensitivity, rapidity and ease of testing, and interpretation. The main disadvantage is that it is a “go/no go” test that does not indicate the progress of fixation and occasional problems with false positives due to other metals and false negatives resulting from aged or improperly prepared solutions. Analysis of expressate or leachate can provide an indication of the extent of fixation, but these tests are more time-consuming and require access to reagents and a visible light spectrophotometer. These tests can be used for any hexavalent chromium-based preservative.

Testing is more problematic on other wood-preservative systems, such as copper-amine-based preservatives, since there are no simple spot tests for copper, borates, or organic co-biocides. The American Wood Protection Association (AWPA) standard AWPA A32-02 “Standard Method for Measuring Losses of Inorganic Preservatives from Treated Wood,” was developed to fill this need (AWPA 2006). This procedure is based on the analysis of leachate from a set of treated borings removed from a charge of treated wood. A similar standard is currently being developed by the Canadian Standards Association (CSA) that is applicable to Canadian wood species. It can be used to either follow the fixation process for a given charge by monitoring the reduction in leachable components over time or, if and when standard limits are defined, test whether a given charge meets these defined limits. The main challenge of this protocol is the need to develop or

identify simple, but precise and reproducible analytical methods that can be applied in the treating plant QC laboratory for the preservative components of interest. Also, the amount of leaching of these systems is greatly affected by the preservative retention and it will be necessary to define an acceptable level of leaching by this method based on a risk assessment for contamination of the treating facilities.

More complex leaching protocols have been developed to assess the risk of emissions from freshly treated wood during storage and to develop post-treatment conditioning to reduce emissions to an acceptable level. For example, a protocol developed in the Netherlands (Homan et al. 1993) subjects a 0.5-m³ pack of lumber to a one hour (20 L) water spray, followed by drying for 23 h. The cycle is repeated for 5 consecutive days and the amounts of contaminants are measured (mg/m³) and compared to a prescribed acceptable level.

13.2 TEST PROTOCOLS DEVELOPED TO EVALUATE LONG-TERM STABILITY OF PRESERVATIVES TO WATER OR SOIL EXPOSURE

Some of the most commonly used and referenced leaching test methodologies were developed in conjunction with laboratory decay tests as a preconditioning treatment to confirm that the preservative had sufficient resistance to leaching to validate it for exterior exposure. These are highly aggressive tests that result in leaching exposure that may be equivalent to many years of natural exposure in “fixed” preservative systems. Perhaps the best known examples of such tests are AWPA E11-06 (AWPA 2006) and EN 84 from the British Standards Institute (BSI 1989). The AWPA recently developed Standard AWPA E20-06 (2006) in recognition of the potential effects of soil contact and properties on leaching of preservatives in ground contact.

13.2.1 AWPA E11-06: “Standard Method of Determining the Leachability of Wood Preservatives Immersed in Water”

For this procedure, six 19-mm cubes of treatable sapwood are vacuum treated with the preservative system of interest, conditioned, and dried. The blocks are placed in 300 ml water and vacuum impregnated with the water. The blocks in water are gently stirred or shaken, and at fixed

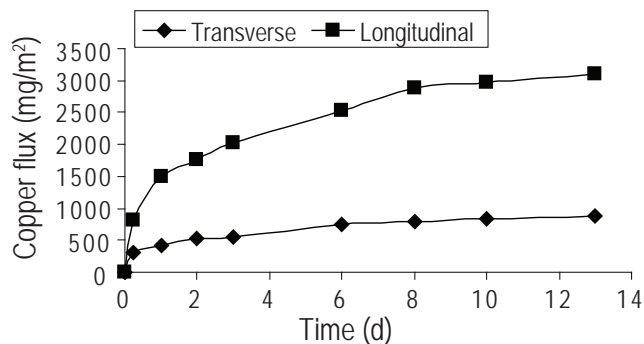


Figure 13.1 Effect of leaching direction on copper leaching from southern pine 19-mm blocks treated with ACQ to a retention of 9.6 kg/m³ and sealed to limit leaching to one direction (Waldron et al. 2005).

periods of 6, 24, and 48 h, and every 48 h thereafter for a total of 2 wk, the water is replaced with fresh water and a sample of leachate collected for analysis.

Leaching is faster from the porous end grain of wood, and the impact of this effect on total leaching varies with specimen dimensions. One-third of AWPA E11 specimen surface area is end grain, which significantly affects specimen leaching. This can be illustrated by assessing leaching rates from ACQ-treated southern pine AWPA E11 specimens (19 mm cubes) sealed to permit leaching in either the longitudinal or transverse directions (Figure 13.1).

Cumulative leaching is determined by analyzing all of the leachate, but it can also be determined by analysis of unleached and leached samples. Cumulative leaching is usually reported as percent of component leached (% leaching), but can also be expressed as flux (e.g., mg/m²) or flux rate (e.g., mg/m²/d). Results for a comparison of three preservative systems (CCA-C, ACQ-D and MCQ) are summarized in Table 13.1. Figure 13.2 shows average results for % leaching (13.2a), flux (13.2b), and flux rate (13.2c) for the copper component of the three preservative systems.

Typically, % leaching as a result of this test method is relatively high because of the small dimensions and high proportion of end grain of the samples; for fixed-preservative systems this may represent several years of natural leaching losses. Leaching results expressed as flux values (e.g., Figure 13.2b) or flux rates (Figure 13.2c) are also high over the 2-wk test period because of the large proportion of end grain in the samples. However, because of the high surface-area-to-volume ratio in these specimens, the total emissions level off rapidly and the cumulative flux may

Table 13.1 Comparison of AWPA E11 leaching characteristics for southern pine samples treated with three preservative systems.^a

Preservative and component	Total retention (kg/m ²)	Cumulative % leached	Cumulative flux (mg/m ²)	Initial flux rate (mg/m ² /d)	Final flux rate (mg/m ² /d)
MCQ-Cu	4.0	1.7 (0.25)	179 (19.3)	232 (27)	1.8 (0.23)
	6.4	1.3 (0.80)	200 (6.2)	213 (41)	1.9 (0.15)
CCA-Cu	4.0	2.4 (0.21)	74 (4.9)	44 (14)	1.4 (0.29)
	6.4	2.2 (0.01)	107 (3.9)	90 (6.7)	2.2 (0.63)
CCA-Cr	4.0	0.6 (0.08)	25 (4.0)	38 (8.0)	0.03 (0.01)
	6.4	0.3 (0.03)	23 (2.8)	42 (10.0)	0.03 (0.01)
CCA-As	4.0	8.2 (0.21)	329 (8.6)	112 (23)	10.5 (0.49)
	6.4	6.3 (0.08)	436 (15.0)	175 (22)	12.2 (1.24)
ACQ-Cu	4.0	12.8 (3.78)	1302 (324)	2222 (393)	4.5 (0.12)
	6.4	13.7 (0.38)	1723 (55)	2614 (89)	7.7 (0.39)

a. Values represent means of three replicates. Standard deviations are in parentheses.

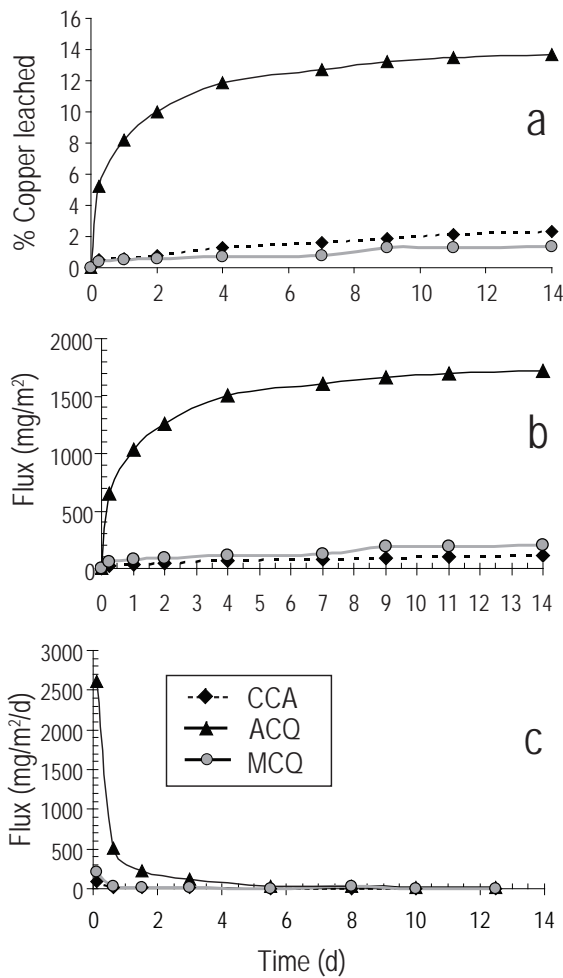


Figure 13.2 Copper leaching from southern pine blocks treated with CCA, ACQ or MCQ to a retention of approximately 6.4 kg/m³ and leached for 14 days according to AWPA Standard E11 and expressed as (a) % loss; (b) flux in mg/m²; and (c) flux rate (mg/m²/d). Adapted from Cooper and Ung (2008).

be lower than observed for long-term exposure of large-dimension products, since much of the readily available preservative is depleted during the test. Stated another way, a large-dimension sample has a larger reservoir of soluble preservative relative to its surface area (Lebow et al. 2008).

The initial flux rate (Figure 13.2c) for AWPA E11 is always very high for the first leaching interval, compared to natural leaching conditions, because the wood is already saturated with water—so rapid leaching can occur immediately and the leaching interval is short (6 h). As noted later, this tends to greatly over-estimate the short-term releases compared to other test methods with longer sampling intervals and exposure to natural leaching conditions.

Different preservative systems and components show different leaching characteristics. ACQ is associated with higher copper emissions than the other preservative systems, and the amount released increases with retention at rates that are higher than for the other systems. The arsenic leaching rate from CCA does not drop off as rapidly as copper leaching, regardless of the system evaluated. This has also been observed in natural weathering exposures (e.g., Taylor and Cooper 2003). Chromium leaching is relatively low, with a relatively high initial flux rate, but very rapid decline in emissions over the life of the test.

These effects suggest that the AWPA E11 test method is appropriate for comparing systems for long-term leaching potential, but not for risk assessments. This testing protocol is used extensively around the world to compare different preservative types or to evaluate effects of different formulations or conditioning treatments because

of its testing simplicity and relatively fast results. However, the small specimen size and high amount of end grain make this a highly aggressive leaching regime that should not be used to predict emission rates by extrapolating the results to larger products or specimens.

13.2.2 EN 84 Standard Wood preservatives: “Accelerated Ageing of Treated Wood Prior to Biological Testing”

This standard was also developed as a conditioning treatment for a laboratory decay exposure test according to European standard EN 113. It is more realistic than the AWPA E11 test because it uses specimens with a longer aspect ratio and less relative end grain (15 mm × 25 mm × 50 mm). In all other regards, the test methodology is similar to that of the AWPA E11, except that five specimens are leached in 500 ml water with water exchange and analysis after 2, 24, 48, 72, and 96 h, then every 48 h for a total of 2 wk. The lower proportion of end grain results in somewhat lower leaching rates and amounts than AWPA E11 (Waldron et al. 2003). Consideration should be given to the grain orientation of specimens, as flat-sawn specimens may leach differently than quarter-sawn specimens (Waldron et al. 2005).

13.2.3 AWPA Method E20-06: “Standard Method for Determining the Leachability of Wood Preservatives in Soil Contact”

It is recognized that soil contact can accelerate leaching and/or degradation of wood preservatives and that this effect is highly dependent on the physical, chemical, and biological properties of the soil (Crawford et al. 2002,

Lebow et al. 2006). AWPA Standard E20-06 was developed to evaluate this effect. Specimens (14 mm × 14 mm × 250 mm) are treated with the preservatives to ground-contact-specified retentions and stored without drying for 5 d. Treated samples are cut into two segments (100 mm for initial analysis and 150 mm for exposure) and 25 specimens (5 replicates from 5 different boards) are buried in soil in containers at moisture contents representing the soil water-holding capacity. Water is added as needed to maintain these moisture conditions. The selected soils (usually 2) represent different soil conditions and the relevant soil physical and chemical properties are measured. After 12 wk of exposure, the exposed and unexposed samples are analyzed for residual preservative and these values are used to estimate preservative loss. The specimen geometry ensures that the relative end-grain amount is reasonable for actual exposure conditions. Losses are usually presented as % loss; since losses are determined at the end of the study, only cumulative flux (mg/m²) and average flux rate (mg/m²/d in test) can be determined.

This test methodology allows one to evaluate the impacts of soil chemistry and texture and biological degradation on the loss of preservatives in soil contact. However, soils are highly variable and it is impossible to standardize soils from one test facility to another. Thus, it is difficult to compare results with other studies or to get reproducible results from test to test. The analysis also requires comparison of retentions in two different segments of wood (although end-matched), and there can be considerable variation in retentions, which results in large relative errors. Table 13.2 and Figure 13.3, showing results for exposure of wood treated with CCA, ACQ, or MCQ in two high organic

Table 13.2 Comparison of AWPA E20-06 leaching characteristics for southern pine samples treated with CCA, ACQ, or MCQ to 6.4 kg/m³ and exposed to two organic soils^a (Cooper and Ung 2008).

Preservative and component	Soil	Total % leached	Total flux (mg/m ²)	Average flux rate over 12-wk test (mg/m ² /d)
MCQ-Cu	1	16.2 (6.2)	1790 (394)	21.3 (4.7)
	2	18.9 (4.3)	2087 (339)	24.8 (4.7)
CCA-Cu	1	7.5 (3.6)	269 (122)	3.2 (1.4)
	2	23.1 (3.1)	822 (135)	9.8 (1.6)
CCA-Cr	1	8.3 (5.2)	521 (336)	6.2 (4.0)
	2	13.3 (3.5)	797 (181)	9.5 (2.2)
CCA-As	1	14.9 (6.0)	919 (397)	10.9 (4.7)
	2	16.1 (5.2)	948 (267)	11.3 (3.2)
ACQ-Cu	1	30.6 (5.9)	3277 (644)	39.0 (7.7)
	2	32.7 (3.4)	3742 (705)	44.6 (8.4)

a. Values represent the mean of 3 replicates. Figures in parentheses represent one standard deviation.

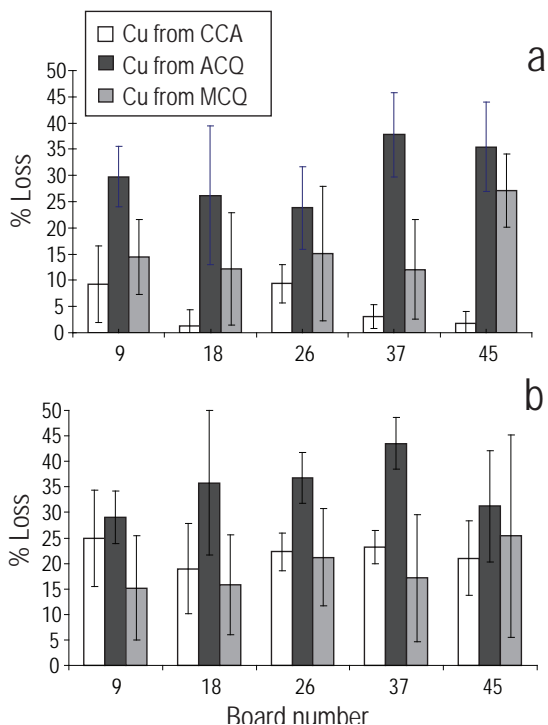


Figure 13.3 Comparison of estimated % copper leaching from CCA, ACQ, and MCQ treated 14-mm × 14-mm × 150-mm stakes exposed in two organic top soils: (a) Group 1 - U of T; and (b) Group 2 - Sault Ste Marie, as tested by AWP A E20-06 (Cooper and Ung 2008).

content soils, exemplify this high variability in results. There is high variability in the estimated % leaching both among boards and among replicates from the same board. Lebow and Halverson (2008) found that this test estimates preservative losses of wood in natural ground contact better than does AWP A Standard E11 testing when soil from the field site was used in the test, but also observed relatively high variability in leaching estimates.

13.2 TEST PROTOCOLS DEVELOPED SPECIFICALLY TO COMPARE OR EVALUATE ENVIRONMENTAL RISKS FROM TREATED WOOD EXPOSED TO WATER

As a requirement for the European Union Biocides Products Directive, OECD undertook the development of scenarios for different leaching exposures and test methods that adequately simulated real-life exposures to provide information on potential risks of preservative-treated wood

(Schotnecht et al. 2004). Two draft protocols were developed for aboveground (Draft Guideline I) and ground or water contact (Draft Guideline II) (OECD 2006). The procedures use EN 84/113 specimens (15 × 25 × 50 mm) with the option to use different specimen dimensions, provided the exposure is consistent in terms of wood surface area to water volume. However, the specimens are end-sealed to better represent full-size treated products.

13.2.1 OECD Draft Guideline I: "Laboratory Method for Wood held in Storage after Treatment and for Wooden Commodities that are not covered, and are not in Contact with Ground"

Wood samples for this test are treated to specified aboveground retentions (or other retentions for considering losses in storage) and may be treated in the laboratory or cut from commercially treated lumber. To simulate aboveground exposure to intermittent rain and drying between rain events, this protocol specifies dipping five replicate samples in 500 ml deionized water for a fixed time twice per day. The latest suggested revision of this guideline recommends a one hour immersion for each dip exposure, and intervals for "rainfall events" are set as 1, 3, 5, 8, 10, 12, 15, 17, and 19 d. The test specimens are allowed to dry under ambient conditions between immersions (4 h) and between successive sampling days. Fresh water is used on each test day and the exposed water is collected for each replicate sample on each sampling day for analysis. Emissions are expressed as mg/m² and flux rates as mg/m²/d from analytical results; the analyses can also be used to determine % leached. For flux rates, the exposure time is considered one day for each sampling day (two 1-hour dips).

There is ongoing debate about the appropriate dipping time for this test and the two 1-h tests represent longer times than originally proposed (Schotnecht et al. 2004). Leaching flux and flux rates (for example, Figure 13.4) appear to be reasonable for the early exposure of treated products to natural weathering. The leaching per unit area rates can be applied to large structures in service to provide a reasonable estimate of leaching rates. Lebow et al. (2008) suggest that the drying phase does not reproduce the range in moisture contents found in natural aboveground exposure, and that this may affect leaching performance.

13.2.2 OECD Draft Guideline II: “Laboratory Method for Wooden Commodities that are not Covered and are in Contact with Fresh Water or Seawater”

This protocol is meant to simulate the exposure of wood in direct contact with water. Wood samples for this test are treated to specified fresh water or seawater contact retentions and may be treated in the laboratory or cut from commercially treated lumber. The method uses the same end-sealed samples as for Guideline I, but samples are exposed to continuous immersion in 500 ml of water, with water exchanged after 6 h, 1 d, 2 d, 4 d, 8 d, 15 d, 22 d, and 29 d. The water samples are collected after each water change for analysis and leaching is reported as above. As with Guideline I, flux rates can be applied to the surface area of treated wood structures to estimate potential for environmental contamination by preservative components.

The specimen geometry and end-sealing result in lower initial leaching rates compared to AWPA E11, but since the leaching time is more than doubled, the cumulative amounts are similar (Table 13.4 vs. Table 13.1). The draft standard also permits other specimen sizes and in Figure 13.5 and Table 13.4, leaching rates are compared for the above testing protocol, except that 38 × 140 × 300 mm (2 × 6 × 12 in.) end-sealed specimens are tested, using a similar surface-area-to-water volume as for the standard procedure. As expected, the % leaching is substantially

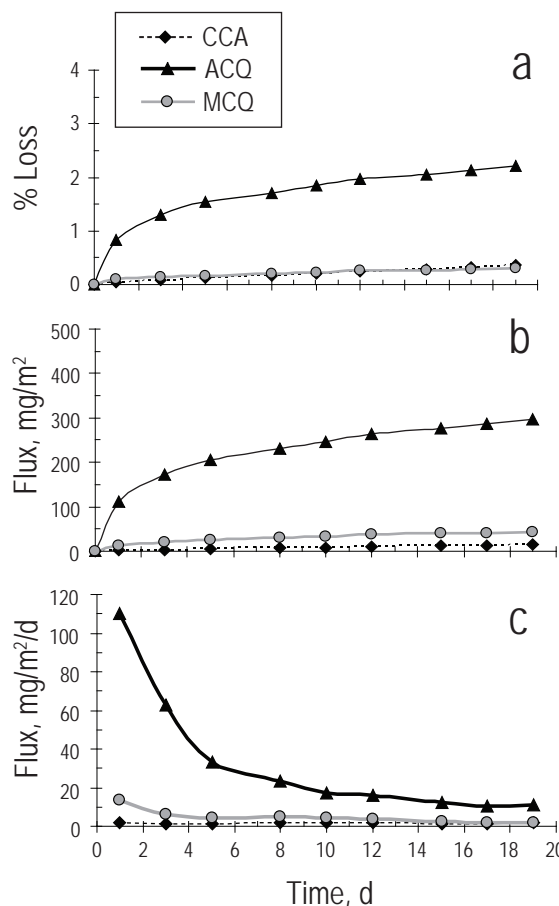


Figure 13.4 Copper leaching data produced using OECD Guideline I of specimens treated with CCA, ACQ, or MCQ to a retention of approximately 4.0 kg/m³ and expressed as (a) % loss; (b) flux in mg/m²; and (c) flux rate in mg/m²/d (adapted from Cooper and Ung 2008).

Table 13.3 Comparison of OECD I leaching characteristics for southern pine samples treated with three preservative systems^a (Cooper and Ung 2008).

Preservative and component	Total retention (kg/m ²)	Cumulative		Flux rate (mg/m ² /d)	
		% leached	flux (mg/m ²)	Initial	Final
MCQ-Cu	4.0	0.30 (0.08)	43.4 (10.6)	13.8 (4.2)	2.1 (0.57)
	6.4	0.24 (0.02)	58 (5.9)	16.0 (1.0)	2.7 (0.50)
CCA-Cu	4.0	0.36 (0.03)	15 (1.1)	2.0 (0.31)	1.8 (0.06)
	6.4	0.42 (0.03)	29 (1.3)	3.5 (0.95)	2.4 (0.04)
CCA-Cr	4.0	0.24 (0.02)	15.1 (1.1)	6.3 (1.29)	0.40 (0.08)
	6.4	0.28 (0.03)	31 (3.5)	15.7 (2.79)	1.00 (0.06)
CCA-As	4.0	0.38 (0.02)	21 (1.3)	4.7 (0.64)	1.60 (0.18)
	6.4	0.29 (0.03)	29 (2.8)	5.2 (0.14)	2.40 (0.38)
ACQ-Cu	4.0	2.21 (0.20)	297 (7.6)	110 (11.1)	11.3 (1.36)
	6.4	3.45 (0.03)	585 (13.4)	267 (9.2)	21.4 (1.08)

a. Values represent means of 3 replicates. Standard deviations are in parentheses.

Table 13.4 Effect of wood orientation on OECD II leaching characteristics for southern pine samples treated with CCA, ACQ, or MCQ to 6.4 kg/m³ Group I: 15 × 25 × 50 mm specimens, Group II: 38 × 140 × 300 mm specimens^a (Cooper and Ung unpublished data).

Preservative, component, and group	Grain pattern	Cumulative		Flux rate (mg/m ² /d)	
		% leached	Flux (mg/m ²)	Initial	Final
MCQ-Cu-I	Flat sawn	1.04 (0.03)	250 (8.1)	152 (10.8)	1.4 (0.23)
MCQ-Cu-I	Quarter sawn	0.41 (0.02)	98 (4.8)	43 (0.8)	1.0 (0.41)
MCQ-Cu-II	Flat sawn	0.18 (0.046)	166 (40.6)	31 (11.4)	3.5 (0.71)
CCA-Cu-I	Flat sawn	1.10 (0.03)	75 (3.4)	9.3 (0.83)	0.7 (0.16)
CCA-Cu-I	Quarter sawn	1.05 (0.06)	57 (3.5)	7.0 (0.76)	0.8 (0.17)
CCA-Cu-II	Flat sawn	0.18 (0.004)	51 (2.63)	6.9 (3.31)	1.4 (0.09)
CCA-Cr-I	Flat sawn	0.98 (0.10)	111 (10.9)	162 (27)	0.30 (0.06)
CCA-Cr-I	Quarter sawn	1.13 (0.29)	100 (24.5)	143 (56)	0.30 (0.06)
CCA-Cr-II	Flat sawn	0.07 (0.01)	24 (4.1)	3.5 (2.07)	0.6 (0.04)
CCA-As-I	Flat sawn	2.41 (0.07)	240 (10.7)	19.6 (2.8)	8.6 (0.93)
CCA-As-I	Quarter sawn	2.17 (0.11)	170 (6.6)	15.5 (4.0)	3.6 (0.46)
CCA-As-II	Flat sawn	0.40 (0.03)	123 (13.8)	7.2 (2.02)	3.8 (0.34)
ACQ-Cu-I	Flat sawn	11.4 (0.44)	1925 (44.4)	687 (51)	26.1 (3.54)
ACQ-Cu-I	Quarter sawn	9.87 (1.00)	1723 (141)	850 (169)	25.9 (3.51)
ACQ-Cu-II	Flat sawn	2.91 (0.37)	2416 (404)	378 (191)	62 (1.2)

a. Values represent means of 3 replicates. Standard deviations are in parentheses.

lower for the larger dimension samples; however, the flux data are also significantly different in several cases and the effect is not consistent among preservatives (Table 13.4). Some of these effects may be explained by slower water absorption by the larger specimens and their greater reservoir of soluble materials, but more study of these effects is warranted.

The rectangular cross section of these specimens can be an issue for Guideline II, Guideline I, and EN 84 tests when preparing specimens for the tests, since transverse leaching is anisotropic. Higher leaching rates tend to occur in the radial than the tangential direction. This can be seen in Table 13.4, comparing leaching from specimens with the thickness in the radial direction (flat sawn) or in the tangential direction (quarter sawn). Thus, leaching protocols should specify the grain orientation when specimens are not of equal thickness and width.

13.2.3 Dynamic testing of commodity size samples

Brooks describes a leaching procedure using commodity size products with dynamic flow of water as a procedure

for obtaining more realistic preservative emissions data for wood in aquatic use. This method and comparative results are presented in detail in Chapter 10.

13.2.4 Synthetic Precipitation Leaching Procedure (SPLP) (USEPA Method 1312)

The SPLP (USEPA 2002a) is an agitated extraction method developed to evaluate the potential impact of acidic precipitation on mobility of contaminants from contaminated soils into groundwater. Simulated acid rain (solution of nitric and sulfuric acids of pH 4.2 or 5 depending on the soil source) is used as the extraction fluid. The procedure requires particle size reduction to less than 9.5 mm. Although developed to evaluate soils, the procedure is often applied to wood samples, as a measure of effects of acid rain on leaching (e.g., Townsend et al. 2004). The small-dimension wood samples contain a high proportion of end grain that results in abnormally high leaching. The use of mineral acids to acidify the leaching solution is more appropriate to simulate acid rain instead of weak or complexing acids (e.g., Cooper 1991, Townsend et al. 2004), although the moderate buffering capacity of the wood

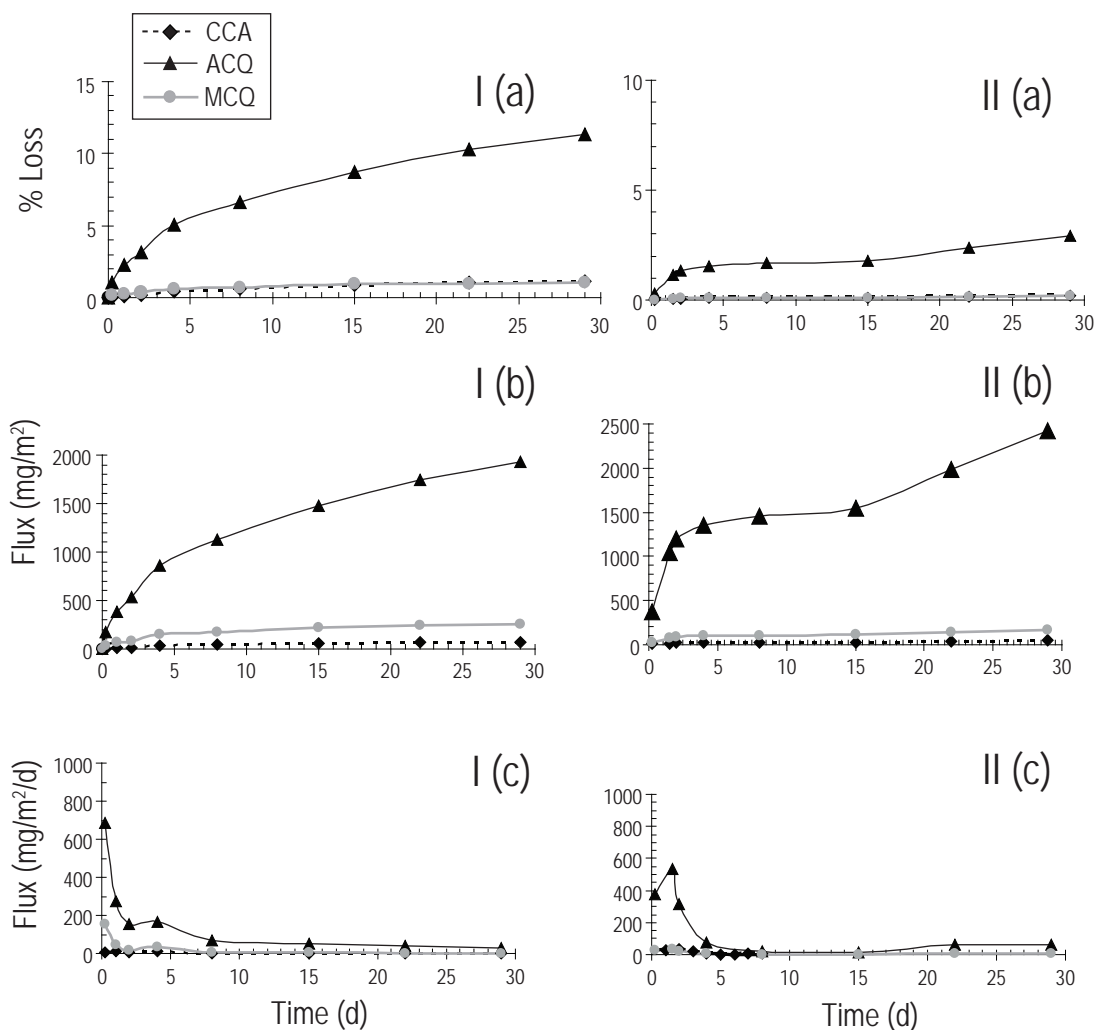


Figure 13.5 Copper leaching by OECD Guideline II for southern pine specimens treated with CCA, ACQ, or MCQ to a retention of approximately 6.4 kg/m^3 and expressed as (a) % loss; (b) flux in mg/m^2 (c) flux rate in $\text{mg/m}^2/\text{d}$. Group I: $15 \times 25 \times 50 \text{ mm}$ specimens, Group II: $38 \times 140 \times 300 \text{ mm}$ specimens (adapted from Cooper and Ung 2008).

will quickly neutralize the acid. As a result, leaching does not differ substantially from that obtained by leaching the same material with normal water.

13.2.5 Toxicity Characteristic Leaching Procedure (TCLP) (USEPA Method 1311)

The TCLP (USEPA 2002b) test is designed to simulate conditions in an unlined sanitary landfill. It is an agitated extraction test whereby material, reduced to pass through a 9.5 mm sieve is tumbled with acetic acid solution at pH 4.95 (20 parts liquid to 1 part solid). The use of relatively concentrated weak acid ensures that the wood sample

will not buffer the extracting solution appreciably and the solution will remain acidic throughout the test. Since weak organic acids are generated during decomposition of organic matter in landfills, this is a reasonable extractant for this method.

The TCLP was developed to allow the classification of wastes as hazardous or nonhazardous based on whether the leachate exceeds the allowable concentration, set at 1% of the drinking water standard for the component of interest. If the TCLP extract contains any one of the listed constituents in an amount equal to or greater than the specified concentrations, the waste possesses the char-

acteristic of toxicity and may be classed as a hazardous waste. Other jurisdictions have adopted this methodology, but with different chemicals included or different limits specified depending on their drinking water standards. As an example, current EPA TCLP limits for a number of wood preservative components are similar to those promulgated by the Province of Ontario, Canada, Ministry of the Environment (OMOE), but differ for arsenic and pentachlorophenol (Table 13.5). TCLP tests on preservative-treated wood generally show that TCLP values are below the specified levels in all jurisdictions for pentachlorophenol (EPRI 1990), creosote components (Goodrich-Mahoney 1992), dioxins/furans, hexachlorobenzene, and chromium (Ung and Cooper 2003; Figure 13.6 and 13.7), but are exceeded in some specific examples for arsenic from CCA-treated wood (e.g., Figure 13.7).

Preservative-treated wood has generally been exempt from consideration of leachate toxicity, but occasionally this exemption is questioned. The TCLP procedure overestimates the potential impacts of treated wood in landfills, since it requires that the wood be ground to pass a 9.5-mm screen for the test, on the assumption that the material may break down physically in the landfill. Wood generally maintains its size and geometry in a landfill and therefore presents a much lower surface area for leaching per unit mass or volume of wood.

13.3 NON-STANDARD TESTING METHODOLOGIES

Many other procedures have been employed to evaluate leaching performance, usually in an attempt to better simulate natural leaching conditions. Brooks (see Chapter 7) provides rationalization for the use of flowing water as

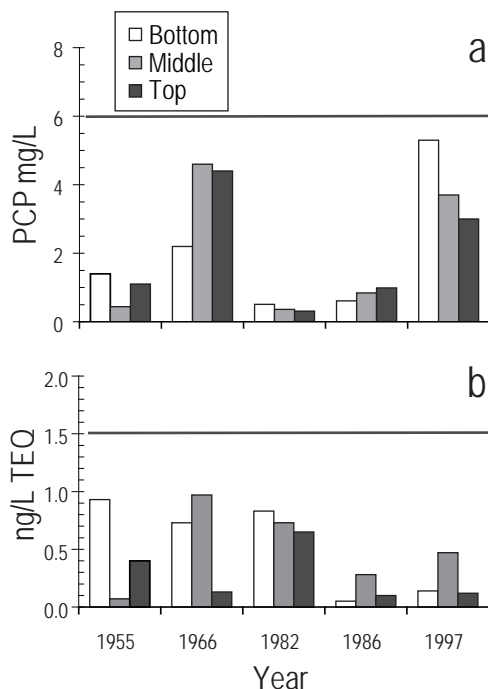


Figure 13.6 TCLP Leachate concentrations for (a) pentachlorophenol (b) dioxins/Furans TEQ from different locations of different vintage red pine poles treated with pentachlorophenol in oil (Ung and Cooper 2003).

a leaching source on full dimension specimens and describes in detail the test methodology. Lebow et al. (2003) showed that ultraviolet light can be an important factor in loss of preservatives from wood exposed out-of-doors and recommend that UV exposure be considered in development of leaching protocols. They have also suggested that many laboratory tests do not simulate the water uptake and loss that occurs in natural exposures, which may affect leaching results (Lebow et al. 2008).

Table 13.5 Comparison of TCLP limits in two jurisdictions: USA (USEPA 2002a) and Ontario Canada (OMOE 2000).

Preservative component	EPA Method 1311	OMOE Ontario Regulation 347 amended by O.Reg. 558/00
	TCLP limit	TCLP limit
Chromium	5 mg/L (ppm)	5 mg/L (ppm)
Arsenic	5 mg/L (ppm)	2.5 mg/L (ppm)
Pentachlorophenol	100 mg/L (ppm)	6 mg/L (ppm)
Hexachlorobenzene (penta)	0.13 mg/L (ppm)	0.13 mg/L (ppm)
Dioxins/Furans (penta)	n/a	1.5 ng/L (ppt) TEQ ^a
Benzo[α]pyrene (creosote)	n/a	0.001 mg/L (ppm)

a. TEQ Toxicity equivalence to 2,3,7,8 tetrachloro-dibenzo-p-dioxin.

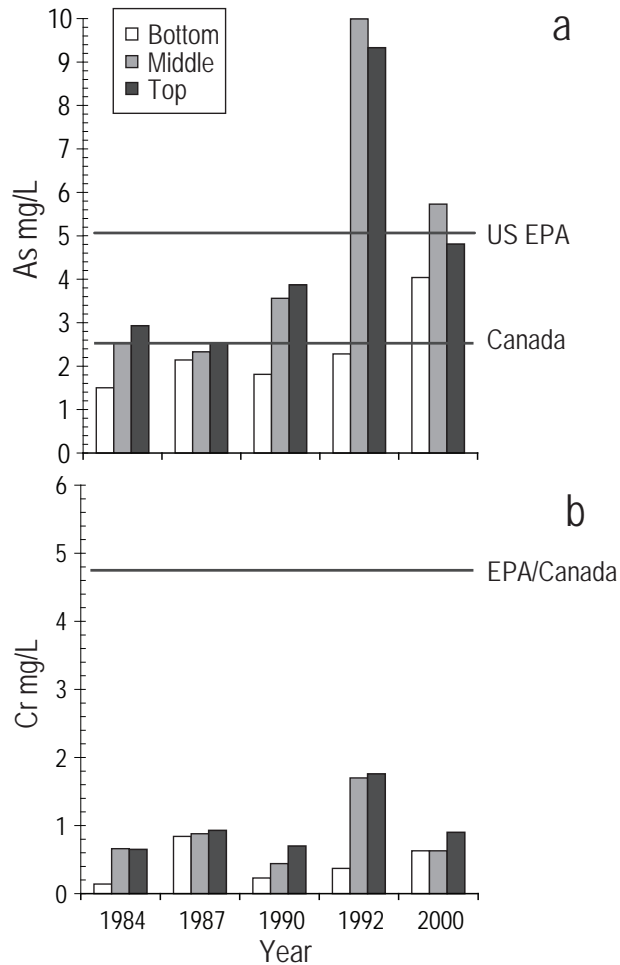


Figure 13.7 TCLP Leachate concentrations for (a) arsenic and (b) chromium from different locations along the length of different age red pine poles treated with CCA-C (Ung and Cooper 2003). Horizontal lines represent specified maximum TCLP concentrations.

13.4 FIELD EXPOSURE TESTING

13.4.1 Introduction

As with treated-wood decay-test evaluations, natural exposure of reasonable size specimens provides the best estimate of the performance of treated wood in terms of resistance to fungal decay, insect attack, or leaching resistance. There are a number of obvious advantages and disadvantages to these types of leaching tests:

13.4.1.1 Advantages

1. They provide the most realistic indication of short-term and long-term leaching rates and amounts.

2. They allow testing of full dimension samples, thereby incorporating effects of long diffusion distances, preservative gradients, shell treatments, and checking in service.
3. They incorporate most of the factors that are hypothesized to contribute to leaching, but are difficult to simulate in small-scale tests such as UV exposure, wetting and drying, freezing and thawing, microbial effects, orientation and exposure of the samples, and development of surface checks.
4. They allow evaluation of other factors such as the effects of water repellents, stains, and other coatings on leaching.
5. Leaching results can be presented as % loss; flux and flux rate and can be presented against cumulative time or cumulative rainfall.

13.4.1.2 Disadvantages

1. Exposure conditions are specific to the actual testing site and to specific weather conditions during the test, so reproducibility among investigators is lower than for the standard laboratory tests; very different results are obtained depending on the time of year when the test is started.
2. Because of the large specimen size, replicate samples tend to be more variable and exposure conditions are highly variable, resulting in high variability in the leaching results.
3. Testing requires a long-term testing commitment, although short-term preliminary results may also provide valuable information.
4. It is costly to maintain the test.

A variety of studies have used short- and long-term natural weathering exposure of treated wood exposed in different geographic locations and with different orientations (horizontal, inclined, and vertical) (Yamamoto et al. 1999; Lebow et al. 2000; Kennedy and Collins 2001; Taylor and Cooper 2001, 2003, 2005; Chung and Ruddick 2004; Schotnecht et al. 2004; Stefanovic and Cooper 2006; Garcia-Valcarcel and Tadeo 2006). In the figures and table below, natural exposure leaching results are compared with different laboratory testing protocols for copper leaching from CCA, ACQ and MCQ treated wood (Cooper and Ung 2008, 2009).

13.4.2 Horizontally oriented samples

Short, end-sealed 89 × 140 mm (2 × 6 in.) samples were placed horizontally (300 mm long) or vertically (600 mm long) over leachate collection trays and exposed to natural leaching over two seasons (excluding winter) in Toronto, Ontario, Canada (Cooper and Ung 2008, 2009). The leaching per rain event was determined from the amounts and concentrations of leachate collected. An example of the high variability among replicate samples is seen in Figure 13.8. In this case, replicates 1 and 3 developed extensive checking in year 2, while replicate 2 did not.

Natural leaching exposures showed less difference among the preservative types than in the accelerated water exposure tests. For example, copper leaching from CCA-treated wood was proportionally higher for this natural exposure. MCQ had the lowest % leaching, followed by CCA. ACQ had the highest value when the leaching values are expressed as cumulative flux values (mg Cu/m²), while the corresponding value for MCQ was about 4 times lower.

Even after two leaching seasons, the % leaching (Figure 13.9a and 13.9b and Table 13.6) was relatively low compared to the AWPA E11 and OECD Guideline II tests. The leaching rate actually increased in year 2 as a result of the higher rainfall and the development of splits in the test samples. Amount of leaching was more-or-less linearly related to the amount of rainfall as observed by Hasana et al. (2008) The flux values were relatively high, and cumulative values in some cases exceeded those from the small-scale leaching tests due to the limited reservoir of soluble components in the small samples. In comparison to the OECD Guideline I test, designed to simulate losses in above ground applications, the OECD I cumulative flux values were exceeded by those for natural weathering exposure after only 20–100 d, depending on the preservative and component.

Flux rate is more difficult to estimate and interpret for the natural weathering exposure. One observation from these tests is that the average daily flux rate over the interval between two succeeding rain events or water collections does not steadily decline as it does for small-scale controlled tests (Figure 13.10) Instead, the rate spikes at different times depending on the rain amount and time between collections. Presenting the results on the basis of amount of rainfall (mg/m²/cm rainfall) produces a more typical flux rate curve, but with anomalous peaks and

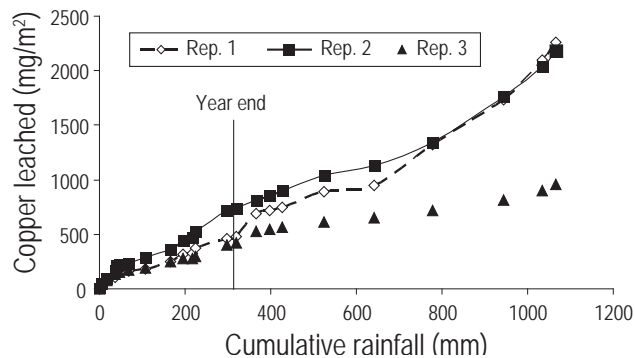


Figure 13.8 Example of variation among replicate samples for natural exposure leaching of 38 × 140mm ACQ-treated southern pine samples (4.0 kg/m³) exposed in Toronto, Canada, over two seasons (unpublished results by author).

increases resulting from conditions that enhance leaching, such as warm weather, long, low-intensity rains, and specimen checking (Figure 13.9f). Clearly, the emission rate will be higher during the actual rain event; but, while it is feasible to measure durations of rain events (e.g., Taylor and Cooper 2005), this is not commonly done. Presenting flux rate as values per rain event or collection provides a better estimate of the short-term releases relevant to “acute” risks (Figure 13.9g).

13.4.3 Vertical oriented samples

Although the vertically oriented samples are exposed to weather on all surfaces except the end grain, rain interception and board wetting is more limited and highly dependent on wind speed and direction. Vertically oriented samples (Figure 13.10) had much lower copper leaching than horizontally oriented samples (Figure 13.9) as also observed by Garcia-Valcarcel and Tadeo (2006). The leaching characteristics among the preservative types were also different than observed for all other tests. Copper leaching from CCA- and to a lesser extent MCQ- treated wood was proportionally much higher than observed for the laboratory testing and the horizontal exposure. Variables that distinguish this method from horizontal exposure and that may contribute to these differences include sporadic wind-dependent exposure of all faces to rain, less water uptake and faster and more complete drying between rain events, low water volume due to lower exposure, more absorption in the samples as the water runs down the specimen, and less check development.

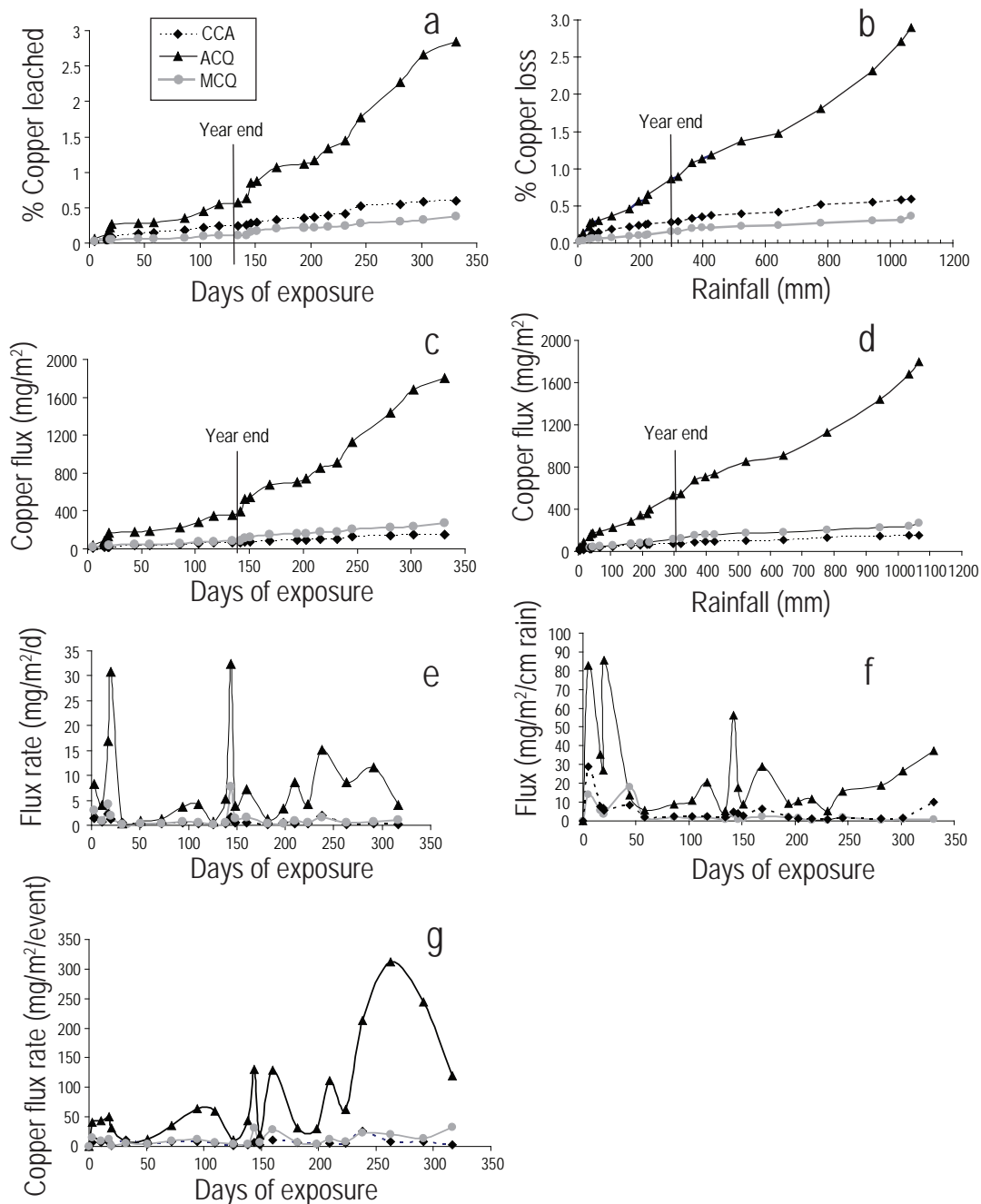


Figure 13.9 Cumulative copper losses from CCA-, ACQ-, and MCQ-treated end-sealed 2 by 6 inch boards expressed as % leaching vs. time (a), rainfall amount (b), and flux in mg/m² vs. time (c), amount of rainfall (d), or flux rate over the collection interval (e) per cm rainfall (f) and per rain event (g). Samples were exposed horizontally to natural weathering in Toronto, Canada (Cooper and Ung 2009).

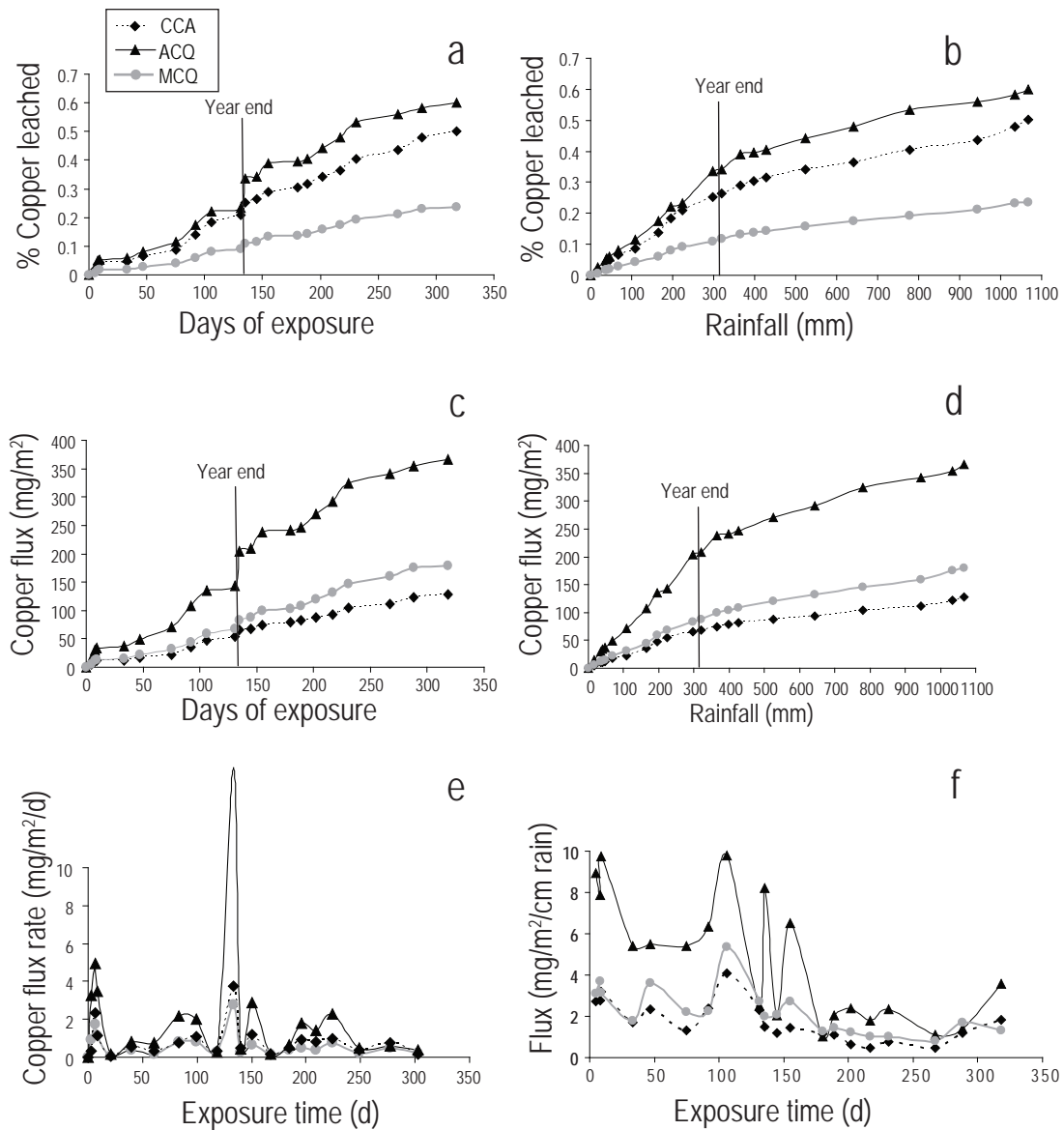


Figure 13.10 Cumulative copper leaching from CCA-, ACQ-, or MCQ-treated southern pine lumber exposed vertically to natural weathering in Toronto, Canada, and expressed as % copper loss vs. time (a) and rainfall amount (b) flux in mg/m² vs. time (c) and amount of rainfall (d) and flux rate over the collection interval (e) and per cm rainfall (f) (Cooper and Ung 2009).

13.4.4 Mulch leaching

The potential for treated wood from construction and demolition waste to be ground into mulch for landscaping has created interest in understanding the potential for leaching from these materials. The significance of leaching from mulch depends on how finely the wood is ground. Leaching of mulch samples was evaluated under the same natural exposure conditions as above (Cooper and Ung 2008).

Mulch samples were prepared from weathered ACQ-treated wood and freshly treated MCQ wood. About 50 mm depth (2 in.) of mulch was placed in perforated plastic

trays either with or without 25 mm of soil (either sand or organic topsoil). The trays were placed out-of-doors over containers to collect rainwater that penetrated through the mulch; water was collected and analyzed after each significant rain event.

Sand retained about 50% of the copper released from ACQ-treated mulch and about 70% of the copper from MCQ-treated mulch. The top soil layer beneath the mulch significantly retained the Cu leached from the mulch contained in both preservative systems, preventing at least 95% of the Cu in the leachate from passing through (Figure 13.11).

Table 13.6 Leaching characteristics for southern pine samples treated with CCA, ACQ or MCQ to a retention of 4.0 kg/m³ and exposed to natural weathering in Toronto Canada during two seasons (excluding winter) in various methodologies^a (Adapted from Cooper and Ung 2008, 2009).

Preservative and component	Sample configuration	Cumulative		
		% leached	Flux (mg/m ²)	Maximum observed flux rate (mg/m ² /day)
MCQ-Cu	Horizontal	0.37 (0.11)	271 (57)	7.8 (1.13)
	Vertical	0.24 (0.08)	179 (36.7)	3.72 (0.17)
	Mulch	1.78 (0.34)	—	—
	Mulch on sand	0.58 (0.08)	—	—
	Mulch on top soil	0.25 (0.11)	—	—
CCA-Cu	Horizontal	0.59 (0.19)	153 (30)	1.97 (0.73)
	Vertical	0.50 (0.08)	128 (12)	2.78 (0.31)
CCA-Cr	Horizontal	0.24 (0.05)	64 (9.9)	0.98 (0.24)
	Vertical	0.26 (0.03)	67 (1.7)	1.30 (0.29)
CCA-As	Horizontal	2.05 (0.33)	485 (24)	6.44 (0.61)
	Vertical	0.72 (0.10)	165 (26)	2.68 (0.21)
ACQ-Cu	Horizontal	2.85 (1.53)	1800 (730)	32.4 (13.8)
	Vertical	0.60 (0.31)	366 (99)	15.2 (5.2)
	Mulch	2.60 (0.28)	—	—
	Mulch on sand	1.35 (0.18)	—	—
	Mulch on top soil	0.18 (0.08)	—	—

a. Values represent means of 3 replicates. Standard deviations are in parentheses.

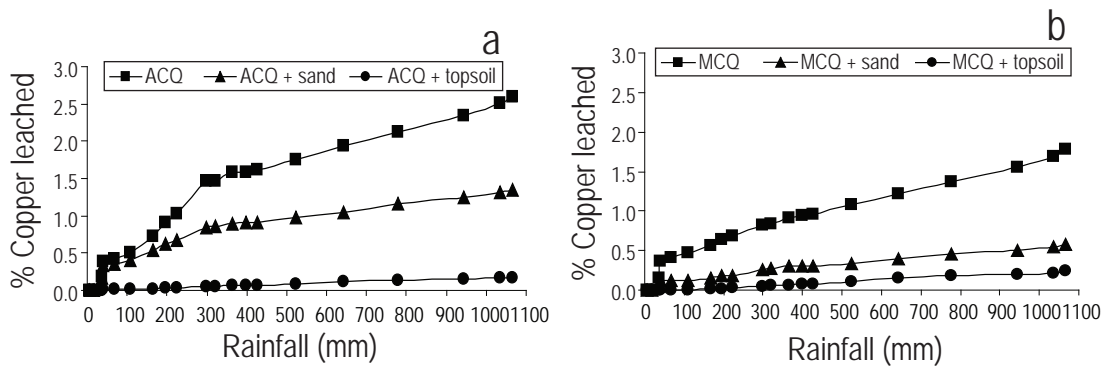


Figure 13.11 Copper losses from ACQ (a) or MCQ (b) treated mulches (4.0 kg/m³) exposed to natural rainfall and the effects of sand or organic topsoil beneath the mulch to trap this copper. Average of three specimens (Cooper and Ung 2008).

13.5 COMPARISON OF LEACHING METHODS

Tables 13.1, 13.2, 13.3, 13.4, and 13.6 compare the copper, chromium, and arsenic (CCA) and copper (MCQ and ACQ) leaching parameters for treated southern pine tested by the different protocols discussed above. In the figures below, the various leaching parameters are presented in

different ways to provide easier comparison and interpretation of results.

The AWPA E11 test method produces much higher leaching losses than all other test methods, especially compared to full-size samples exposed to two seasons of natural weather exposure (Figures 13.12 and 13.13). The end sealing of the OECD method II samples had an obvious effect on reducing the leaching losses for most com-

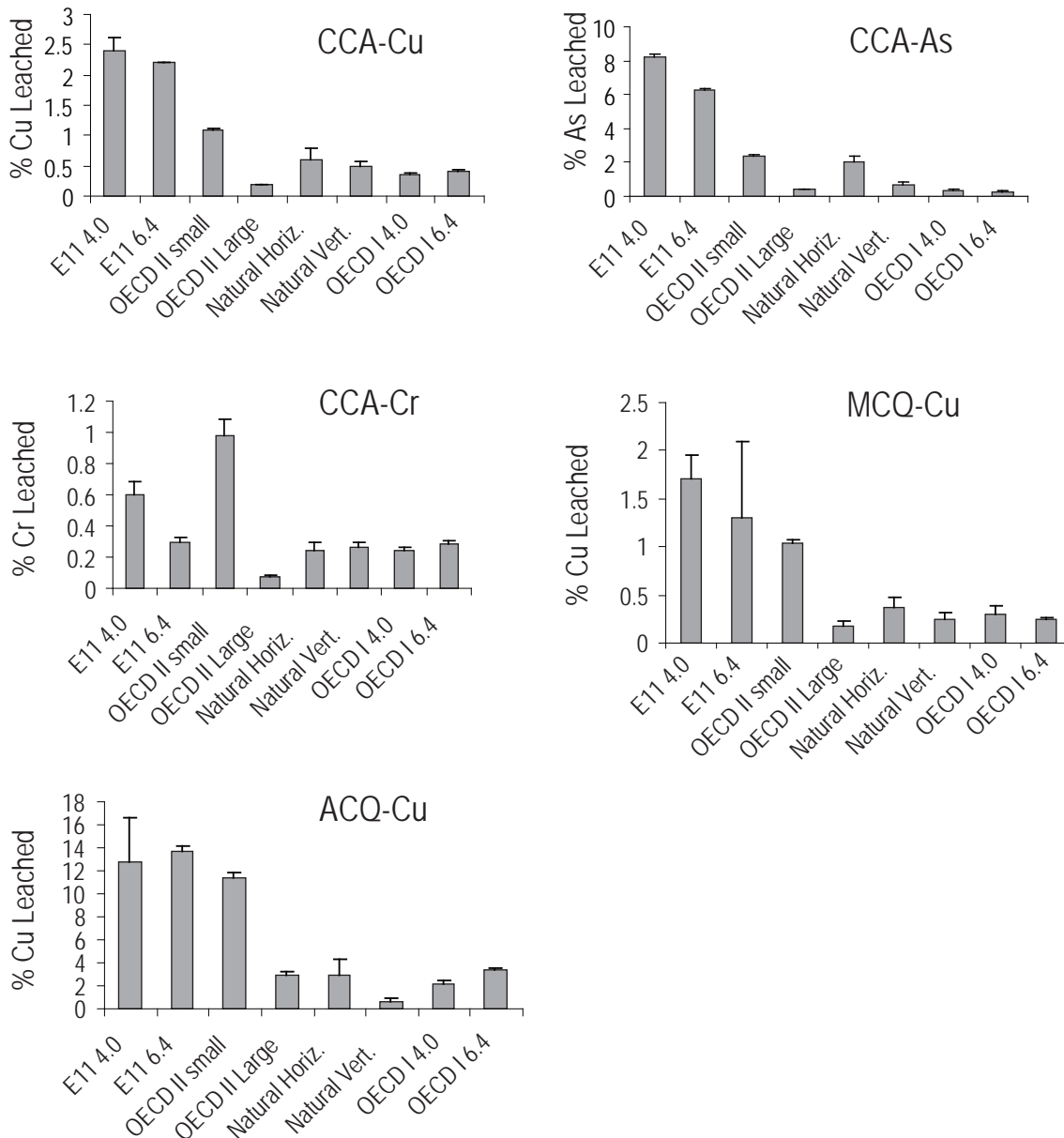


Figure 13.12 Effect of leaching protocol on % preservative component leached from southern pine sapwood treated to a target retention of 4.0 kg/m³ with CCA, ACQ, or MCQ (Cooper and Ung 2009).

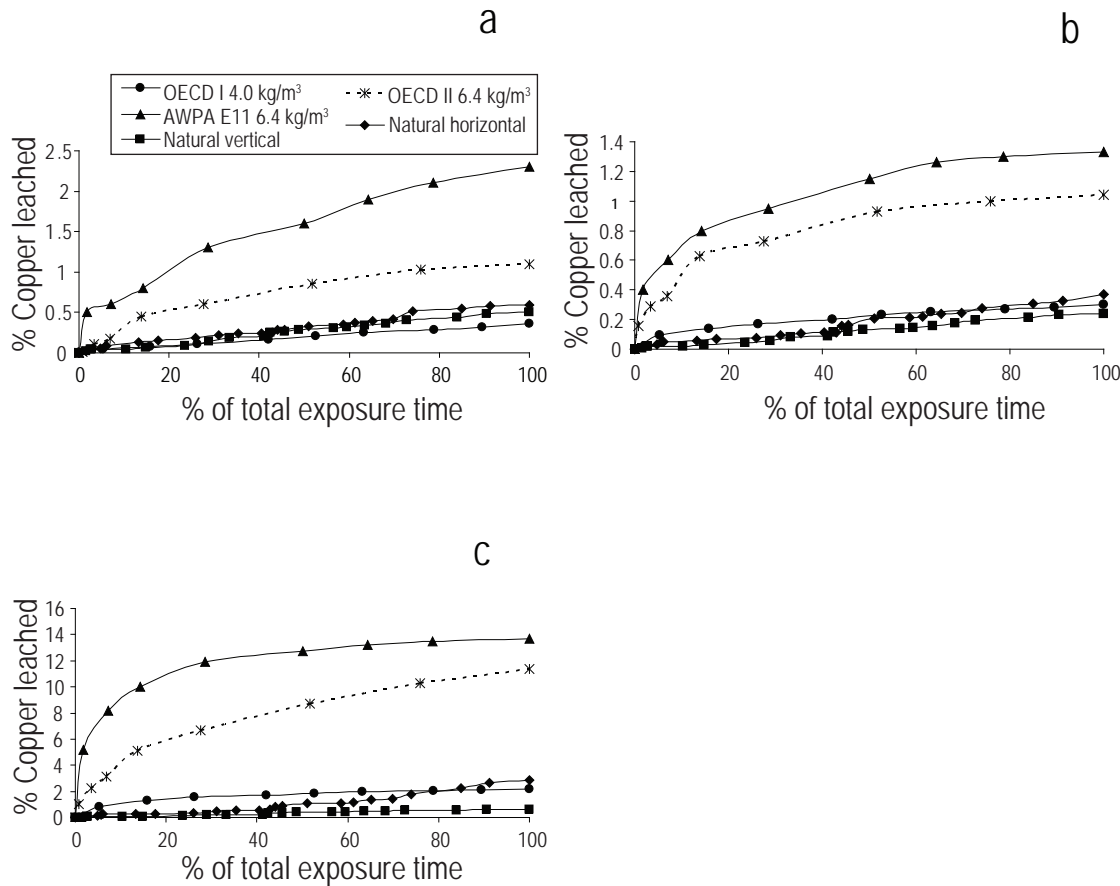


Figure 13.13 Effect of test method on % cumulative copper leaching from southern pine samples treated to a retention of 4.0 kg/m³ with CCA (a), MCQ (b), and ACQ (c) and normalized to % of exposure (Cooper and Ung 2009).

ponents, although the % loss was still much higher than for the full-dimension specimens and for the periodic dipping exposure of the OECD I test. The OECD method I test was generally more representative of long-term % losses of most components, although the test appeared to under-estimate arsenic losses compared to two seasons of natural exposure. Samples for the OECD tests and natural horizontal weathering were end sealed with silicone sealant; we have determined that this results in unexplained, high relative leaching of chromium (Figure 13.12). It is recommended that another sealant such as epoxy resin be used when testing chromium containing preservatives.

Total cumulative leaching flux (Figure 13.14) and leaching fluxes vs. percentage of time in test (Figure 13.15) indicated that while AWP A E11 and OECD II emissions were initially high, they leveled off as the readily soluble components were depleted. Cumulative flux over extended natural weathering leaching may exceed that from the small-scale tests as a result of the larger reservoir of soluble materials (Lebow et al. 2008) and effects of specimen checking. The OECD I protocol resulted in relatively low cumulative emissions that correspond to those in natural horizontal exposure after only a few weeks or months, depending on the formulation. For most preservative components, the variability among replicate samples was

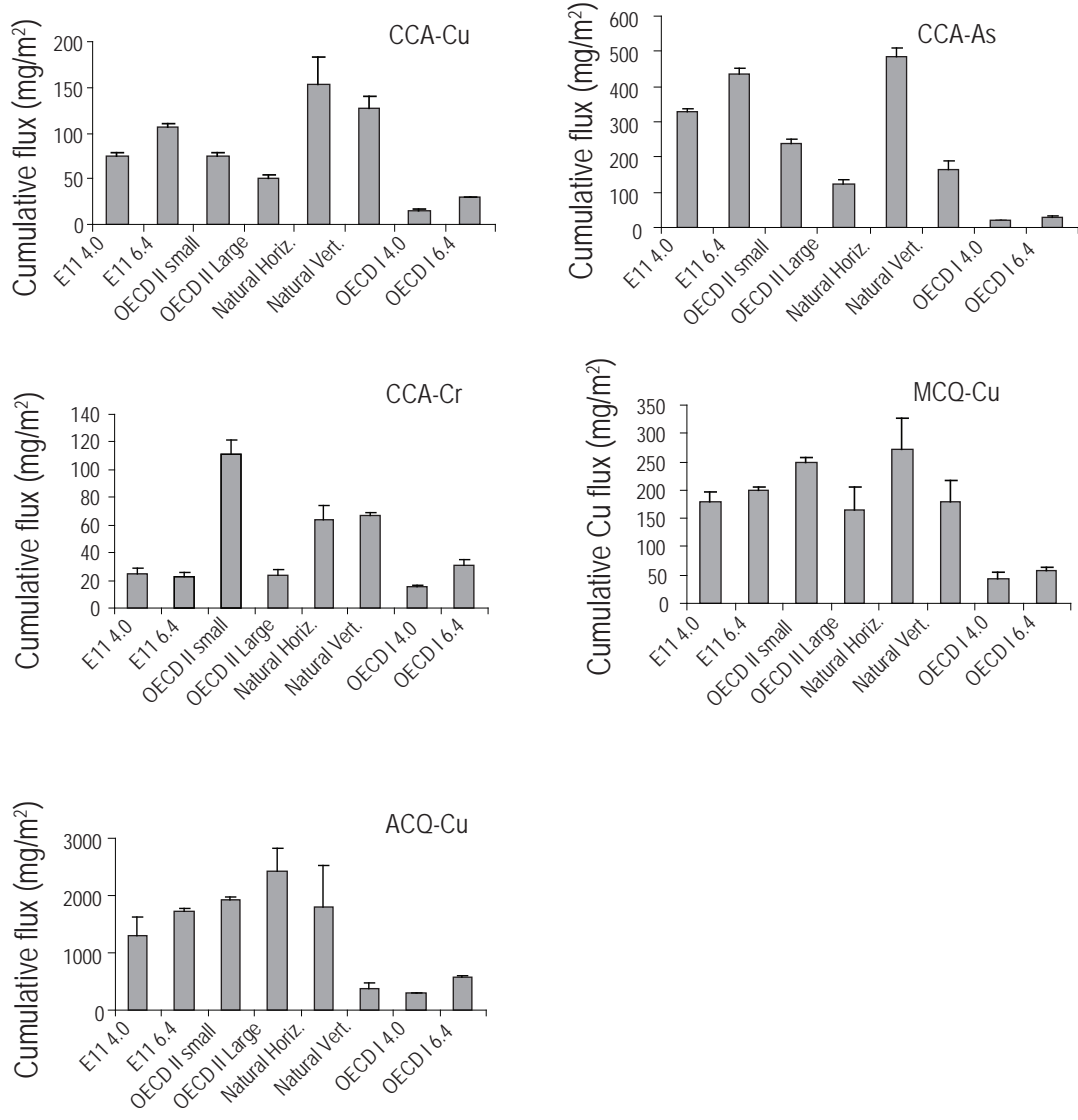


Figure 13.14 Effect of leaching method on cumulative flux (mg/m²) of preservative components leached from southern pine sapwood treated with CCA, ACQ, or MCQ (Cooper and Ung 2009).

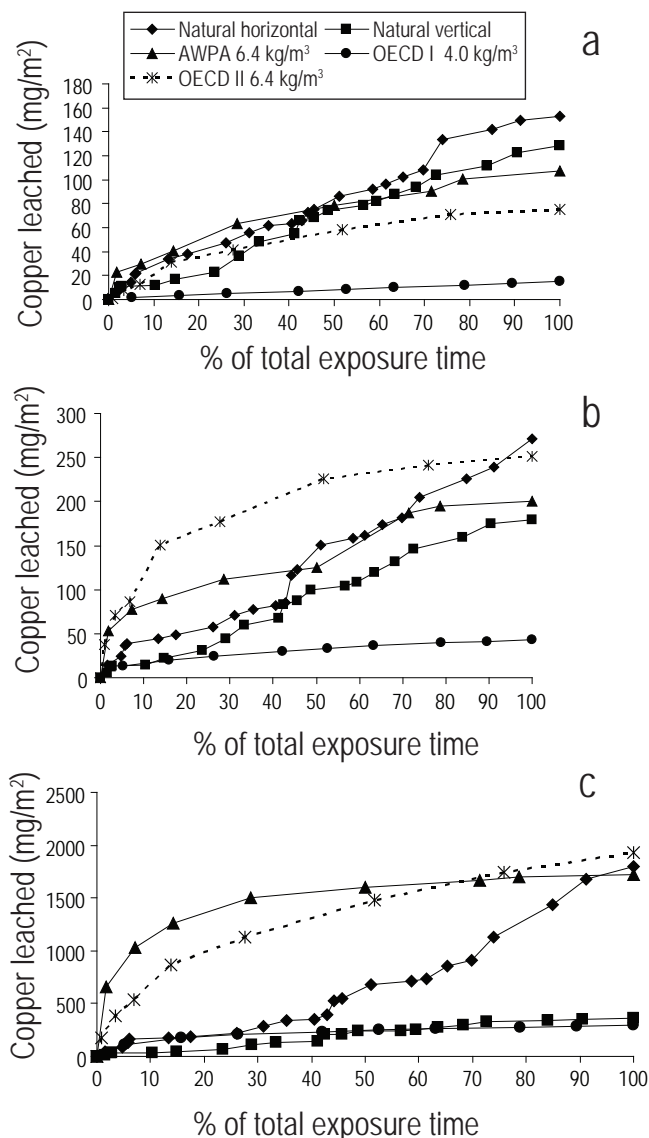


Figure 13.15 Effect of leaching method on copper flux rates from southern pine treated to a target retention of 4.0 kg/m³ with CCA (a), MCQ (b), and ACQ (c) as normalized to % of exposure time in the test (Cooper and Ung 2009).

higher for natural weathering exposure than for the controlled test methods, highlighting one of the advantages of laboratory tests.

The maximum flux rates were invariably much higher for samples subjected to AWWPA Standard E11 than for any of the other protocols (Figure 13.16). In addition to the obvious effects of small specimen size and high amount

of end grain, this is attributed to the short initial sampling time (6 h), which resulted in a higher estimated daily flux than in procedures that used a longer initial sampling period. Despite the emission spikes associated with high rainfall occurring periodically in the natural exposure test (Sections 6.1 and 6.2), all laboratory test protocols gave maximum flux rate values as high as or higher than those observed in the natural weathering trials.

The relationships between copper leaching parameters for ACQ and MCQ treated wood relative to leaching from CCA-treated wood for the end points of all of the tests are shown in Figure 13.17. Generally the % leaching was slightly lower for MCQ and significantly higher for ACQ compared to CCA, while flux in mg/m² was slightly higher for MCQ and much higher for ACQ relative to CCA. The relationship between MCQ and CCA leaching was relatively consistent among all test methods; however, there were some inconsistencies among test methodologies when comparing ACQ to CCA and MCQ, with the natural exposures, and especially vertical exposure showing less difference in the leaching characteristics among the three preservative treatment types.

13.6 SUMMARY AND CONCLUSIONS

1. Small-scale laboratory leaching tests such as AWWPA E11, BSI EN 84, and OECD draft methods are relatively simple, fast, and reproducible, making them acceptable for harmonizing evaluation of different preservatives over different jurisdictions.
2. In general, leaching responses of different preservative systems are proportional for different testing protocols. These small-scale tests give a relative indication of how different preservative systems will respond to leaching forces; however, there are exceptions where some systems perform relatively better in laboratory tests than in field leaching tests, suggesting that some full-scale leaching tests should be conducted to complement accelerated leaching tests.
3. In absolute terms, small-scale accelerated test methods are not usually representative of natural leaching of full size products.

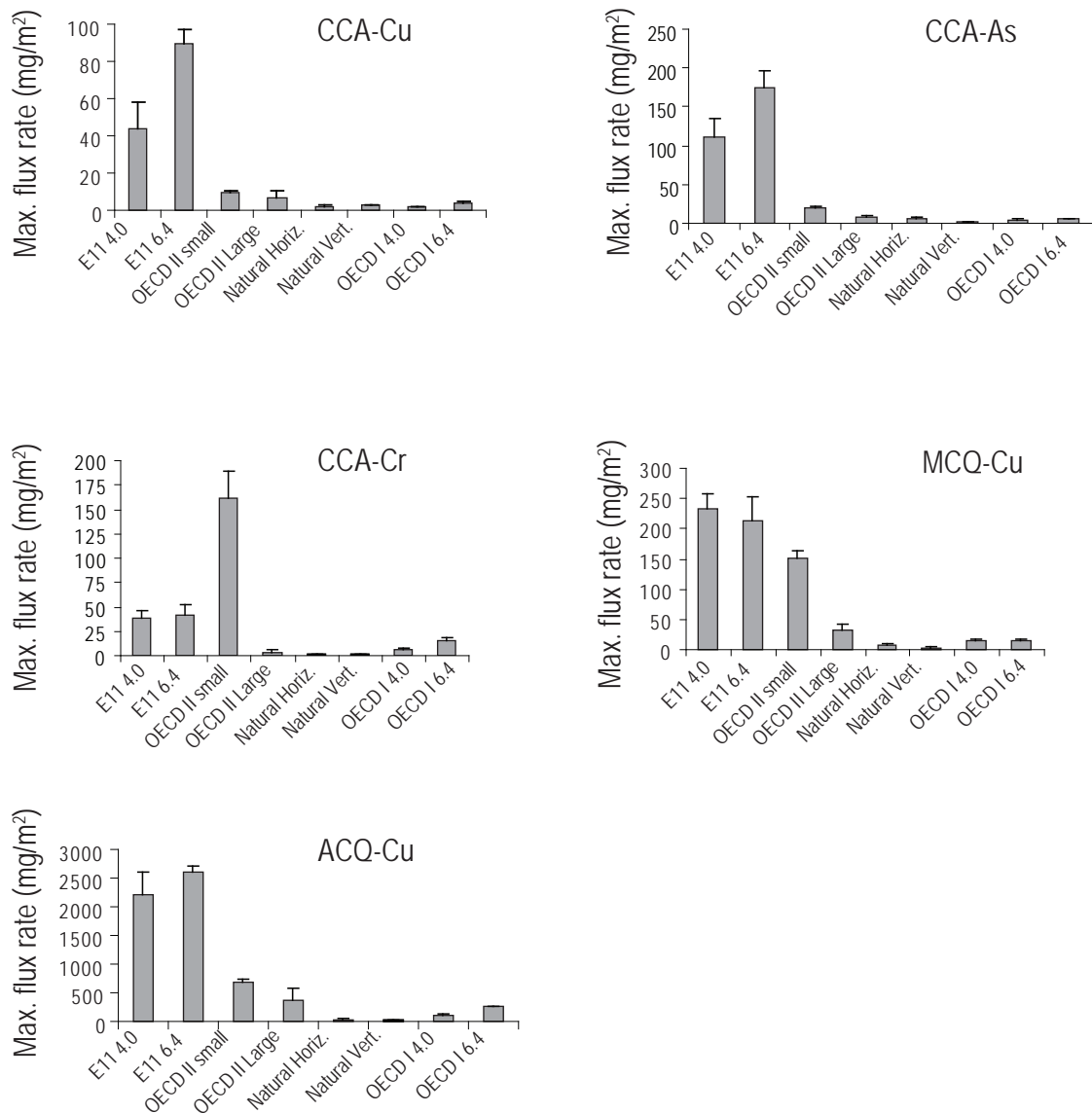


Figure 13.16 Effect of leaching protocol on maximum flux rate ($\text{mg}/\text{m}^2/\text{d}$) of preservative component leached from southern pine sapwood samples treated to a retention of $4.0 \text{ kg}/\text{m}^3$ with CCA, ACQ, or MCQ (Cooper and Ung 2009).

- Leaching test methodologies such as AWPA E11 and BSI EN 84, designed to evaluate persistence in wood for efficacy testing, result in unrealistically high initial emission rates and high estimates of % leaching of preservatives. However, over the long run, they may under-estimate cumulative flux values that occur under natural weathering conditions because of the limited amount of soluble materials present in the small volume specimens.
- Leaching procedures developed to assess risk of treated wood under different exposure conditions, such as OECD methods I and II, provide better estimates of leaching parameters in the early stages of natural exposure. Leaching parameters for OECD I, meant to represent aboveground exposures, are consistent with vertically installed wood exposed to natural weathering, but underestimate values for long-term exposure of horizontally installed specimens.

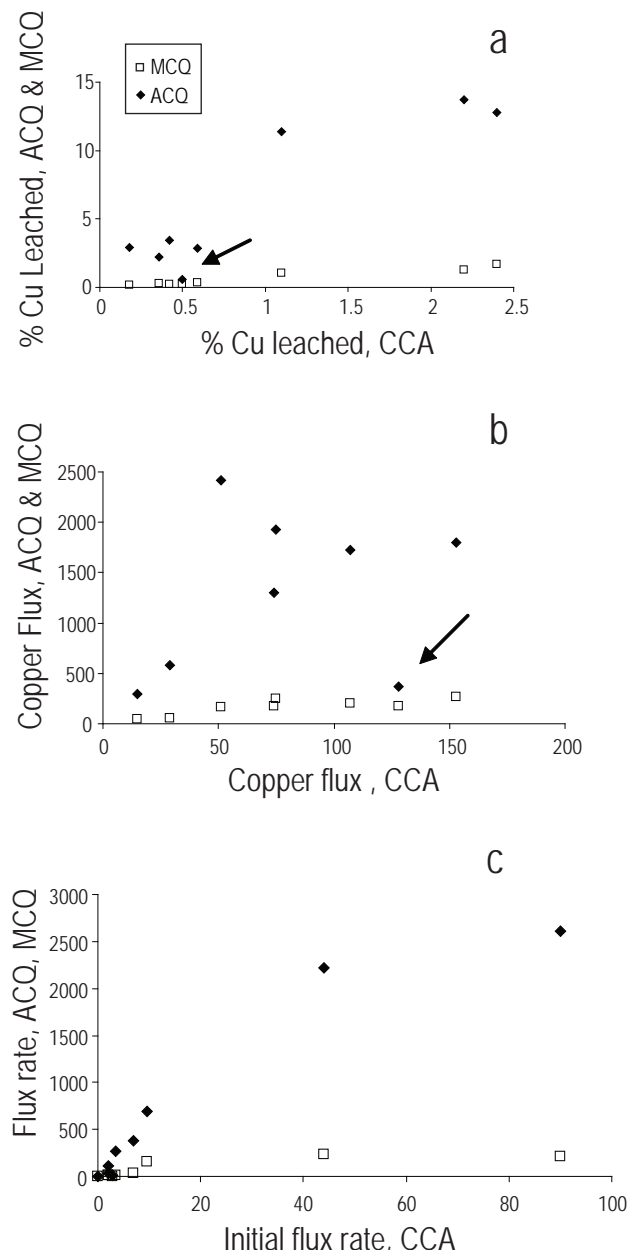


Figure 13.17 Comparison of cumulative leaching % (a) cumulative flux mg/m^2 (b) and initial (or maximum) flux rate $mg/m^2/d$ (c) for the different test results (excluding AWP E20). Note arrows point to vertical exposure results for ACQ (adapted from Ung and Cooper 2008).

4. Natural weathering exposure results in greater variability in leaching results compared to laboratory testing. Amounts of components leached are strongly affected by specimen checking and are proportional to frequency and amount of rain.

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