Harmonization of leaching tests: Leaching behaviour of wood

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The leaching behaviour of preservative treated wood was studied, using three existing leaching methods. This study was part of a European project "Harmonization of Leaching / Extraction Tests " (EU project SMT4-CT96-2066). The leaching methods were: 1. A diffusion tank test of 3 months with medium sized samples of 200 mm length (NEN7345), 2. A laboratory protocol of 3-4 days stirring in water with small samples of about 50 mm length (ENV 1250-2), and 3. A leaching protocol of five days with large samples of one metre length (shower test). Milled wood samples were used for assessment of the total potential of leachable preservative at different pH (pH-stat test). The wood samples were taken from CCA-C treated Norway spruce and Scots pine treated with a Cu-quat. From the results it was concluded that the leaching behaviour of wood is very similar to that of other materials containing organic matter. The pH has a significant effect on leaching of preservatives from wood. The leaching behaviour of the small samples (ENV 1250-2) showed much wider variations in results. It is concluded that the NEN7345 diffusion tank test method, which was developed for inorganic building materials, is also suitable for assessment of the leaching behaviour of preservative treated wood. An adaptation of the acidic pH conditions to neutrality of the leaching water used is recommended.

1 Introdution

Background

The use of leaching test methods is increasing in different areas, ranging from preservative treated wood to contaminated soil, waste, inorganic construction materials to drinking water pipes. These tests are developed in ad-hoc situations at research institutes, in national programs, and in CEN and ISO technical committees. A European Network "Harmonization of Leaching/Extraction Tests" was started in 1995. The first aim of this network was to harmonize approaches in existing leaching tests. The second aim was to form a network of experts and exchange information among different fields and define the problems in specific fields. This is needed to build a common strategy for the use, validation and interpretation of leaching/extraction tests, and finally to formulate recommendations for the implementation of more generally applicable approaches and subjects for further research.

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In 1996 the Network resulted in the start of a European Standardization, Measurement and Testing project. A research programme was started using 13 different leaching substrates and 15 different existing leaching protocols. This article presents the results of the leaching behaviour study on wood. The actual leaching tests were performed in 1997.

Aim of the leaching study on wood

Wood preservatives for application in ground contact most often contain heavy metal components (for example arsenic, copper, chrome) or organic active ingredients (for example Polycyclic Aromatic Hydrocarbons or PAH). During the service life of the preservative treated wood the toxic components may leach from the wood if exposed to rain and fresh surface water. Accumulation of the toxic components in the soil, groundwater or surface water may cause a health risk to the biological organisms in these environments. The aim of this study was to gain more information on the leaching behaviour of preservative treated wood in three existing leaching protocols with relation to:

- relevance of the different tests to practice,
- relation between retention, availability and leachable fraction,
- the volume of wood and water,
- the role of the pH and DOC (Dissolved Organic Carbon) of the water,
- similarities in leaching behaviour with other materials,
- release patterns, and data presentation,
- long term leaching behaviour for in service conditions.

The results of this leaching study on wood will be compared to those of other materials within the European project (van der Sloot, publication in preparation).

Selection of leaching protocols

For this study on preservative treated wood three existing leaching protocols were selected, that were in use in The Netherlands:

- NEN 7345: Leaching characteristics of building and solid waste materials - Leaching tests - Determination of the leaching behaviour of inorganic components from building materials, monolithic waste and stabilized waste materials.
- ENV 1250-2: Wood preservatives - Method for measuring losses of active ingredients from treated timber - Part 2: Laboratory method for obtaining samples for analysis to measure losses by leaching into water or synthetic seawater.
- Shower test : A leaching test for assessing preservative losses from treated timber under simulated open storage conditions (Havermans c.s. 1993).

The tank test protocol (NEN7345) was developed in the Netherlands for assessment of the leaching behaviour of stony (inorganic) building materials, and it is implemented in the Dutch law by the Building Substances Decree. It was decided not to implement this Building Substance Decree to impregnated wood. However, some experimental results using the NEN7345 diffusion test (Van Eetvelde, 1996, TNO unpublished data) showed that this protocol may be applicable to preservative treated wood as well. Some small adaptations related to the different composition and nature of the woody material were to be expected. This leaching protocol has been discussed on a European level for inorganic building materials, but is relatively new in the technical committee on

timber (CEN TC 38). The NEN 7345 leaching protocol was selected in this research to further explore its potential applicability to wood, and to study the correlation with the other protocols.
The leaching protocol of ENV 1250 (beaker test) was developed within the expert working groups of CEN TC 38 "Durability of wood and wood-based products", and was accepted as an experimental leaching method. There has been much discussion within CEN TC 38, as to whether the small size of the wood samples (50 x 25 x 15 mm) was suitable for assessment of the leaching behaviour of treated wood in service conditions. Many experts in this area believed that these sizes are not representative for service conditions and can not be interpreted or correlated to situations in the field. The title of the standard clearly indicates that this protocol is only representative for laboratory conditions. Since this was the only European protocol accepted as a leaching method (and not as a pre-conditioning method, cf. EN 84), it was selected for this study.
The shower test protocol is accepted by the Dutch Ministry of Housing, Physical Planning and Environment as a means of control of Wood impregnation plants, to prevent excessive leaching by rain of freshly preservative treated wood to soil (during storage prior to use in service). Because it is aimed at a particular practical situation, this protocol is deviating much more from the other mentioned leaching protocols. This method was included because of the importance of its existing use in regulations for wood impregnation plants in The Netherlands (BRL 0601,1993).

2 Experimental

Material

The wood samples for the experiments were all taken from industrially preservative treated wood for use in building. The wood preservatives selected were CCA type C, as the most common preservative used in Europe and world wide; and Cu-quat, a relatively new type of wood preservative that is common in The Netherlands as an alternative to CCA. CCA type C is consisting of arsenic, copper and chrome oxides and Cu-quat is a mixture of copper oxide and didecyldimethylammonniumchloride (a quaternary ammoniumchloride, DDAC). The preservative treated wood for the three leaching protocols was selected from the same batch. So, the same wood quality was used in the each of the three test methods performed. One batch of CCA-C treated Norway spruce (total retention of the preservative about 5-6,5 kg/m3) and one batch of Cu-quat treated Scots pine (total retention of the preservative about 5 kg / m3) were selected. The CCA-treated wood was steam-fixed after impregnation, as is most often done in The Netherlands.
The dimension of the wood samples in each of the test protocols was adapted to the dimensions of the largest samples. These were the samples for the shower test with planks of 1metre length and cross sections of about 145 x 45 mm (CCA-C wood) and 90 x 90 mm (Cu-quat wood). All end grains of smaller cut wooden samples were sealed using a polyurethane coating. Demineralized water is used in all leaching methods.

Leaching methods

An overview of relevant test parameters in the three selected leaching protocols is given in table 1.

Table 1: Overview of relevant test parameters.

Test method	Exposure condition	Leaching medium	Ratio volume water/wood	Duration (days)
ENV 1250-2	beakerglas	Demi-water, pH about	5, 3	4
NEN 7345	glass tank	Demi-water, pH about 4	5	64
Shower test	sprinkling water	Demi-water, pH about 6	0,04	5

ENV 1250-2: Wood preservatives - Method for measuring losses of active ingredients from treated timber - Part 2: Laboratory method for obtaining samples for analysis to measure losses by leaching into water or synthetic seawater. According to the standard a set of five fully impregnated blocks of 50 x 25 x 15 mm Scots pine sapwood is leached for four days in 500 ml water, using a magnetic stir. Samples were taken after 1, 3, 7, 15, 31 and 79 cumulative leaching hours, with a 16 hours drying period after the third sampling. Two tests were performed per preservative type.
The dimensions had to be adjusted to the size of the material available for the other test methods. From the same planks that were selected for the above mentioned tank test (NEN7345), the remaining pieces were used to cut smaller blocks of about 10-45 mm thickness and about 45 mm in length. A selection of the wooden parts from the outer area with the highest penetration depth of the preservative was made, as this would compare best to full penetration of sapwood. As it was not possible to cut blocks completely saturated with preservatives, the blocks were partly end-sealed. This was performed to reach the same ratio of treated wood and exposed surface of the treated wood as in the original ENV1250 protocol with fully penetrated sapwood blocks.

NEN 7345: Leaching characteristics of building and solid waste materials - Leaching tests - Determination of the leaching behaviour of inorganic components from building materials, monolithic waste and stabilized waste materials. This method describes a monolithic tank test of three months with leaching samples taken after 0,25, 1, 2, 4, 8, 16, 32 and 64 days. At the start of each sampling period the water in the tank was refreshed and the pH was adjusted to about pH 4, as stated in NEN7345. Two tests were performed per preservative type.
Per preservative two wooden planks of 1 metre were taken and two pieces of 200 mm length were cut from each plank, excluding the outer end grain. Two pieces (one of each plank) were used per tank, containing five times as much water as the volume of the wood.

Shower test: A leaching test for assessing preservative losses from treated timber under simulated open storage conditions (Havermans et al. 1993). In this test a large batch of about 0,5 m^3 of wood was used in each test and exposed to artificial rain for five days. The wood was mounted in a pile with a top surface of about one square meter. Each day 20 litres of artificial rain was sprayed in 1 hour. The leaching water was collected underneath the pile of wood and sampled. Each day a drying period of 23 hours was allowed, including slight heating by infrared lamps for 11 hours at about 28°C. Per preservative only one test was performed, since the large volume of wood is expected to rule out variation due to different retention per plank.

pH stat test: The influence of pH on the leaching of contaminants from various materials is assessed by extracting the material at a liquid/solid ratio of L/S =10 for 24 hours under pH controlled conditions using automated pH control equipment (8 positions) with NaOH or HNO_3 addition. A common pH range covered ranges from pH = 4 to pH = 12 (Comans et al, 1993, van der Sloot et al, 1997). This procedure is now being standardized in CEN TC 292 (CEN TC 292 WG6, 1998). A procedure giving comparable results is the Acid Neutralization Capacity (ANC) procedure, in which several aliquots of the material to be tested are extracted with different amounts of acid or base required to reach a specific end pH. The amount of acid or base needed is derived from and acid/base titration of the material (van der Sloot and Hoede, 1997, CEN TC 292 WG6, 1998). The wood samples in this test were cross sections taken adjacent to one of the samples used for the tank test and beaker test mentioned above. The wood samples were ground to a particle size of less than 1 mm to improve the availability of impregnated components during leaching. Per preservative one test was performed. In this test only the heavy metal components were analysed in the leachates (not the quat component DDAC).

Analysis methods of leachates
All leaching samples collected were analyzed for copper, chrome, arsenic or copper and quat (DDAC type) respectively. The analysis methods used were ICP-AES for copper and chrome, AAS for Arsenic and titrimetric for DDAC.

3 Results and discussion

Results
The results of the released components of the leaching samples expressed in mg/(m^2*day), i.e. copper, chrome, arsenic of the CCA-C treated wood and copper and quat of the Cu-quat treated wood, are summarized in table 2. Cumulative leached components are presented in table 3 and figures 4 and 5. The results of the pH stat tests are graphically presented in figure 1 for CCA-C treated wood and in figure 2 for Cu-quat treated wood. In figure 3 the acid/base titration data illustrating the sensitivity to pH change is given.

Discussion
The problem in comparing the influence of different leaching methods on the results is that the exposure periods are often not the same and that the results are not expressed in the same way. For wood for example, results of analysis are often not related to the exposed wood surface, but are expressed as a percentage of the amount of wood preservative impregnated, in mg per kg wood or in mg per m^3 wood. Therefore in this study the results of the three leaching methods are expressed

*Table 2: Analysis results of components in mg/(m²*day) of leaching samples according to three leaching protocols on CCA-C and Cu-quat treated wood.*

	ENV 1250 beaker test				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**Quat (DDAC)**
0,04	415	48	82	727	482
0,13	152	12	11	148	315
0,29	85	4,8	7,9	215	121
1-1,29	16	1,1	1,5	99	14
1,96	22	1,3	4,4	60	72
3,96	6,7	0,5	2,7	16	10

	NEN 7345 tank test				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**Quat (DDAC)**
0,25	277	13	17	242	4280
1	94	12,3	11	100	360
2	68	8,0	5,0	118	325
4	28	1,9	12	27	80
9	13	0,6	2,3	4,9	12
16	10	0,7	2,2	3,6	9
36	1,3	0,1	0,7	1,7	12
64	0,4	0,1	0,6	0,9	3,9

	Shower test (Havermans c.s.)				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**Quat (DDAC)**
1	29	1,7	2,2	48	117
2	24	1,3	2,0	43	89
3	19	1,0	2,0	31	44
4	16	0,9	1,8	27	54
5	11	0,4	1,6	26	57

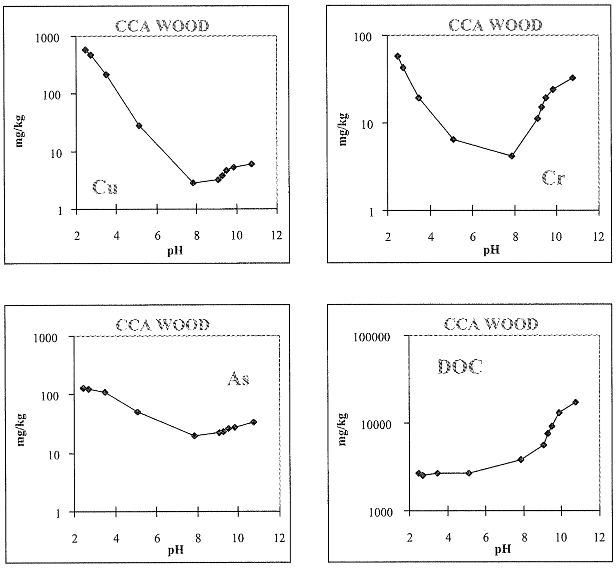

Figure 1: Graphic results of pH stat leaching tests on milled CCA-treated wood.

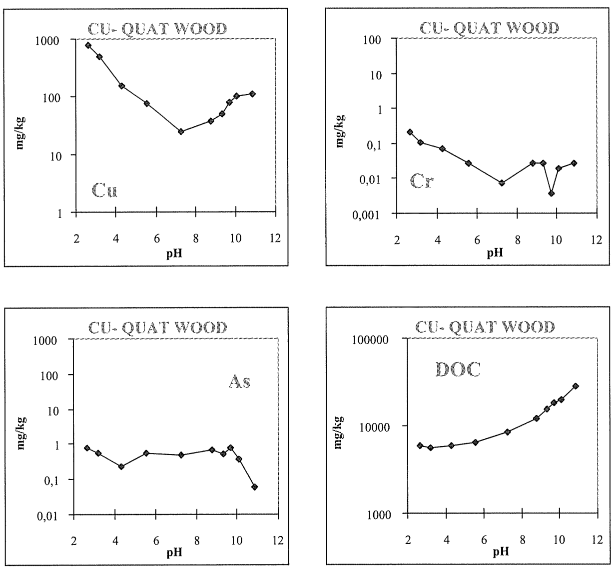

Figure 2: Graphic results of pH static leaching tests on milled wood treated with Cu-Quat.

in cumulative figures in mg/m² on sampling time t=x of the experiment or in mg/(m²*day), see tables 2 and 3. For the beaker test (ENV 1250) and the shower test it should also be noted, that one or four periods of drying are included in the protocol. Finally it should be noted that absolute comparison of the two wood preservatives can not be made, since the wood species and preservative retention are different. The influence of specific material characteristics for wood on leaching rates is not the subject of this study.

The general issues referring to leaching methods are discussed below.

Relevance of the different tests to practice: The ENV1250-2 is a dynamic laboratory scale leaching test with four days exposure of very small samples to stirred water. The interpretation of the ENV 1250-2 leaching data is extremely difficult, since there is no relation to actual dimensions of the wood used in service and the actual leaching conditions in service. Thus, the practical use of these leaching data is very limited. The ENV 1250-2 method could be used as an initial indication of the availability of a wood preservative to leaching.

The NEN 7345 is a tank test using samples with dimensions related to actual practice. Because of the monolithic nature, extra attention should be paid to the representative nature of the selected wood samples. The tank test seems to be the most suitable for prediction of the rate of leaching of components to water from preservative treated wood in service conditions. As can be seen in the release as a function of time the release levels off to a plateau, that is lower than the "availability", which illustrates the relatively strong bonding of preservative in the wood matrix. This can not be observed in the short test procedures. In addition, a tendency of square root of time release can be observed (slope 0.5 in log-log plot of release against time) until the leachable fraction is depleted. Cr does not follow such release behaviour and its leachable portion appears to be depleted rather quickly. This is due to the fast change of the more mobile hexavalent Cr into trivalent Cr when impregnated into the wood. This process of fixation has been reported many times. A fast reduction of Cr VI is occurring on the wood surface and more in depth within 1 to 14 days after impregnation with CCA (cf. Coggins and Hiscocks, 1979, Cooper and Ung, 1989, McNamara, 1989 part 1 and 2), depending on the temperature.

The tank test results might also give indications for leaching into soil. However, a study on leaching from wood in different types of soils kept at very high moisture content has shown that the depletion of CCA and DDAC components is generally even larger in soil then in water (Wang et al., 1998). This is probably due to the presence of organic acids in soil.

Finally, the shower test is expected to simulate less severe leaching by rain. This is confirmed, considering the cumulative leaching results after four days in the tank test and the shower test respectively. The large wood quantity is chosen to level out the large differences in uptake per wood sample that may occur at impregnation plants. However, the test is relatively expensive and inefficiënt, and is not suitable for harmonization within Europe. The data can not be used for other purposes than an arbitrary pass/fail criterion for judgment at the impregnation plant of the state of fixation of the wood preservative. The pass/fail criteria for The Netherlands are stated in the certification guideline for vacuum pressure impregnated wood (BRL06/01, 1993), and are under revision at the moment.

Relation between retention, availability and leachable fraction: In table 4 a comparison is made for CCA and Cu-Quat treated wood of the total content of wood preservative components, the "potential

Table 3: Cumulative leached components (in mg/m²) in leaching samples according to three leaching protocols on CCA-C and Cu-quat treated wood.

	ENV 1250 beaker test				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**quat**
0,04	17	2	3	29	19
0,13	30	3	4	42	46
0,29	44	4	6	78	66
1-1,29	60	5	7	177	80
1,96	74	6	10	217	128
3,96	88	7	15	248	149

	NEN 7345 tank test				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**quat**
0,25	69	2	4	60	1070
1	140	11	13	136	1340
2	208	19	18	254	1665
4	265	23	42	307	1825
9	327	26	53	332	1884
16	395	31	68	357	1945
36	422	33	83	391	2183
64	434	35	100	417	2291

	Shower test (Havermans c.s.)				
	CCA type C treated Norway spruce			Cu-quat treated Scots pine	
Sampling period (days)	**Cu**	**Cr**	**As**	**Cu**	**quat**
1	29	2	2	48	117
2	52	3	4	91	206
3	71	4	6	123	250
4	87	5	8	149	303
5	98	5	10	175	355

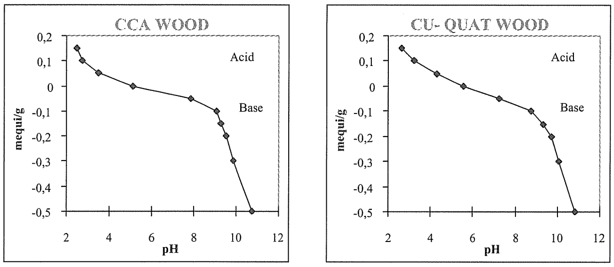

Figure 3: Graphic results of acid/base consumption in the pH static leaching tests on milled wood treated with Cu-quat and milled CCA treated wood.

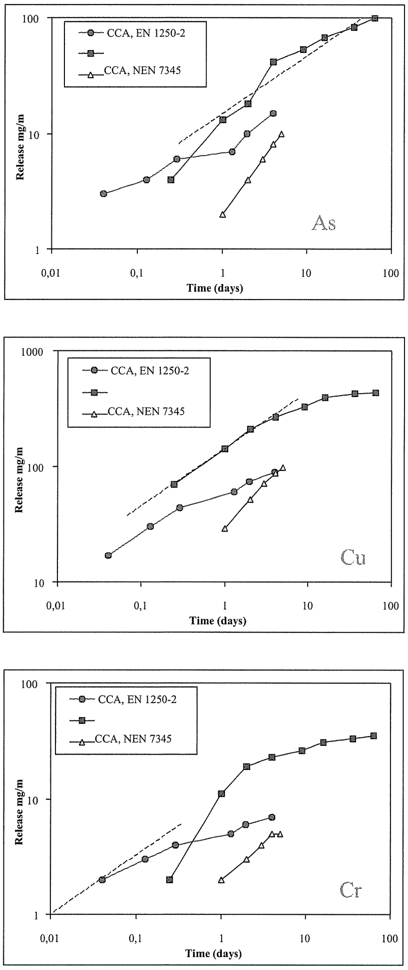

Figure 4: Graphic results of cumulative leaching of As, Cu and Cr in mg/m^2 from CCA treated wood. The dotted line represents a slope of 0.5, which corresponds to a square root of time relation (diffusion).

availability for leaching" of components in the pH static leaching results and the actual leaching data from NEN 7345. The potential availability and the actual leaching give a measure of the matrix interaction of the agents. Cr shows the strongest interaction as only 3 % is potentially leachabe and only 0.5 % is actually leached. The Cu availability in Cu-Quat treated wood is higher than in CCA treated wood, whereas the actual leachability is about the same when expressed in percent of total. It is not possible to say to what extent this higher Cu availability is due the type of preservative, or to the different wood species used. Van Eetvelde et al. (1996) has reported higher leaching rates of copper from Scots pine compared to Norway spruce. However, Quat is also leached to a large extent, indicating that the fixation characteristics of Cu-quat are much lower.

Table 4. Relation between total retained, potentially available and actual leachable fraction for CCA and Cu-Quat treated wood.

	CCA			Cu-Quat	
	Cu	**Cr**	**As**	**Cu**	**Quat**
Ratio of potentially availabe to the total (%)	58	3	9	91	-
Ratio of actual release (limit approached) to the total (%)	10	0,5	1	9	83
Ratio of actual release (limit approached) to the potentially leachable (%)	17	14	17	10	-

The volume of wood and water: From the cumulative leaching results after four days in the ENV 1250-2, NEN7345 and the shower test it is concluded that the NEN7345 showed the highest leaching rates. Leaching in the tank test is a factor 2.5 to 8 higher than the beaker test and the shower test. The ratio of wood volume to water volume is about the same in ENV 1250-2 and NEN7345, and the ratio of the exposed wood surface to the volume of water is about a factor 5 lower in the NEN7345. Therefore the difference is most likely due to the elevated pH in the tank test.
The role of pH and DOC of the water: The leaching behaviour of metals from preservative treated wood in the pH static leaching tests showed great similarity to the leaching behaviour of other materials that may generate DOC (Dissolved Organic Compounds). Examples of other materials are sewage sludge, compost, soil, sediment, etc. (Harmonisation of Leaching/Extraction Tests, 1997, cf. Van der Sloot, 2000). The results showed that acidic as well as alkaline environments are increasing the leachability of heavy metals to a large extent (see figures 4 and 5). Mobilization of metals at neutral to alkaline pH due to DOC metal interaction appears to be an important release controlling mechanism. The initial pH of 4 in the NEN7345 is therefore considered too low (too acidic) in the case of wood as a material with a low buffer capacity. It leads to uncharacteristically high initial leachability

compared to leaching in contact with surface water with a neutral pH. The NEN7345 should be adapted to a neutral leachant to make it more suitable for wood.

Release patterns and long term leaching behaviour: The NEN 7345 is the only test that gives actual leaching results after more than two weeks exposure. The leaching rate of components from the preservative treated wood approaches a plateau after about 16 to 36 days. After three months the leaching rates for heavy metals are in the order of 0,1 to 0,9 mg/(m^{2}*day), and about 4 mg/(m^{2}*day) for the quat (DDAC) component. Between the three tests, the easiest comparison of results is after four days, thus neglecting the more variable results in the initial sampling periods of ENV1250-2. From the cumulative leaching figures after four days (see table 3) for CCA-treated wood it is concluded that the level of leached heavy metals in the beaker test ENV1250-2 is in the same order of magnitude as the shower test. This is about 3-5 times lower as in the tank test NEN7345. The higher leaching rates in NEN 7345 compared to ENV 1250-2 are confirming earlier reported results by Van Eetvelde et al. (1996). In general, the leaching rate of the heavy metals is decreasing in the order copper > arsenic > chromium compared to earlier studies with CCA-treated wood using NEN7345 (TNO, unpublished data). The higher leaching rate of copper is in line with other leaching studies using NEN7345; the higher leaching rate of arsenic compared to chromium is not (cf. Van Eetvelde & Homan c.s., 1995).

For the Cu-quat treated wood the patterns are different: The differences in cumulative copper leaching tests are much smaller (248, 307 and 149 mg/m^{2} in ENV1250, NEN7345 and the shower test). Copper leaching is higher in the beaker test then in the shower test. For the quat component, the leaching in the shower test is a factor 2 higher than in the beaker test, but a factor 6 lower than in the tank test NEN7345.

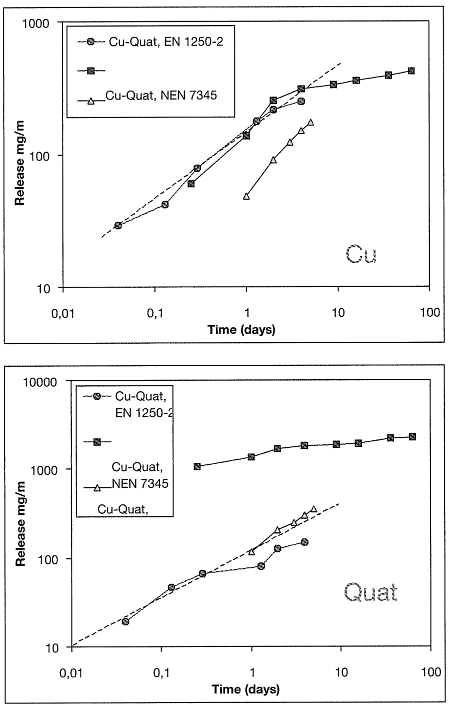

Figure 5: Graphic results of cumulative leaching of Cu and quat in mg/m² from Cu-quat treated wood. The dotted line represents a slope of 0.5, which corresponds to a square root of time relation (diffusion).

4 Conclusion

From the results it is concluded that from the three leaching tests (ENV 1250-2, NEN 7345 and shower test) and the pH static leach test studied on CCA-C and Cu-quat treated wood samples:

- The rate of leaching from preservative treated wood is declining over time, reaching a plateau after about 2-4 weeks exposure in water,
- Both acidic and alkaline environments are enhancing the leachability of heavy metals from preservative treated wood. In neutral to alkaline conditions the role of DOC in metal interaction appears to be an important release controlling mechanism.
- The NEN 7345 is the most suitable leaching method for characterization of the leaching behaviour of preservative treated wood for in service conditions. Due to the influence of low pH mentioned above, it is advised to adapt the NEN 7345 procedure to use neutral water instead of pH 4, if applied to preservative treated wood.
- The leaching pattern of wood in pH static leaching tests shows similarities in behaviour to other materials containing degradable organic matter, such as sewage amended soil. At pH > 6 the role of DOC becomes important in leaching trace metals from wood.
- During initial leaching periods both tests with submerged samples (ENV 1250-2 and NEN 7345) showed higher losses of heavy metals then the shower test with intermittent rainfall. This is a result of the fact that not all wood surfaces of the pile subjected to the test are leached. The difference between total wood surface exposure and partial wood surface exposure amounts to a factor of 4 - 6. Since the exposed wood surface can not be measured accurately the shower test results are less suitable to use for release predictions.
- The shower test results are more relevant if preservative treated wood is applied in above ground situations, whereas the tests with submerged samples are more applicable to wood applied in ground and/or water contact. The large scale of this test has several disadvantages (high cost, relatively poor repeatability). Furthermore the presentation of cumulative leaching in mg leached component per m^3 wood does not allow for easy comparison to other leaching data. Harmonization of the shower test within Europe is therefore not recommended. With a smaller scale experiment or even a translation from fully immersed test data the results for above ground conditions could be estimated.
- Presentation of leaching data as fluxes in mg/(m^{2}*day) may be misleading in relation to the net release to the environment. The cumulative release expressed in mg/m^2 as function of the time showed that ENV 1250 and NEN 7345 lead to more comparable end results.

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