

BIODEGRADABILITY STUDIES OF CERTAIN WOOD PRESERVATIVES IN GROUNDWATER AS DETERMINED BY THE RESPIROMETRIC BOD OXITOP METHOD

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Abstract. This study focuses on the biodegradability measurements of tall oil-based wood preservatives and their raw materials in groundwater as determined by the respirometric BOD OxiTop method. Certain substances were also analyzed in standard conditions described by OECD 301 F. Results show that creosote oil, a traditional wood preservative, does not biodegrade at all in groundwater over a period of 28 days, while tall oil and linseed oil biodegrade to some degree (13.4% and 14.8%, respectively). On the other hand, degrees of biodegradation of linseed oil, tall oil and creosote oil in the extra mineral and microbe including conditions described by OECD 301 F were 72.9%, 54.3% and 24.9% after 28 days, respectively. Studied raw materials of tall oil-based wood preservative production biodegrade to some degree in groundwater (variability from 11.9% to 18%) and quite effectively in OECD 301 F test (variability from 39% to 71.7%) over a period of 28 days. Abiotic degradation may also be significant with the studied substances.

Keywords: biodegradation, BOD OxiTop method, groundwater, wood preservatives

1. Introduction

Pure groundwater is one of our most important natural resources. In order to protect our water resources, it is necessary to examine the behavior of potential pollutants in an aqueous environment. Typical wood preservatives, like CCA (copper-chromium-arsenate), creosote oil and PCPs (pentachlorophenols), have caused various environmental hazards. Several contaminations have been caused by careless usage of preservatives and/or location of a wood preservation plant on a groundwater area or close to lakes and rivers (Lesage and Jackson, 1992; Lyytikäinen *et al.*, 2001). A trend towards less hazardous wood preservatives is apparent. The use of some traditional wood preservatives is already restricted by legislation. For example, the Environmental Protecting Agency (EPA) in the USA has already banned the use of the most poisonous CCA preservatives containing arsenic in residential structures beginning the 1st of January 2004 (www.epa.gov). European Union has also restricted the use of arsenic containing wood preservatives. Their use is only allowed in special purposes located far from centres of population and in special professional

applications. (www.ymparisto.fi). Due to restrictions of presently used wood preservatives, more environmentally friendly, alternative products are developed and used.

Tall oil is a good model substance of natural oils, which can give a possibility to protect wood as water repellents (Jermer *et al.*, 1993; Paajanen and Ritschkoff, 2002; Paajanen *et al.*, 1999; Van Eeckevelde *et al.*, 2001a, b). It is a renewable by-product of the pulp and paper industry. Two million tons of tall oil is produced in the world yearly. Tall oil-based products have already been common on the market for some time, e.g., as detergents, lubricants and surfactants. Biodegradability of tall oil-based wood harvester hydraulic oils and chain oils have been studied (Lauhanen *et al.*, 2000; Kuokkanen *et al.*, 2001, 2004; Vähäoja *et al.*, 2005). But wood preservation with tall oil products is a fairly new application, and research is still needed, including environmental aspects. This study focuses on determining biodegradation of different wood preservatives (mainly tall oil-based ones) in the groundwater environment, thus producing more information on their environmental effects. Some extra information was also gathered by means of the standardized biodegradation tests described by OECD 301 F (OECD, 1992). Determinations were carried out by means of the respirometric BOD OxiTop method.

2. Experimental

2.1. INSTRUMENTATION

The main interest of this study was the biodegradability of wood preservatives in natural groundwater. The instrument used was the respirometric BOD OxiTop (WTW), which has been proved to be a reliable method in biodegradability determinations in our earlier studies (Kuokkanen *et al.*, 2001, 2004; Vähäoja *et al.*, 2005) and also by other researchers (Conzelmann, 1996; Mittenzwey *et al.*, 1998; Reuschenbach *et al.*, 2003). Reuschenbach *et al.* compared BOD determinations of certain organic compounds using OxiTop and Sapromat methods. They found out that both systems were reliable respirometric test methods for determining the BOD of organic compounds.

Recently the applicability of this method was studied in the biodegradability studies of some forestry hydraulic and chain oils (Kuokkanen *et al.*, 2001, 2004, Vähäoja *et al.* 2005). As far as we know, the BOD OxiTop method has not been earlier used in the biodegradability studies of oils. Some studies related to polymeric materials have been done with this method previously (Mittenzwey *et al.*, 1998). This method is especially designed to determine the BOD of waste water (WTW).

The BOD OxiTop method is based on a very accurate pressure measurement. When organic matter degrades, it demands a certain amount of oxygen. When oxygen is consumed from the gas phase, pressure falls. At the same time carbon dioxide is produced, but it is absorbed onto solid sodium hydroxide pellets in this

method and it does not affect the pressure measured. Measurement time can be selected freely by the user. The measurement is fully automated and the instrument calculates biological oxygen demand (BOD) in mg/l as given in Equation (1).

$$\text{BOD}[\text{mg/l}] = \frac{M(\text{O}_2)}{RT_m} \cdot [(V_{\text{tot}} - V_l)/V_l + \alpha T_m/T_0] \cdot \Delta p(\text{O}_2) \quad (1)$$

$M(\text{O}_2)$ is the molecular weight of oxygen (32000 mg/mol), R is a gas constant (83.144 hPa mol⁻¹ K⁻¹), T_m is the measuring temperature (K), T_0 is 273.15 K, V_{tot} is the bottle volume (ml), V_l is the liquid phase volume (ml), α is a Bunsen absorption coefficient (0.03103) and $\Delta p(\text{O}_2)$ is the difference in partial oxygen pressure (hPa) as given by WTW.

2.2. EXAMINED SUBSTANCES

The wood preservatives used in this study were either commercial or experimental substances. The experimental substances were studied by means of a R&D project focused on developing effective and environmentally sound tall oil-based wood preservatives. The experimental substances were prepared from commercial tall oil fatty and rosin acids, whose properties are given in Table I. The studied substances and their carbon contents are presented in Table II. The preparation procedures of experimental substances are discussed afterwards.

The first step in (20/80) Fe(II) salt of TOFA/TOFA mixture preparation was formation of sodium salt of tall oil fatty acid. Sodium hydroxide (0.708 g) was added

TABLE I
Properties of commercial tall oil fatty and rosin acids

	Fatty acids		Rosin acids		
Acid value	194		168		
Saponification value	196		175		
Iodine value, Wijs	155				
Major components [%]	Oleic acid	29	Abietic acid	38	
	Linoleic and other polyunsaturated acids	64	Neoabietic acid	3	
	Saturated fatty acids	3	Palustric acid	8	
	Free rosin acids	1.8	Pimaric acid	2	
	Unsaponifiables		2	Isopimaric acid	3
				Dehydroabietic acid	21
				Other rosin acids	11
				Free fatty acids	5
			Combined acids	3	
			Unsaponifiables	6	

TABLE II
Studied substances and their carbon contents

Sample	Carbon content (%)	Sample type
Tall oil	73.15	C
Linseed oil	77.59	C
Creosote oil	84.97	C
Tall oil fatty acid	76.68	C
(20/80) Fe(II)-salt of TOFA/TOFA mixture	71.09	E
(15/85) Fe(II)-salt of TOFA/TOFA mixture	71.42	E
(10/90) Fe(II)-salt of TOFA/TOFA mixture	75.46	E
(5/95) Fe(II)-salt of TOFA/TOFA mixture	75.69	E
(20/80) Fe(III)-salt of TOFA/TOFA mixture	74.92	E
Oxidized (20/80) Fe(III)-salt of TOFA/TOFA mixture	71.23	E
(20/80) Mn(II)-salt of TOFA/TOFA mixture	73.74	E
(95/5) TOFA/TORA mixture	76.54	E
(85/15) TOFA/TORA mixture	77.17	E
(70/30) TOFA/TORA mixture	70.77	E
Neopentylglycol (NPG) ester of TOFA	77.62	E
Pentaerythritol ester of TOFA	77.17	E

C = Commercial substance, E = experimental substance, TOFA = Tall oil fatty acid, TORA = tall oil rosin acid.

to the solution of TOFA (50 g). Then 4.921 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in aqueous solution were added to the TOFA sodium salt. The solution was stirred 60 min at 95 °C until all the TOFA sodium salt had reacted. After that the aqueous phase was removed and the oil phase was washed twice with water in a separating funnel. In the resulting TOFA ferro-salt about 20% of pure TOFA had reacted with Fe(II) ion. This mixture was diluted with pure TOFA to get three other Fe(II) salt concentrations (15%, 10% and 5% Fe(II) salt containing mixtures). The TOFA Fe(III) salt preparation was similar to the earlier one with the exception that the iron-salt used was $\text{Fe}_2(\text{SO}_4)_3 \cdot 5\text{H}_2\text{O}$ and the amounts of iron-salt and NaOH were 4.226 g and 2.124 g, respectively. Oxidized Fe(III) salt mixture was prepared by oxidizing (20/80) Fe(III) salt of TOFA/TOFA mixture in oxygen atmosphere 24 h. (20/80) Mn(II) salt of TOFA/TOFA mixture was prepared similarly than corresponding iron salt mixtures with the exception that MnSO_4 was used as salt.

Mixtures of tall oil fatty acids (TOFA) and tall oil rosin acids (TORA) were prepared by weighing these acids into a beaker, which was heated at 80 °C until all of the rosin acids were dissolved. Esterification of tall oil fatty acids with pentaerythritol or neopentylglycol was done in a nitrogen atmosphere at 200 °C. The preparation took six hours. During the reaction water was removed in order to increase the yield of esters.

2.3. MEASUREMENTS IN GROUNDWATER CONDITIONS

The water used in these simulations was supplied by the Paavola water treatment station. This groundwater was thought to be a typical example of groundwater in the area nearby the city of Oulu and information on the quality of the water was easily available. Of course, properties of groundwater may vary depending on its way of formation. Chemical composition and variation of the BOD value after 28 days (5.8 ± 2.5 mg/l, $n = 13$) of this water has been presented in our earlier publication (Kuokkanen *et al.*, 2004). In order to evaluate the temporal variability of the BOD value of the groundwater used, a blank test was still done every time a new batch of ground water was taken into use. The amount of wood preservative used varied between 80–120 mg/l. These tests were simulation tests of natural conditions and hence no surfactants were used. Due to negligible nitrogen concentration of the water nitrification inhibitor was not used either.

As in earlier publications, the temperature of the measurement was 20.0 ± 0.2 °C. It should be noted that the temperature of real groundwater is much lower (4–7 °C in Finland) than in these simulations. Hence, biodegradabilities would be lower in that temperature, but otherwise behave in a similar way as here. The measurement period was always 28 days. The carbon content of samples was determined using a Perkin Elmer 2400 Series II CHNS/O-analyzer, which is based on combustion of sample and determination of carbon as carbon dioxide. The carbon contents are given as an average of two or three determinations. Precision of determinations of BOD/ThOD values of oils in groundwater based on data produced by OxiTop method has been observed to be good (Kuokkanen *et al.*, 2004; Vähöja *et al.*, 2005).

2.4. MEASUREMENTS IN CONDITIONS DESCRIBED BY OECD 301 F

The studied wood preservatives were used in concentrations of 60–370 mg/l. The nutrient solution in which the experiments were carried out contained following minerals: KH_2PO_4 , K_2HPO_4 , $\text{NaHPO}_4 \cdot 2\text{H}_2\text{O}$, NH_4Cl , CaCl_2 , $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in concentrations given in the OECD 301 F (OECD, 1992). Waste water was added for microbe source and nitrification was prevented with *n*-allylthiourea. All measurements lasted 28 days and temperature was 20.0 ± 0.2 °C.

2.5. ABIOTIC DEGRADATION

The significance of abiotic reactions (e.g., hydrolysis, chemical oxidation) on the degrees of biodegradation was also wanted to clarify. Certain wood preservatives were added to distilled water in concentrations of 65–120 mg/l and the samples were heated in an autoclave in 125 °C under pressure of 1.9 bar. This treatment lasted 15 min and it was carried out in order to kill the microbes of the sample. Oxygen demands of the samples were followed at 20.0 ± 0.2 °C 28 days.

3. Results and Discussion

3.1. GROUNDWATER CONDITIONS

This chapter includes all the results for different wood preservatives produced in groundwater simulations. Results are given as degrees of biodegradation (BOD/ThOD) in percentages vs. time.

3.1.1. *Common Wood Preservatives in Groundwater*

Some wood preservatives containing organic carbon were studied. The degrees of biodegradation vs. time of linseed oil and tall oil in groundwater are presented in Figure 1. Creosote oil was also studied but it did not show any aptitude for biodegradation in the groundwater environment during the 28 days. Perhaps microbes need a longer time to adapt to creosote oil biodegradation. Based on this observation, creosote oil could be evaluated to be a toxic substance, which inhibits normal degradation processes caused by microbes in groundwater. At least it can be said to be very slowly biodegradable in groundwater, thus causing a significant environmental threat.

On the other hand, linseed oil and tall oil seem to be less prejudicial to the groundwater environment, because they immediately began to biodegrade in the groundwater (see Figure 1). Degrees of biodegradation of linseed oil and tall oil after 28 days were 14.8% and 13.4%, respectively. Hence, they can be said to be moderately biodegradable in groundwater. Biodegradation is still effective after 28 days, because linseed oil and tall oil did not reach a steady phase (e.g., a constant value of BOD/ThOD percentage).

3.1.2. *Certain Possible Raw Materials of Tall Oil-Based Wood Preservation Production*

Tall oil-based wood preservatives should obey the rules of green chemistry if they are to be sold as natural, environmentally friendly products. The basic principle

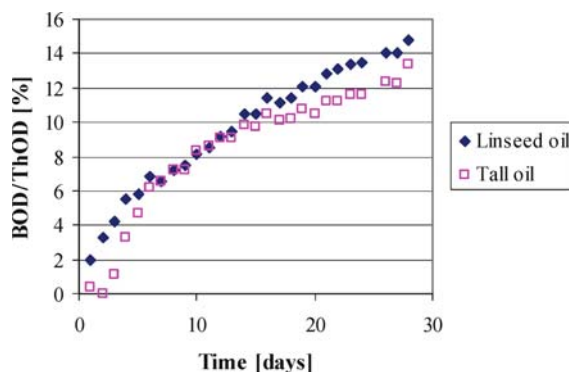


Figure 1. Plots of BOD/ThOD value vs. time of some natural wood preservatives in groundwater.

of green chemistry is that when new products are designed, produced, used or discarded, harmful chemicals must not be either used or produced. Hence, the raw materials and by-products of tall oil-based wood preservative production should be harmless to the environment. The nature of these raw materials was wanted to clarify with the help of biodegradation tests in groundwater.

Biodegradation of tall oil fatty acid (TOFA) and its mixtures with Fe(II) and Fe(III) salts in groundwater is presented in Figures 2 and 3. When Fe(II) salt is mixed with TOFA, the BOD/ThOD value after 28 days seems to rise slightly (from 12.9% for TOFA to 18.0% for 15/85 Fe(II)/TOFA and 20/80 Fe(II)/TOFA). However, the amount of Fe(II) salt has no significant effect on the biodegradation. It can be possible that the Fe^{2+} cation consumes oxygen and oxidizes to Fe^{3+} . This amount of consumed oxygen is impossible to separate from the oxygen consumption of biodegradable carbon, and that is why all oxygen consumption is evaluated to be combined with biodegradation in this discussion. 20/80 Fe(III) salt of TOFA/TOFA mixture behaves in a quite similar way than the other iron containing substances

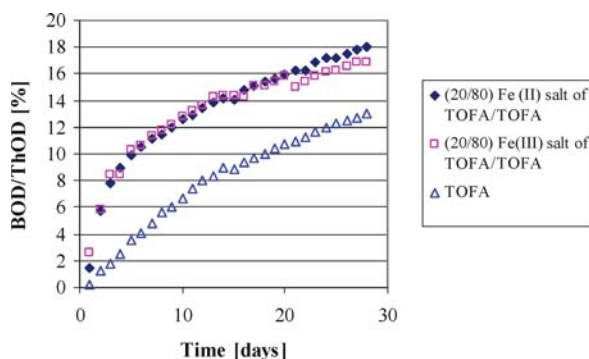


Figure 2. Plots of BOD/ThOD value vs. time of TOFA and its iron salt mixtures in groundwater.

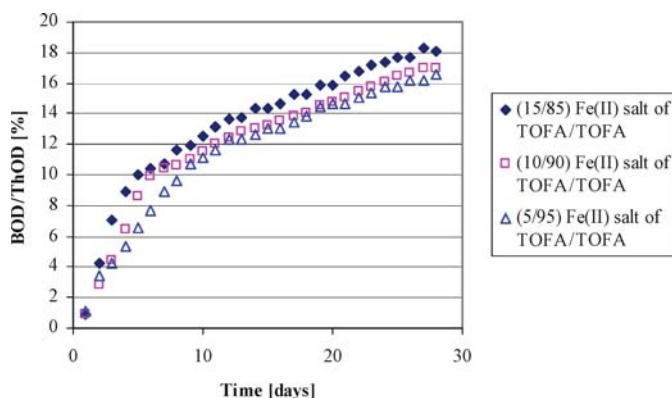


Figure 3. Plots of BOD/ThOD value vs. time of certain iron salt mixtures of TOFA in groundwater.

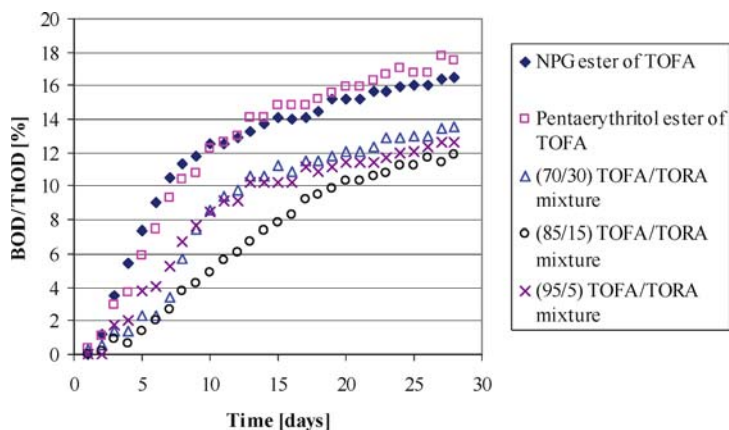


Figure 4. Plots of BOD/ThOD value vs. time of some TOFA esters and TOFA/TORA mixtures in groundwater.

and the change of the iron oxidation state does not seem to have any significant influence on the degree of biodegradation in this particular case.

Biodegradation of some usual tall oil fatty acid esters and mixtures of tall oil fatty acids and tall oil rosin acids in groundwater is presented in Figure 4. The degrees of biodegradation of the studied esters were almost similar, 16.5% for NPG ester and 17.5% for pentaerythritol ester, respectively. An interesting result is that tall oil fatty acid esters seem to biodegrade almost at the same rate as corresponding tall oil fatty acid-iron (II) and (III) salt mixtures. Groups attached to TOFA chain seem to have almost similar effect on the rate of biodegradation of these substances in groundwater conditions, when compared with pure TOFA. As seen in Figure 4, mixing of TOFA with TORA does not seem to affect biodegradation significantly. Only very small differences are observed when their degrees of biodegradation are compared with the biodegradability of pure TOFA. The BOD/ThOD percentages of TOFA/TORA mixtures after 28 days vary from 11.9% (85/15 TOFA/TORA) to 13.5% (70/30 TOFA/TORA), while the corresponding value of pure TOFA is 12.9%. Perhaps mixing bigger amounts of TORA with TOFA could have a more significant effect on biodegradation. However, this was not studied, because TOFA/TORA mixtures are dispersions. When bigger amounts of TORA are used, TOFA and TORA layers separate very effectively, thus making of a representative sample for a biodegradation test is difficult.

3.2. CONDITIONS DESCRIBED BY OECD 301 F

This biodegradation study has been continued over one year by now. Some persons of this group concentrated on developing new, potential tall oil-based wood preservatives, while the others have studied their biodegradation as a part of this R&D project. That is why a couple of new substances were studied in this section

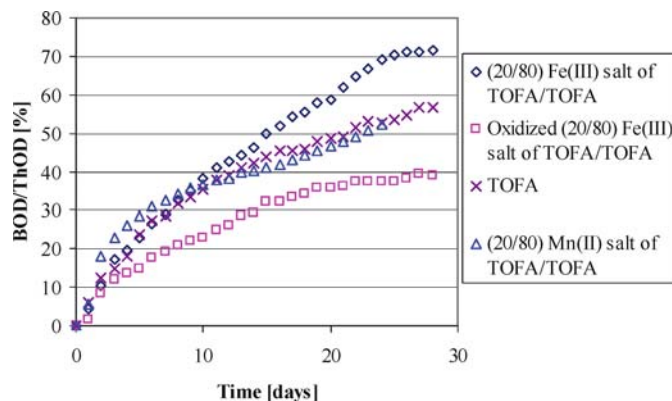


Figure 5. Plots of BOD/ThOD value vs. time of some potential raw materials of tall oil-based wood preservatives determined in the conditions described by OECD 301 F (chosen measurement scale for Mn (II) salt was exceeded after 25 days).

and they have not been still studied in groundwater conditions. All the substances studied in groundwater conditions were not studied in standard conditions described by OECD 301 F in this study. The reason for that is that *the main interest was to find out if these substances have any aptitude for biodegradation in groundwater*. However, biodegradation of certain substances studied in groundwater are also discussed in this chapter for the comparison of different conditions affecting the biodegradation. The biodegradation of some raw materials of tall oil-based wood preservatives in the conditions described by the OECD 301 F is presented in Figure 5.

20/80 Fe(III) salt of TOFA/TOFA has a degree of biodegradation over 70% after 28 days. Tall oil fatty acid (TOFA) has reached to the value of 56.9% in the same time. It was already noticed in the groundwater experiments that iron containing substances degrade somewhat better than TOFA. Nevertheless, when Fe(III) salt is oxidized thoroughly before the biodegradation test, BOD/ThOD value decreases to 39%. This indicates clearly that oxidation of iron consumes a great deal of the oxygen and the significance of the iron oxidation seems to be less in the groundwater conditions. The reason for the inevitable iron oxidation must be that a major part of the iron is in reduced form (Fe^{2+}) in these substances due to redox reactions happened in the production. Biodegradation reactions or chemical redox reactions in suitable conditions cannot be separated and they both are discussed here as biological oxygen demand of biodegradation reactions. What is interesting is that manganese salt biodegrades almost as fast as TOFA. So, manganese does not seem to oxidize significantly in these conditions. However, all the substances discussed here are moderately biodegradable in the extra mineral and microbe containing conditions. The limit value of 60% in ten days for a readily biodegradable substance was not met with any of these substances.

The BOD/ThOD values of certain commercial wood preservatives in extra mineral and microbe containing conditions are presented in Figure 6. Linseed oil

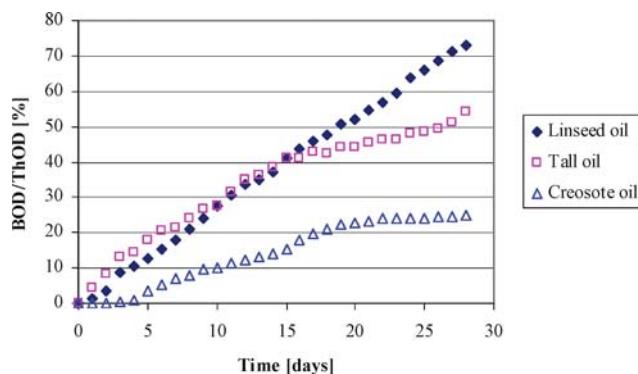


Figure 6. Plots of BOD/ThOD value vs. time of certain commercial wood preservatives determined in the conditions described by OECD 301 F.

biodegrades more effectively than tall oil as seen earlier also with a lesser degree in the groundwater conditions. The BOD/ThOD values of linseed oil and tall oil in the conditions described by OECD 301 F were 72.9% and 54.3% after 28 days, respectively. Creosote oil reaches a BOD/ThOD value of 24.9% in the same time and it already reached its steady phase in 20 days. Hence, it can be said that creosote oil is very slowly biodegradable, in addition to its toxicity, as seen also in the groundwater experiments. Thereby, when environmental reasons only are discussed, linseed oil and tall oil are much better choices for wood preservation than creosote oil.

3.3. ABIOTIC DEGRADATION

Some of the most effectively biodegradable substances were chosen for the abiotic degradation tests. More slowly biodegrading substances have hardly significantly better degradation properties than these ones. Results of abiotic degradation tests are shown in Table III.

As it can be seen from Table III, degradation percentages in sterile environment are relatively large. Thus, it seems that abiotic degradation can be important in the

TABLE III

Abiotic degradation of certain wood preservatives after 28 days

Sample	Abiotic degradation (%)
Linseed oil	7.8
Tall oil	7.4
(20/80) Mn(II)/TOFA mixture	9.7
(20/80) Fe(III)/TOFA mixture	3.8

groundwater environment with linseed oil and tall oil, because their degradation was not a lot quicker in the groundwater. However, when the degradation tests are done in the extra mineral and microbe containing environment, the biodegradation is significantly more important than abiotic degradation. In all, abiotic degradation can be quite important with these natural oils, whereas its importance is not necessary very significant with synthetic bio hydraulic oils as discussed elsewhere (Kuokkanen *et al.*, 2004).

4. Conclusions

Several wood preservatives and their raw materials were studied in biodegradability experiments in groundwater. Natural wood preservatives (tall oil and linseed oil), as well as the most common raw materials of tall oil-based wood preservatives, biodegraded moderately in the groundwater environment, whereas creosote oil, the traditional wood preservative, had no biodegradability at all during the studied short time period (28 days) in groundwater. Extra experiments carried out in the standard OECD 301 F conditions containing extra amount of minerals and microbes supported the observations made in groundwater. Abiotic degradation may also be significant with the studied substances.

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