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Treatment of Scots pine wood with oil in water and water in oil emulsion systems: Effects on boron leaching and water absorption

Eylem D. TOMAK, Ümit C. YILDIZ, Ahmet CAN

Karadeniz Technical University
Faculty of Forestry
Forest Industry Engineering Department
61080 Trabzon, Turkey

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IRG SECRETARIAT
Box 5609
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Treatment of Scots pine wood with oil in water and water in oil emulsion systems: Effects on boron leaching and water absorption

Eylem D. Tomak¹, Ümit C. Yıldız, Ahmet Can

Karadeniz Technical University, Faculty of Forestry, Forest Industry Engineering Department,
61080 Trabzon, Turkey

¹eylemdizman@yahoo.com

ABSTRACT

The aim of this study was to investigate the water absorption and water repellent efficiency of oils and oil water emulsions [oil-in-water (O/W) and water-in-oil (W/O)] and possibilities of reducing the amount of boron leaching from treated wood. Scots pine wood samples treated with waste or sunflower oil and water formulations contained 3%BA. The level of water absorption and water repellent efficiency were determined with cyclical wetting tests, total of 14 days. The results show that emulsion systems could decrease water absorption of wood and improve water repellent efficiency compared to those of only boron treated and untreated control ones. Especially W/O emulsion systems showed almost equal water repellent efficiencies as with pure oil even though retentions of the emulsion in wood were lower than pure oil's retentions. After leaching period, amount of boron remained in treated wood with W/O formulation gave some promising results to a degree of 35% based on the initial boron level whereas samples treated with O/W formulations did not show good performance to keep the boron in wood. The W/O emulsion systems might be a potential method to decrease boron leaching and increase water repellency of wood.

Keywords: boron, leaching, oil, emulsion, scots pine

1. INTRODUCTION

Boron compounds have been known as environmentally friendly wood preservatives that are very effective against to both insects and fungi (Yalınkılıç 2000, Temiz et al. 2008a). Beside many advantages of the compounds on wood applications, boron has a strategic importance for Turkey due to having the world's major source (64%) of boron mineral deposits (URL-1). However they susceptible to leaching under wet conditions and this disadvantage limits their utility and effects service life negatively (Yalınkılıç 2000). Obanda et al. (2008) extensively reviewed the approaches to reduce boron leaching from wood and stated no commercially viable solution of leaching. One approach is to restrict water access to wood by impregnating various hydrophobic agents together or apart from boron to overcome or delay boron leaching in time (Yalınkılıç 2000, Temiz et al. 2008a).

Natural oils seem to be capable of decreasing water uptake and improving water repellence of wood (Koski 2008). Additionally unsaturated oils such as drying oils can oxidize when exposed to oxygen in the air, which results in a more protective layer on the surface of the wood (Hyvönen et al. 2006). Their water repellent effectiveness in wood has been reported by many researchers. Wang and Cooper (2005a) reported reduction on the rate of water absorption by palm oil, soy oil and slack wax treatment. Improved water repellencies were reported in the case of using pyrolysis oils by Temiz et al. (2008c), linseed oil by Schulte et al. (2004), tall oil rosin

by Schultz et al. (2007), linseed oil, rape oil and three waxes by Treu et al. (2004), palm oil and soybean oil by Wang and Cooper (2005b), linseed oil, rapeseed oil and a proprietary resin derived from linseed oil by Spear et al. (2006). Linseed and hemp oil have recently been studied more intensely for that purpose, as well as tall oil resins (Homan and Jorissen 2004). van Eeckevelde et al. (2003) have tested Scots pine sapwood samples impregnated in a vacuum treatment at 40 °C with linseed, coconut and some tall oils for water performance.

High retention and deep penetration of oil into wood are necessary in order to provide good long-term performance in outdoor conditions (Temiz et al. 2008b, Koski 2008). However high retention levels increases wood weight and therefore application become impractical and uneconomical. High oil uptake also affects the oil stability within the wood. Due to high retention, oxidation slows and oils tend to be exuded from the wood. Problems of high oil uptake in the wood can be solved with the process technique. It has been shown that the emulsion technique is one way of solving the problem. Almost equal water repellent efficiencies were obtained with emulsion treatments like as pure oil treatment even when the oil retentions were halved (Hyvönen et al. 2006, Hyvönen et al. 2007a; b, Koski 2008).

Although oils usually show good water repellence, they do not show good resistance against to fire and wood degradation organisms in many times. Addition of biocides to oil treatment is needed for further protection. Combination of boron compounds or biocides with oils could improve fire and biological resistance (Palanti and Susco 2004, Lyon et al. 2007, Temiz et al. 2008a, Podgorski et al. 2008). One of the reported treatment techniques is impregnation with boron compounds first and then treatment with oil (Lyon et al. 2007, Temiz et al. 2008a, Podgorski et al. 2008). However these two-step treatments appear to be an impractical treatment process. Boron compounds does not soluble in pure oil even heat used. So this makes impossible to impregnate the wood in one step. For this respect emulsions that combine boron and oil in one formulation could help to impregnate the wood with single treatment. In this case dilution of oil with water was chosen as the retention controlling method and combining oil and boron in one formulation.

In this study, oil in water and water in oil emulsions were prepared at two different ratios (1/4 and 2/3) by using two surface active agents. The aim of the study was to investigate the water repellent efficiency of waste and sunflower oil emulsions and the possibilities of reducing the retention and leaching amount of boron from treated wood.

2. EXPERIMENTAL METHODS

2.1. Sample and formulation preparation

Scots pine (*Pinus sylvestris* L.) logs obtained from Blacksea region from Turkey. Sapwood samples with dimensions of 15 x 25 x 50 mm were used for the experiments. The moisture content of samples was around 12%. Sunflower oil and used (waste) vegetable oil was provided by the Ordu Oil Company-Turkey and Ezici waste oil collecting company-Turkey, respectively. Two kinds of emulsion systems (W/O and O/W) with a ratio of 1/4 and 2/3 were used. Emulsions were prepared with a dispenser and continued to mix with a shaker for 12 hours. As a surface active agent, detergent based sodium dodecyl sulphate (SDS), Merck and polymer based Kollicoat (KC), Basf were used with different concentrations depend on the oil and water ratio. KC is a polystabilizer in emulsions (Ahmed et al. 2003). Water in O/W emulsion formulations contained 3% BA while 3% BA was inside the total W/O emulsion formulations. Both oils and

surface active agents were used for preparation of O/W formulation, whilst only waste oil and KC was used for W/O formulation.

2.2. Impregnation and treatments

Wood samples were impregnated with O/W formulations at 600mm-Hg vacuum for 30 min and a pressure of 5 bar for 60 min at room temperature. After the impregnation procedure, samples were taken out from the impregnation system and residual solutions on the wood surface wiped off. The absorption of the formulation was calculated with a formula (Eq.1) by using the weights before and after the impregnation as described by Hyvönen et al. (2006).

$$A=(W_{ai}-W_{bi})/V(\text{kg}/\text{m}^3) \quad \text{Eq.1}$$

Where,

W_{ai} : Sample weight after impregnation (kg)

W_{bi} : Sample weight before impregnation (kg)

V : Sample volume (m^3)

Samples treated with O/W formulation contained KC wrapped up in an aluminum foil and put in an oven at 80 °C for 24 hours after the impregnation due to possibility of polymerization process. In the case of using SDS in O/W formulations, samples were air dried for 3 days at room conditions and then oven dried at 80°C for 48 hours to evaporate the water. The dry weight of the samples was measured. Retention was calculated to according to Eq.2 (Hyvönen et al. 2006). Then samples were conditioned for four weeks.

$$R=(W_{odt}-W_{odi})/V(\text{kg}/\text{m}^3) \quad \text{Eq.2}$$

Where,

W_{odt} : Oven dry sample weight after impregnation (kg)

W_{odi} : Oven dry sample weight before impregnation (kg)

V : Sample volume (m^3)

Impregnation conditions with W/O emulsions were the same like as O/W emulsions. However three parallel treatments were made. Samples in the first group were wrapped up in an aluminum foil and put in an oven at 80 °C for 24 hours after the impregnation. Heat applied at 100 °C and 160 °C during the impregnation of other two groups, respectively. Retention and absorption values of the samples were calculated as mentioned above. Samples were then conditioned.

2.3. Water absorption and water repellent efficiency test

Every variation of five treated and untreated oven dried wood samples was placed into a beaker filled with deionised water. After 6, 24, 48h and thereafter at 48-hour intervals, the samples were removed from the water, dabbed of with tissue. The dimensions and weights of the samples were measured for a total of 14 days. Relative water uptake (WA) and water repellent efficiency (WRE) were calculated after each water replacement according to Eq.3 and Eq.4.

$$WA = [(W_2 - W_1) / W_1] \times 100 \quad \text{(Eq.3)}$$

$$\text{WRE} = [(W_{A_c} - W_{A_t}) / W_{A_c}] \times 100 \quad (\text{Eq.4})$$

Where,

W_2 = wet weight of the wood samples after wetting with water, W_1 = initial dry weight,
 W_{A_c} = Water absorption values of untreated controls, W_{A_t} = Water absorption values of treated samples.

2.4. Boron analysis in wood

The boron content of both leached and unleached wood samples was analyzed using Inductively Coupled Plasma Spectrometer (ICP-AES). Samples used in water absorption test were evaluated as leached samples for boron analysis. Sample preparation was done according to AWP A7-93 (1993). Wood samples were ground in a Wiley mill with a hole size of sieve 0.5 mm (IKA MF10) and oven dried at $103 \pm 2^\circ\text{C}$. 1g of ground wood was weighed to the nearest 0.01g and placed in a 100 ml flask. Nitric acid (65%) was then added to the flask, which was placed on a hot plate. Following to brown fumes, hydrogen peroxide (30%) was added dropwise to clear the solution. After cooling, the contents in the flask were filtered through Whatman #4 filter paper and were diluted with distilled water for measurement. For each treatment, five replicate samples were ground and analyzed.

3. RESULTS AND DISCUSSION

3.1. Retention and absorption of oils

The retention and absorption of O/W emulsions (kg/m^3) in pine wood samples is shown in Fig.1. When using pure oil at the same impregnation parameters, the retention values would be approximately $500\text{-}550 \text{ kg/m}^3$. Retention values could be decreased by using O/W emulsions to degree of 70-90% depends on the ratio of oil and water in the formulation and active surface agent. As an expected, when water ratio increased in the formulation, retention values considerably decreased because of the high amount of water evaporation. Surface active agents affected the absorption of formulations. As shown in Fig.1, SDS gave higher absorption than KC. However KC showed higher retention than SDS. This is probably due to, KC a polyvinyl alcohol- polyethylene glycol graft copolymer might polymerize inside wood after heat treatment. The absorption differences between O/W formulations may derive from different viscosity of the solutions, the particle size of distribution of the emulsion and the boric acid concentration in the formulation. It is reported that different surface-active agents influence the physical properties of emulsions (viscosity, particle size distribution, stability) differently and thereby the penetration and distribution of emulsions within the wood structure may differ (Hyvönen et al. 2007b). Hyvönen et al. (2006) also reported some other reasons for the absorption differences that could be because of air bubbles in the emulsions and shear stability of the emulsion. Sunflower (SO) and waste oil (WO) in the O/W formulations showed similar retention values to each other whilst in the case of using SDS, absorption of SO/W formulations was a bit higher than that of WO/W formulations. This might be due to viscosity differences between the oils. Fig. 2 shows the retention and absorption of W/O emulsions (kg/m^3) in pine samples. Retention of W/O formulations was higher than that of O/W formulations, even heat used. The retention values could be nearly halved and decreased approximately 15% when the water/oil ratio was 2/3 and 1/4, respectively. Hyvönen et al. (2006, 2007b) also halved the retention of tall oil in wood by using emulsion techniques. Heat application during impregnation affected the retention values. Especially treatment at room temperature and 100°C showed similar absorption and retention

levels however treatment at 160 °C differed from others. KC might have played an important role with the heat on the retention levels. From the results, it can be concluded that boric acid solution and oil can be combined in one formulation and wood can be treated with the formulation uniformly.

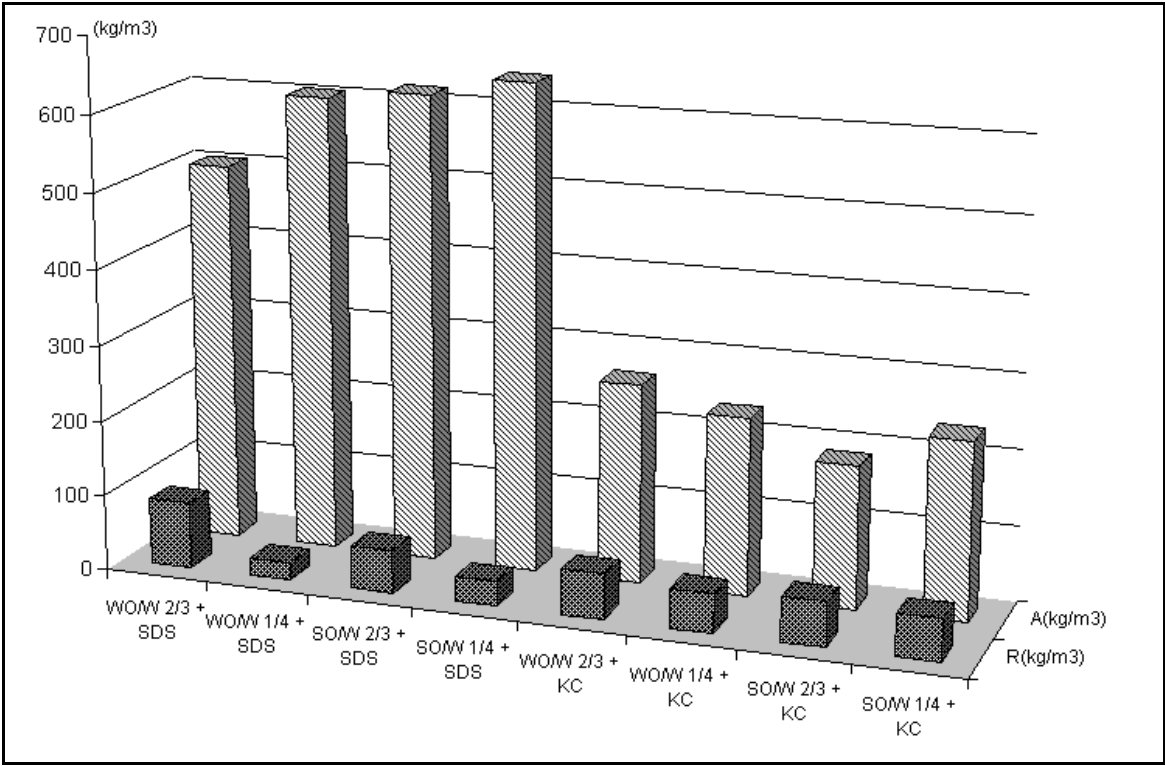


Figure 1: Retention and absorption of oil/water emulsion formulation (kg/m³)

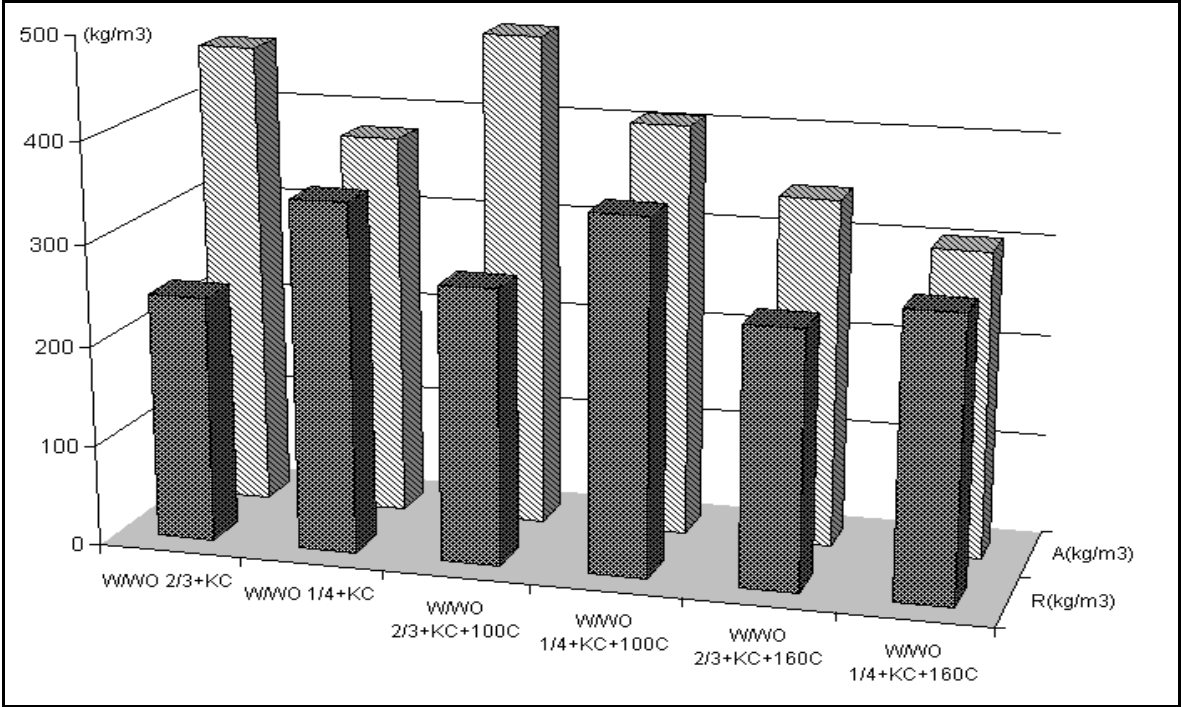


Figure 2: Retention and absorption of water/oil emulsion formulation (kg/m³)

3.2. Water absorption and water repellent efficiency

Boric acid impregnation had no positive effect on WA and WRE in addition WA levels of boric acid treated samples after 336h in water were a bit higher (115%) than that of the control samples (110%) during both early and subsequent leaching periods. This was also proven by Baysal et al. (2006). WA and WRE of pure waste oil treated samples was around 22% and 80% at the end of 336 hours in distilled water. For reader's convenience these results are not shown in Figs. WA of samples treated with O/W formulation is shown in Fig. 3 and Fig. 4 for SDS and KC, respectively. However samples treated with O/W formulations showed quite high WA rates (70-90%) compared to pure oil's rates. Except for wood treatment with SO/W+KC with a ratio of 2/3, all treated samples with O/W formulations absorbed nearly same amount of water likely untreated controls at immersion time of 96h. From 96 hours to 336 hours treated samples began to differ from controls by absorbing less water again. Surface active agents also affect the water absorption and water repellent efficiencies (Fig. 6 and Fig.7). In general, SDS used formulations showed better performance than KC used ones. This might be due to different influence of agents on physical properties of emulsion (Hyvönen et al. 2007b). Among the O/W treated variations, SO/W (2/3)+KC demonstrated best water repellent efficiency (40%) after the immersion in distilled water for 336 hours (Fig. 7). There were not clear differences on efficiencies between the O/W ratios. Low performance of O/W emulsions on water repellencies might correlate with the retention of oils. The amount of oil inside wood might not be enough to form a hydrophobic layer on the wood surface for good effectiveness of O/W formulations.

Figs. 5 and 8 present the water absorption and water repellent efficiency results of W/O formulations. Although retention of pure waste oil was much higher than the emulsion treatments, the efficiency of these treatments were quite good. Depending on the W/O ratio, water absorption rates were between the range of 22-48%, whereas controls and boron treated samples showed 110% and 115%, respectively. Water repellent efficiency rates of treated samples were from 52% to 80%. Water repellents considerably reduces hygroscopicity, water uptake, shrinkage and increases WRE and ASE of boron compounds (Colak et al. 2004, Yalınkılıç et al. 1998, Baysal et al. 2006, Baysal et al. 2007). As the oil amount increased in the formulation and temperature raised, water repellencies were more improved. Due to oil treatment, water uptake by capillarity is strongly reduced by closing or filling the lumina in the wood. In the emulsion technique, the amount of oil is lower so that the effectiveness of the treatment may be based more on the penetration rates of oils during the impregnation and drying properties of oils when exposed to air. Drying characteristic of oils may have an effect on creating a hydrophobic layer on the wood cells that prevents water uptake (Hyvönen et al. 2006, Koski 2008). Sunflower and waste oil used in the study are drying oils (iodine value>130) with having high iodine value of 140 and 135, respectively. These oils oxidize to form a tough elastic film when exposed to air. As the amount of oil inside wood increased, empty places in the wood structure may be filled with oil and polymerized with the oxygen in the air. This might be the reason for having good effectiveness with W/O formulations. Hyvönen et al. (2006, 2007b) reported that with the emulsion treatments almost equal efficiencies reached than with pure tall oil even when the retentions were considerably halved. In this study although O/W emulsions did not show good effectiveness, emulsion technique seems to improve WRE of wood even boric acid inside the formulation.

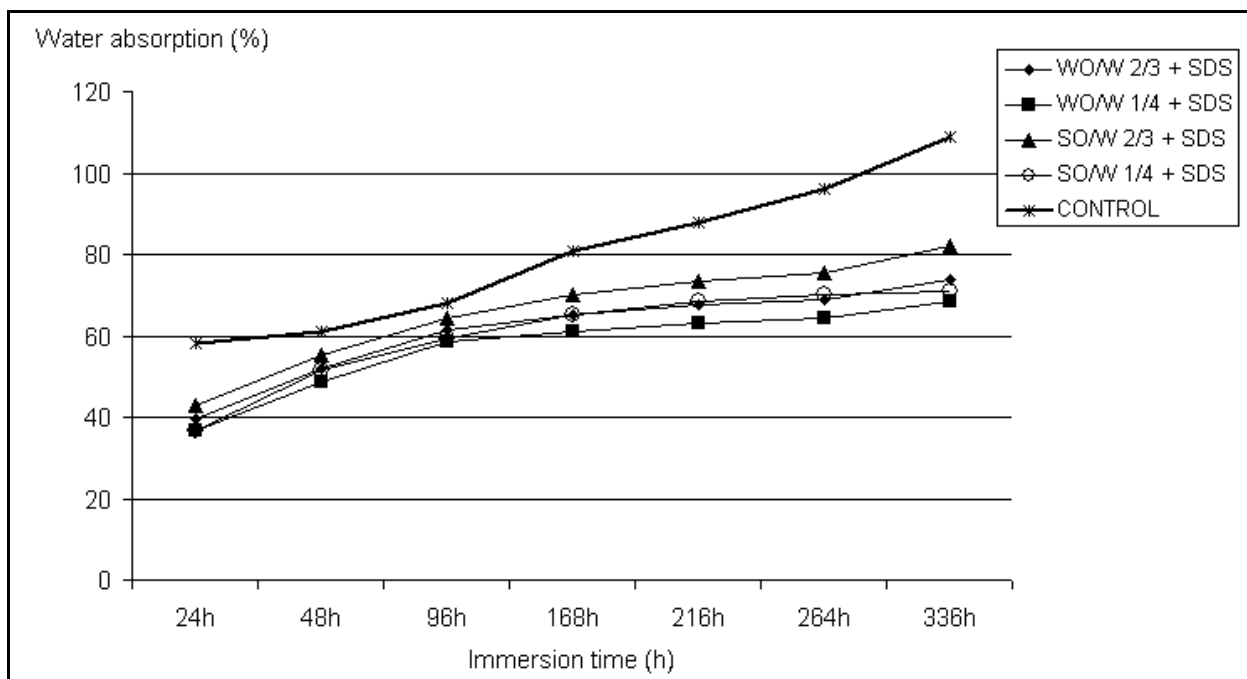


Figure 3: Water absorption of O/W emulsion prepared with SDS and BA

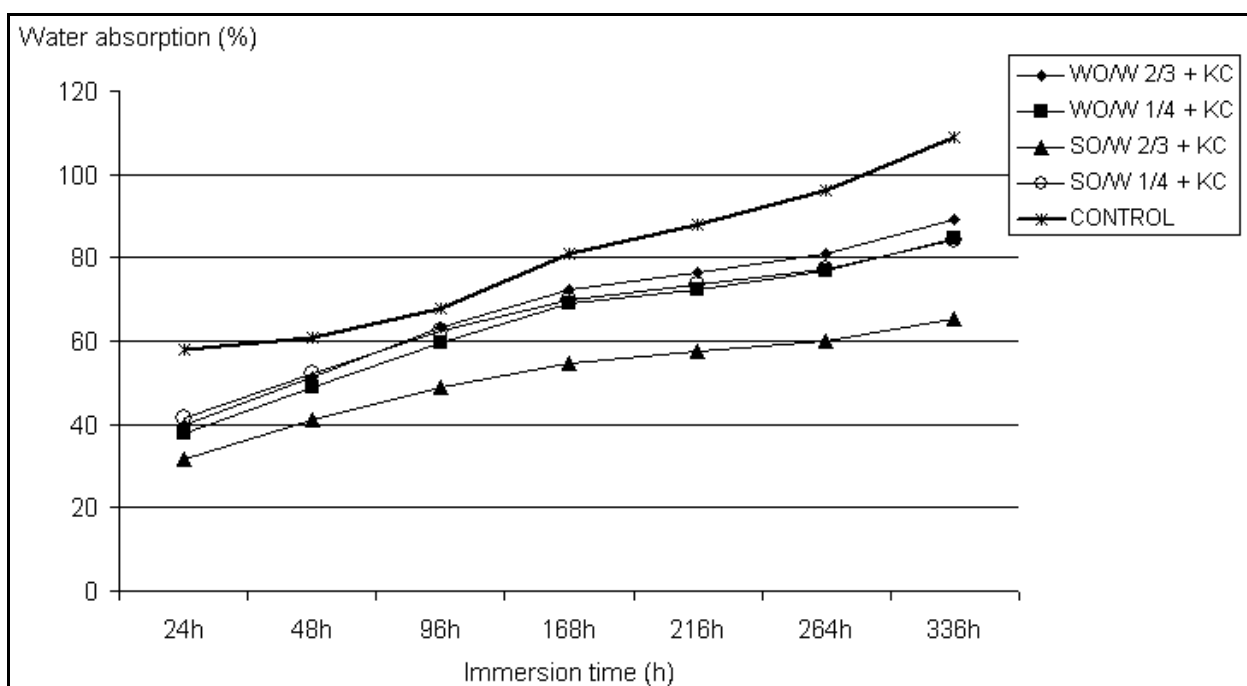


Figure 4: Water absorption of O/W emulsion prepared with KC and BA

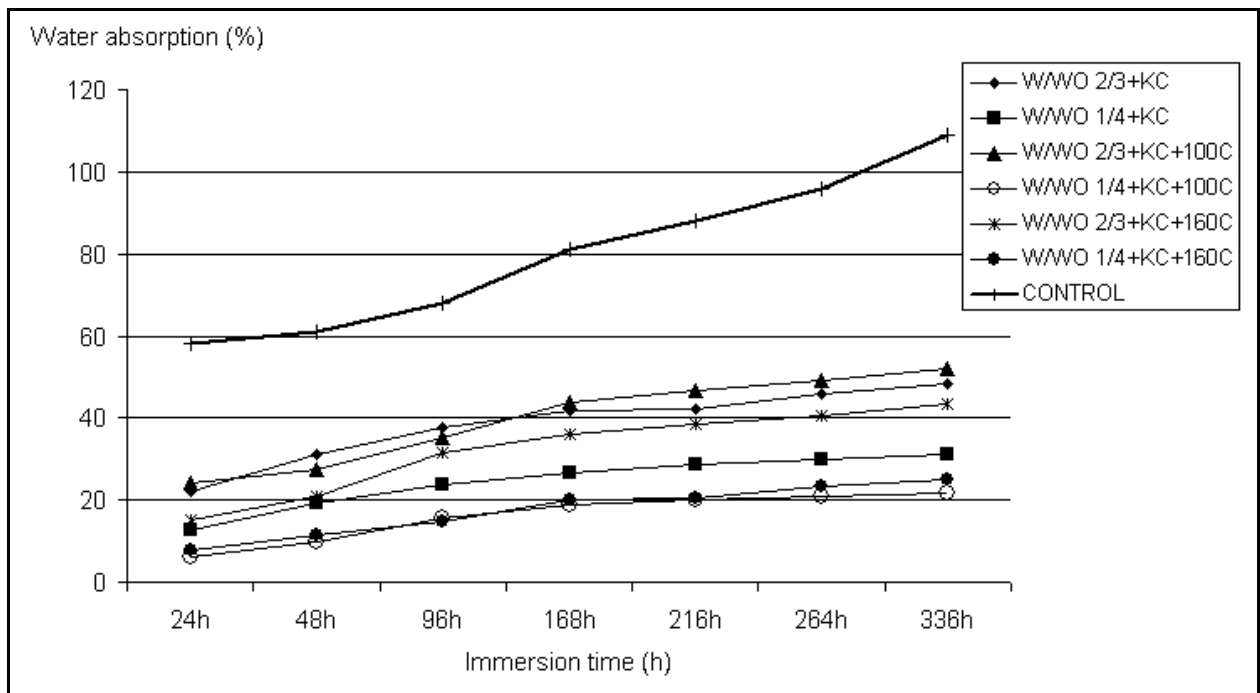


Figure 5: Water absorption of W/O emulsion prepared with KC and BA

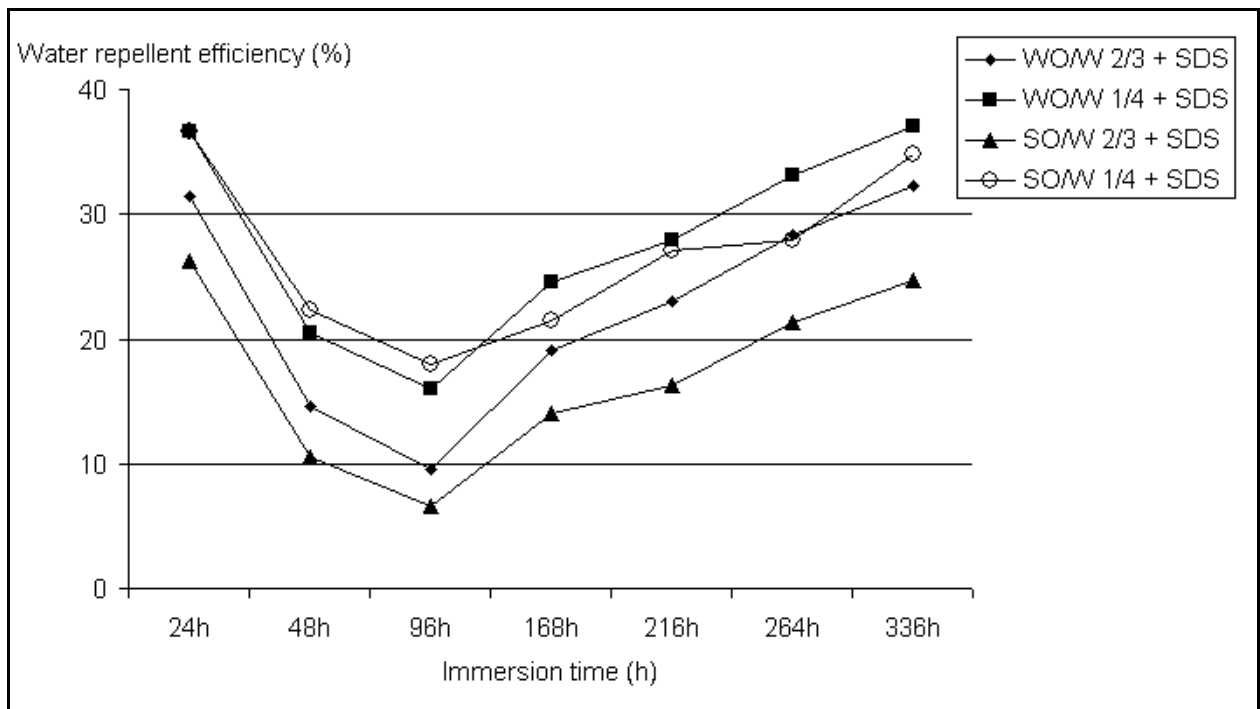


Figure 6: Water repellent efficiency of O/W emulsion prepared with SDS and BA

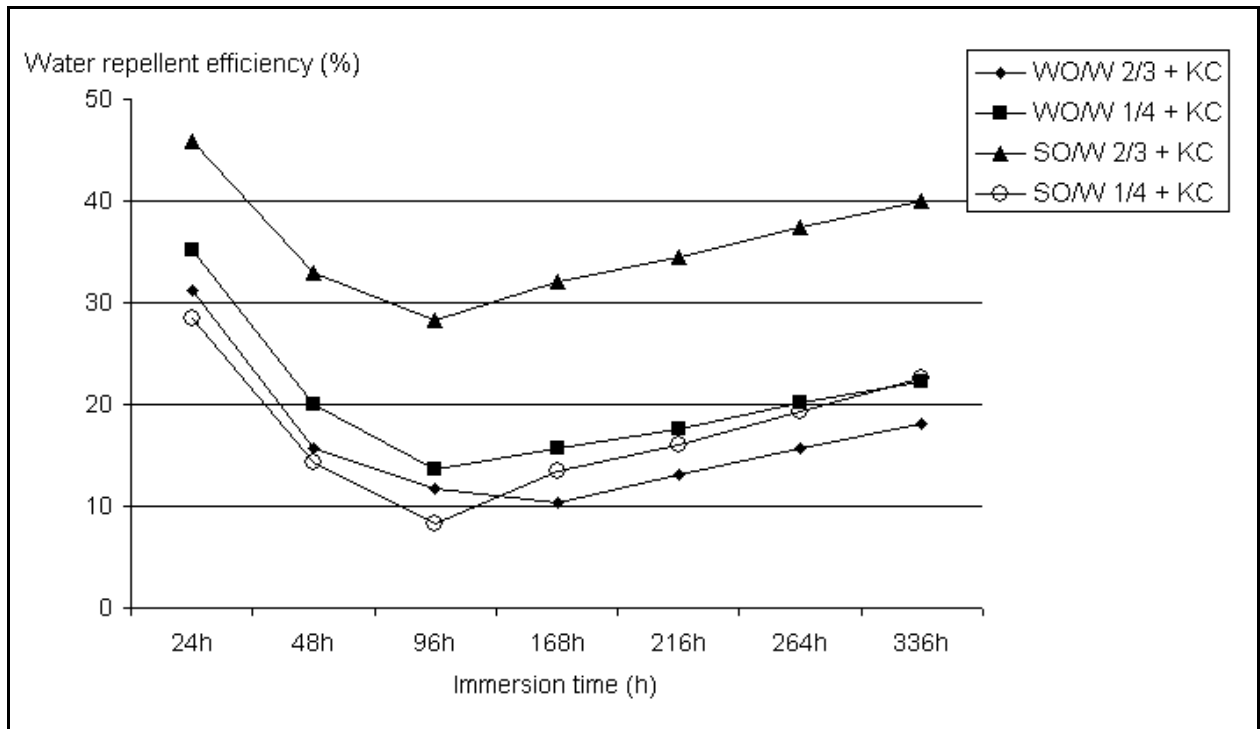


Figure 7: Water repellent efficiency of O/W emulsion prepared with KC and BA

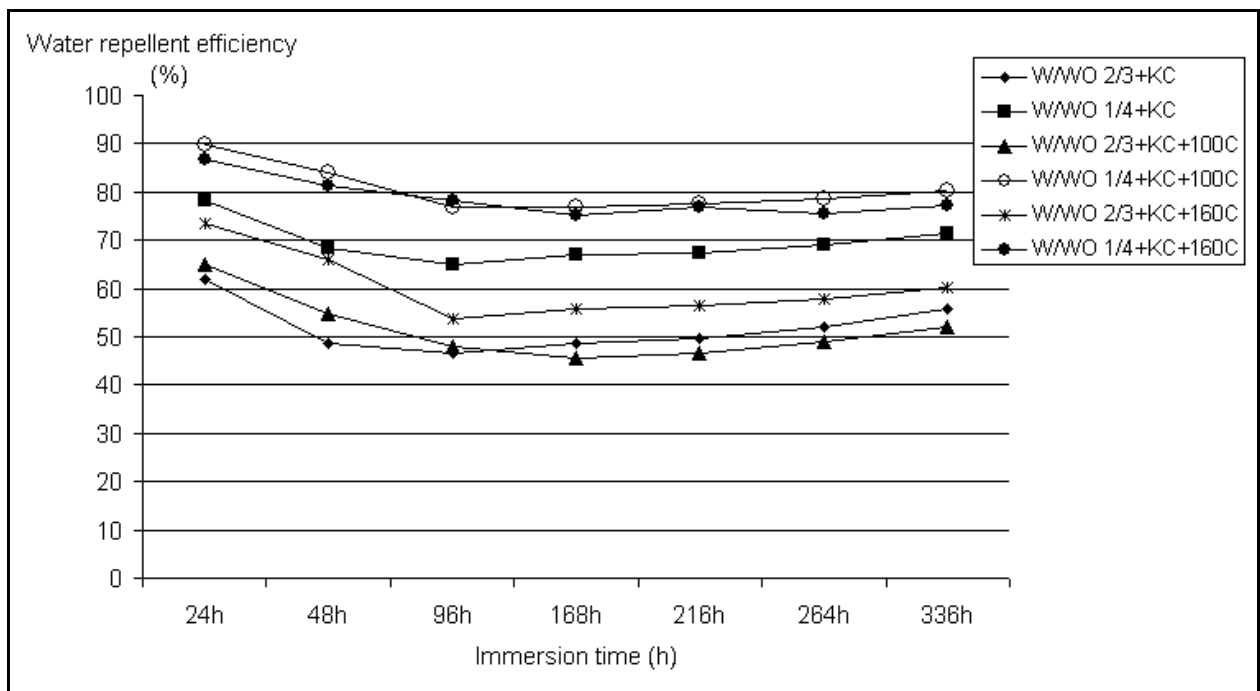


Figure 8: Water repellent efficiency of W/O emulsion prepared with KC and BA

3.3. Boron amount inside wood after leaching

Nearly all boron content (95%) of only boric acid treated samples has been removed during leaching. Figure 9 and 10 shows a percentage of boron amounts in wood samples after leaching for 336 hours in distilled water as measured by ICP. As can be seen from Fig. 9, O/W emulsions did not reduce the level of boron leaching remarkably. Waste oil with KC treatment seemed to be

more efficient than other O/W emulsions. Water repellent efficiency levels were also quite low with these formulations due to lack of enough oil in wood. In this respect, oils in O/W formulations could not create a hydrophobic layer on the surface of the wood cells, thereby they did not hinder water uptake. This could explain the high level of boron leaching from treated wood. On the contrary to O/W emulsion, W/O formulations have a potential to reduce boron leaching. Between the treatment methods, impregnation with W/WO at room temperature and then heat treatment at 80°C for 24 hours showed the best performance as keeping the boron in wood a level of 25 and 35%. More boron leaching occurred in the samples treated with the emulsions accompanied by heat application. However in the case of using W/WO at a level of 1/4 with 160°C heat application increased the amount of unleached boron in wood. The color of these samples turned to black as if they formed char on the surface. LeVan and Tran (1990) mentioned the char-forming catalytic effect of borax and boric acid, which has subsequently been shown in a number of studies (Baysal 2002, Wang et al. 2004, Ustaomer 2008). An increased amount of char might provide additional surface insulation and also inhibit water uptake. In addition these samples showed quite good water repellent efficiencies. Lyon et al. (2007) applied boron and oil treatment to wood as two step process and they found that linseed oil seemed to be more efficient than soybean oil, and much better than rapeseed oil, in preventing leaching around to rate of 17 to 34%. In our previous study, similar rates obtained in the case of using sunflower and waste oil with two step treatments (unpublished data).

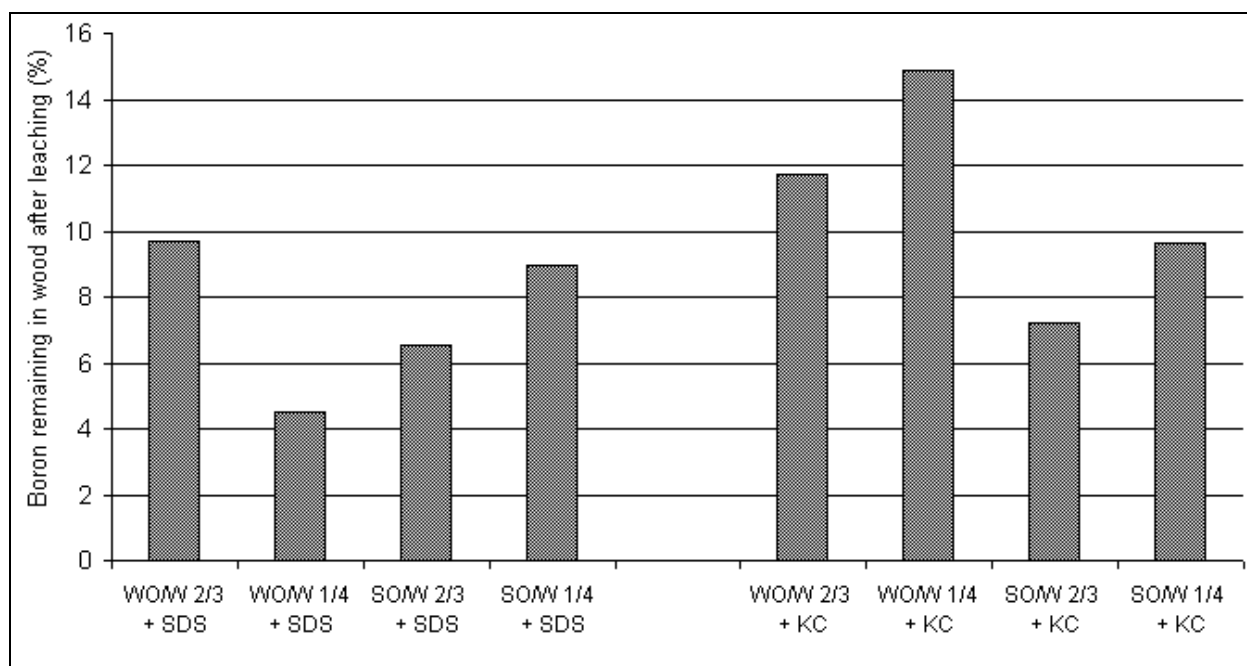


Figure 9: Boron remaining (%) in O/W emulsion treated wood samples after leaching 336h in distilled water

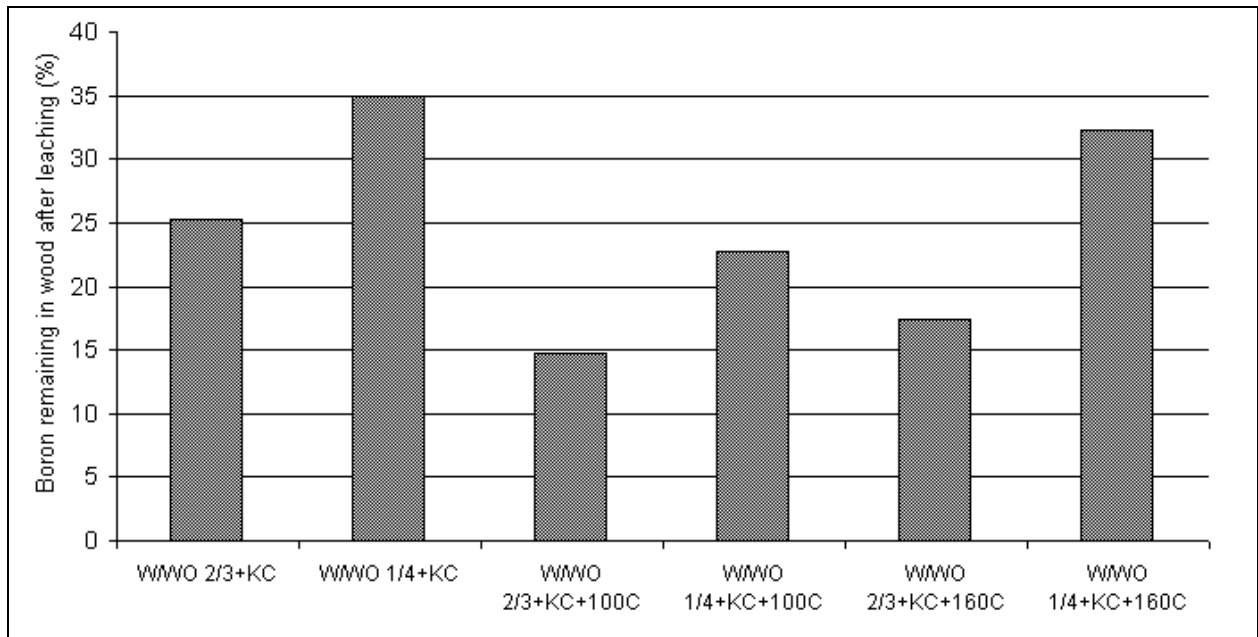


Figure 10: Boron remaining (%) in W/O emulsion treated wood samples after leaching 336h in distilled water

4. CONCLUSIONS

Oil retentions could be decreased by using W/O and O/W emulsion formulations. BA can be imparted to the formulation. After impregnation with emulsions, wood surface was not seemed oily. However in the case of using pure oil, the surface seems more oily and tacky. This is also important improvement for aesthetical aspect for some usages of wood. Emulsion treatments reduced the water uptake of pine wood and boron treated wood. Water repellencies could be improved more with water-in-oil treatments than oil-in-water treatments. With W/O formulation almost equal efficiencies can be reached as with pure oil even if the retentions are considerably lower. Surface active agent had an important role on the performance of the emulsion. Polymer based KC was better than detergent based SDS. W/O formulations seems a potential method of decreasing the amount of oil and combining the boron and oil in one formulation and also keeping the boron inside wood to a level of 35%. However more detailed studies are needed on the stability of emulsions. Decay tests and mechanical tests are necessary to a better understanding of the effectiveness of emulsions as a possible wood preservative.

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