

**EFFECTS OF SILICA GEL ON LEACHING RESISTANCE  
AND THERMAL PROPERTIES OF IMPREGNATED WOOD**

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**ABSTRACT**

The aim of this study was to increase the resistance of fire retardant chemicals for leaching in order to improve the fire resistance of wood. For this purpose, Scots pine (*Pinus sylvestris* L.) and Chestnut (*Castanea sativa* Mill.) wood impregnated with 3 % aqueous solutions of boric acid, zinc chloride and aluminum chloride at 60 °C by using vacuum- press impregnating method according to ASTM D1413-07 standard. In order to increase leaching resistance of the chemicals, wood samples post-treated with sodium silicate and then sodium bicarbonate to form insoluble silica gel in wood. After leaching test, it was determined that silica gel treatment decreased the amount of leachant in all three impregnation solutions. Effect of silica gel on the thermal behavior of treated samples was determined via thermogravimetric analysis (TGA). According to the results of TGA silica gel treatment improved the fire resistance property of wood especially in boric acid treated wood.

KEY WORDS: environmental performance; fire retardation and flammability; thermal analysis

**INTRODUCTION**

Wood has many inherently good properties, such as wide availability, renewability, a high strength to weight ratio, thermal and sound insulation, and low cost, which make it a preferred building material. However, wood also possesses several undesirable properties that impair its performance and limit its application. Flammability of wood is one of the most severe problems, and is one source of huge loss of life and property (Lee et al. 2004). The flammability of wood can be reduced by treating wood with fire retardants. Most existing fire retardants are effective in reducing different reaction to fire parameters of wood such as ignitability, heat release and flame spread. Unfortunately most of the treatments are not durable in exterior applications. The durability of fire

retardant treatments when exposed to humid conditions is closely linked to the water solubility of the chemicals used. Exceptions are, of course, fire retardants which react and thus form more complex and insoluble substances in the wood structure (Östman et al. 2001).

Borates belong to the oldest wood preservatives still in use and have several advantages as wood preservative in addition to imparting flame retardancy, providing sufficient protection against wood destroying organisms, having a low mammalian toxicity, and low volatility. However, boron compounds are susceptible to leaching under certain conditions, as they are not chemically fixed within the wood (Baysal et al. 2007). Neither investigations to reduce leaching by application of surface coatings nor the fixation of pure borate compounds were very successful (Ratajczak and Mazela 2007). Many metal salts can be used to reduce the flammability and smoke generation of wood. It was reported that the presence of metal compounds generally promoted the formation of char as well as water and carbon dioxide, and decreased the yield of tar (Fu et al. 2008). But, it was indicated that metal ions leach easily from the treated wood and metal ions containing preservatives could not protect wood at exterior conditions as long as expected (Yamaguchi 2002).

Aluminum chloride and zinc chloride are inorganic metal compounds, which have fire-retarding effects (Aslan 1998). Zinc chloride was the first large-scale commercial waterborne preservative in North America. Partially because of its negative effects on strength, but mostly because of related problems like decreased dimensional stability and increased hygroscopicity, waterborne preservative use declined during the first half of the 20th century. However, with increased environmental awareness of potential mammalian effects associated with the use of oil-type preservatives rekindled an interest in waterborne preservatives, especially the leach-resistant waterborne preservatives (Winandy 1995).

In recent years, polymers, water repellent reagents, and phenolic resins have been tested for their ability to reduce impregnation chemical leachability, increase dimensional stability, and provide high biological and fire resistance (Baysal et al. 2006). Since several silicon compounds may have the ability to increase the water repellency and react with cell wall components, such compounds can be used for limiting impregnation release from treated wood (Kartal et al. 2007).

Water glass is utilized, e.g. for the production of silica gels (drying, chromatography), as flame retardants, glue, extender, binding agents in paints, coating materials for building protection, sealing, and solidification materials of soils, etc. (Mai and Militz 2004).

LeVan (1984) indicated that leach resistant fire retardants could be formed by reacting soluble salts with metal salts to form insoluble, metallic salt complexes. Sodium silicate (Water glass) reacted with calcium chloride formed an insoluble, hydrated calcium silicate. Treatment with aqueous water glass ( $\text{Na}_2\text{O} \cdot n\text{SiO}_2$ ,  $n = 2.06-2.31$ ) was conducted either in a single step (Matthes et al. 2002) or in a two-step procedure (Furuno et al. 1991, Furuno et al. 1992, Furuno 1992, Furuno et al. 1993). In these studies all impregnated wood specimens displayed strongly negative moisture excluding efficiencies (MEE) because of the high hygroscopicity of both the water glass and the unreacted salts in the lumen of the cells. Leaching experiments revealed that considerable amounts of chemicals were washed out from the specimens (Furuno and Imamura 1998). For relatively insoluble bonds or films, liquid sodium silicates can be reacted with a variety of acidic or soluble metal compounds. Neutralizing an alkali silicate with acidic materials polymerizes the silica and forms a gel. Chemical setting agents that can be used in this manner include: mineral and organic acids, carbon dioxide ( $\text{CO}_2$ ) gas, and acid salts such as sodium bicarbonate and monosodium phosphate ( $\text{NaH}_2\text{PO}_4$ ) (PQ Corporation 2006).

The aim of this study was to reduce leaching of fire retardant chemicals by application of silica gel. Scots pine (*Pinus sylvestris* L.) and Chestnut (*Castanea sativa* Mill) samples were impregnated with boric acid, zinc chloride and aluminum chloride. And then samples were treated with aqueous

sodium silicate and sodium bicarbonate to form insoluble silica gel. Effects of silica gel treatment on the amount of leachant and thermal properties were investigated.

## MATERIAL AND METHODS

Sapwood of Scots pine (*Pinus sylvestris* L.) having density of 0.517 g.cm<sup>-3</sup> and Chestnut (*Castanea sativa* Mill) having density of 0.695 g.cm<sup>-3</sup> were used. Nondeficient, proper, knotless, and normally grown wood (without reaction wood, decay or insect or fungal damages) wood materials were selected. The specimens having a size of 20 mm (longitudinal) x 20 mm (radial) x 20 mm (tangential) were subjected to the treatments and leaching tests.

Boric acid (H<sub>3</sub>BO<sub>3</sub>), zinc chloride (ZnCl<sub>2</sub>) and aluminum chloride (AlCl<sub>3</sub>.6H<sub>2</sub>O) were used as fire retardant impregnating chemicals. Sodium silicate (Na<sub>2</sub>O.nSiO<sub>2</sub>, n:2.2) and sodium bicarbonate (NaHCO<sub>3</sub>) were used for silica gel treatment. All the chemicals were purchased from Yildiz Chemical Co. in Turkey. 3 % aqueous solutions of fire retardant chemicals were prepared for impregnation.

Air-dry samples were treated by vacuum-pressure method according to ASTM D 1413-07 (2007) standard. Before the impregnation process, the average moisture content of the samples was about 12 %. The samples were weighed before impregnation and placed in a vacuum chamber. After vacuum period (620 mm-Hg, 30 min), the impregnating solution containing, boric acid, zinc chloride or aluminum chloride were filled into the vacuum chamber until the samples were covered. Temperature of the solutions was 60 °C. Pressure was increased to 3 bars and lasted for 5 min. After the internal pressure was decreased to atmospheric pressure, impregnation solution was unloaded. Then, the samples were removed, wiped lightly to remove solution from the wood surface, and re-weighed (nearest 0.01 g) to determine gross retentions for each treating solution and sample. The retention for each treatment solution was calculated as following:

$$R = (G \times C / V) \times 10 \text{ (kg.m}^{-3}\text{)} \quad (1)$$

where  $G = T_2 - T_1$ ,  $T_2$  is the sample weight after impregnation (g),  $T_1$  is the sample weight before impregnation (g),  $V$  is the volume of sample (cm<sup>3</sup>), and  $C$  is the concentration of solution (%).

Samples were dried for 24 h at ambient temperature after impregnation and acclimatized at 20 +2 °C and 65 + 5 % relative humidity before further treatment.

Samples pre-impregnated with boric acid, zinc chloride and aluminum chloride were oven dried and weighed. Then samples were placed in vacuum chamber and 710 mm-Hg vacuum was applied for 30 min and sodium silicate having concentration of 20 % and temperature of 60 °C was filled. After 2 h treatment samples were removed from solution and dried for 24 h at ambient temperature and another 24 h at 80 °C in an oven. Dried samples were immersed for 3 h in sodium bicarbonate solutions having concentration of 5 % and temperature of 55-60 °C for precipitation and silica gel forming in the wood. After immersion, samples were dried for 8 h at ambient temperature and at 80 °C in an oven until they were reached constant weight. The weight percent gain (WPG) of the samples after silica gel treatment was determined, on an oven-dried weight basis, by measuring of the samples before and after silica gel treatment.

The leaching procedure was conducted in water as described by Baysal and Yalinkilic (2005). Ten specimens per treatment were immersed in a 1000-ml beaker containing 800 ml distilled water (10-fold volume of specimens) and stirred with a magnetic stirrer (400-500 rpm) at room

temperature for 8 h, followed by drying at 60 °C for 16 h. After each leaching period the water was exchanged with fresh water, added in a ratio of 10 volumes of water to 1 volume of wood. These procedures were repeated 10 times. The amount of leachant L was calculated from the difference of the oven-dried weight after treatment and oven-dried weight after leaching as following:

$$L = [(W_{oi} - W_{of}) / W_{oi}] \times 100 \quad (2)$$

where  $W_{oi}$  is the oven-dried weight of treated wood after silica gel treatment and  $W_{of}$  is the oven-dried weight of treated wood after leaching.

A thermogravimetric analysis (TGA) instruments Labsys TGA simultaneous TG-DTA apparatus was employed for the thermogravimetric tests. This apparatus detects the mass loss with a resolution of 0.001 mg. The temperature is measured in the sample holder. The char residue, maximum temperature of pyrolysis, and rate of weight loss can be obtained from TGA. High purity argon was used for the tests at a flow rate of 150 mL.min<sup>-1</sup>. The argon was purged for 20 min, before starting the heating program, to establish an inert environment. The sample mass was 15 mg. The experiments started with a drying session (up to 30 °C). The subsequent thermal decomposition was carried out from 30 °C up to 500 °C at a heating rate of 10 °C.min<sup>-1</sup>. Temperature of rapid pyrolysis and total weight loss (%) at 500 °C were determined.

## RESULTS AND DISCUSSION

The amounts of retention according to impregnation chemicals and some properties of the impregnation solutions are given in Tab. 1. Densities and pH values of impregnation solutions did not vary after impregnation. The amounts of retention in Scots pine were higher than that in chestnut in the case of all three chemicals. Scots pine has higher porosity and permeability than chestnut and thus absorbs more impregnation solution.

Tab. 1: Properties of impregnation solutions and amount of retentions

Impregnation solutions	C (%)	Before impregnation		After impregnation		Amount of retention (kg.m <sup>-3</sup> )	
		pH	D (g.ml <sup>-1</sup> )	pH	D (g.ml <sup>-1</sup> )	Scots pine	Chestnut
Boric acid	3	5.2	1.01	5.2	1.01	16.1	9.8
Zinc chloride	3	5.5	1.02	5.5	1.02	15.4	9.7
Aluminum chloride	3	2.6	1.02	2.6	1.02	11.4	7.4

C: concentration of impregnation solutions; D: Density

The highest retention was determined in Scots pine treated with boric acid as  $16.1 \text{ kg.m}^{-3}$ . Retention of boric acid was  $9.8 \text{ kg.m}^{-3}$  in chestnut. Although it is not comparable exactly because of variation of the sample size and impregnation conditions it can be said that the boric acid retentions reached in this study were much higher than that in studies reported by Ozcifici (2001) ( $1.96 \text{ kg.m}^{-3}$  boric acid retention in Scots pine) and Keskin (2007) ( $3.38 \text{ kg.m}^{-3}$  boric acid retention in oak). Amounts of retention of boric acid and zinc chloride were similar. The lowest retention ( $7.4 \text{ kg.m}^{-3}$ ) was determined in chestnut treated with aluminum chloride. This is an expected result because Yalinkilic (1993) indicated that aluminum chloride is a good fire retardant but impregnation with it is difficult.

Tab. 2: Weight percent gain and amount of leachants in wood pre-impregnated and treated with silica gel

Wood species	Treatments	$W_o$ (g)	$W_{oi}$ (g)	WPG (%)	$W_{of}$ (g)	L (g)	L (%)
Scots pine <i>Pinus sylvestris</i> L.	Boric acid	3.77	3.77	-	3.62	0.15	4.1
	Boric acid + Silica gel	3.63	3.90	7.52	3.80	0.11	2.7
	Zinc chloride	3.91	3.91	-	3.65	0.26	6.6
	Zinc chloride + Silica gel	3.80	3.96	4.24	3.87	0.09	2.2
	Aluminum chloride	3.64	3.64	-	3.39	0.24	6.7
	Aluminum chloride + Silica gel	3.63	3.82	5.13	3.68	0.14	3.6
Chestnut <i>Castanea sativa</i> Mill.	Boric acid	4.94	4.94	-	4.83	0.11	2.2
	Boric acid + Silica gel	4.89	5.34	9.11	5.24	0.10	1.8
	Zinc chloride	5.01	5.01	-	4.81	0.20	4.0
	Zinc chloride + Silica gel	4.94	5.23	5.75	5.15	0.07	1.4
	Aluminum chloride	4.85	4.85	-	4.63	0.22	4.5
	Aluminum chloride + Silica gel	5.02	5.31	5.73	5.09	0.22	4.2

$W_o$ : Oven-dried weight before silica gel treatment;  $W_{oi}$ : Oven-dried weight before leaching (Oven-dried weight after silica gel treatment); WPG: Weight percent gain after silica gel treatment;  $W_{of}$ : Oven-dried weight after leaching; L: Amount of leachant (g or %)

Impregnated samples were treated with silica gel to reduce leaching. The WPG of the samples after silica gel treatment and amount of leachants after leaching test were shown in Tab. 2. For all three impregnation chemicals the WPGs in chestnut after silica gel treatments were higher than WPGs in Scots pine. Due to higher cell wall content of chestnut, more silica particles could fix to samples. Also, Mai and Militz (2004) noted that the impregnation of hardwoods with inorganic silicon compounds resulted in a significantly higher weight percent gain than that of the softwood. The highest WPG after silica gel treatment was determined as 9.11 % in boric acid treated chestnut. The lowest WPG was determined as 4.24 % in zinc chloride treated Scots pine. For both wood species boric acid treated samples gave higher WPG values after silica gel treatment. Mai and Militz (2004) indicated that metal salt solutions can be used in order to precipitate the silicate

within the wood structure by replacing the sodium ions in water glass and boric acid was one of them. Also, Obut and Girgin (2006) noted that the aqueous sodium silicate solution could be gelled using borate compounds ( $\text{H}_3\text{BO}_3$ ,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ,  $\text{NaBH}_4$ ) and concluded that borate ions in solution promoted the formation of siloxane network. So, boric acid in the cell lumen contributed the formation of silica gel in treated wood.

As seen in Tab. 2, silica gel treatment reduced leaching of impregnation chemicals for both wood species. Silica gel layer formed in wood pores and on the outer surface of samples may prevent water absorption of treated wood specimens. Limitation of water-impregnating chemicals contact in the cell wall through low water absorption is expected to limit chemical leaching (Yalinkilic 2000). When sodium silicate dries by dehydration a gel forms but this gel is soluble and not adequate for films or bonds that will be exposed to weather or high moisture conditions. For relatively insoluble bonds or films, liquid sodium silicates can be reacted with a variety of acidic or soluble metal compounds. Neutralizing an alkali silicate with acidic materials polymerizes the silica and forms a gel. Multivalent metal compounds react with silicate solutions to form coatings or bonds by precipitation of insoluble metal silicate compounds (PQ Corporation 2006). Thus, sodium bicarbonate was used to precipitate silica and to form higher water resistant gel layer.

Amount of leachant (L %) for Scots pine were higher than that of chestnut. According to Cockcroft and Laidlaw (1978) a dense, relatively impermeable wood will not wet so easily as permeable one, and therefore there would be less tendency for back diffusion and leaching, so that any preservatives would be expected to have greater permanence in denser species. Because of higher density and lower permeability of chestnut amount of leachant were less than that of Scots pine. The amount of leachant from the specimens treated with boric acid 3 % concentrations was 1.5 and 1.2 times higher than that of the specimens treated with the boric acid + silica gel for Scots pine and chestnut, respectively. These results consistent with previous studies reported by Kartal et al. (2007) and Baysal et al. (2006).

For the thermal degradation, of the three components in wood, it is proposed that the thermal degradation of hemicelluloses occurs firstly while cellulose, then, thermally decomposes through two competing pathways. At lower temperatures, gradual degradation ultimately forms a carbonaceous residue. At higher temperatures, a rapid volatilization occurs via the formation of levoglucosan, which can be further decomposed into volatile and flammable products. On the other hand, lignin decomposes with lower rates by char oxidation. The pyrolytic behavior of wood is the overall behaviors of the three components, which makes it extremely complicated. However, the thermal degradation of wood is affected to a large extent by cellulose (Gao et al. 2006). Results of the thermogravimetric analysis (TGA) are shown in Tab. 3. According to the results, all the impregnation chemicals except aluminum chloride at chestnut reduced the total weight loss and temperature of rapid pyrolysis compared to untreated wood. With consistent these results Gao et al. (2006) indicated that such additives can lead to a lowering of the decomposition temperature and a higher char yield. According to LeVan et al. (1990), the increased amount of residual char in TG results was associated with the reduction of the combustible volatiles. The lowest total weight loss (52.1 %) and temperature of rapid pyrolysis was determined in samples impregnated with zinc chloride. Rowell and LeVan-Green (2005) indicated that zinc chloride is very effective in reducing the yield of levoglucosan. It was noted that zinc chloride reduced levoglucosan yield from 10.1 % to 0.3 %. They also reported that total weight loss determined at 500 °C by TGA was 74 % for zinc chloride treated wood and 81 % for boric acid treated wood. Wang et al. (2004) reported that total weight loss of boric acid treated wood at 500 °C was 65 % and temperature of rapid pyrolysis was 340 °C. It can be said that our findings in this study were better than that of these previous studies.

Tab. 3 : Results of thermogravimetric analysis

Wood Species	Treatments	Total weight loss at 500 °C (%)	Temperature of rapid pyrolysis (°C)
Scots pine <i>Pinus sylvestris</i> L.	Untreated	67.3	359
	Untreated + Silica gel	58.1	306
	Boric acid	53.5	332
	Boric acid + Silica gel	55.8	310
	Aluminum chloride	60.3	328
	Aluminum chloride + Silica gel	56.8	311
	Zinc chloride	52.1	271
	Zinc chloride + Silica gel	57.1	318
Chestnut <i>Castanea sativa</i> Mill.	Untreated	66.9	351
	Untreated + Silica gel	63.0	311
	Boric acid	57.4	312
	Boric acid + Silica gel	60.4	306
	Aluminum chloride	70.4	332
	Aluminum chloride + Silica gel	63.3	316
	Zinc chloride	61.5	311
	Zinc chloride + Silica gel	60.4	312

Silica gel treatment of wood contributed the fire retarding effects of impregnation chemicals. In the case of pre-impregnated and silica gel treated samples, the lowest weight loss was determined as 55.8 % in boric acid and silica gel treated Scots pine. As seen in Tab. 3 silica gel has a fire retarding effect regardless other impregnation chemicals. In the case of unimpregnated samples, silica gel lowered the total weight loss from 67.3 % to 58.1 % for Scots pine and from 66.9 % to 63 % for chestnut. Also temperature of rapid pyrolysis was lowered. Especially, for both wood species in the case of boric acid impregnation, silica gel lowered the decomposition temperatures and shifted the TG curves to lower temperature (Fig. 1 and Fig. 2). Le Van (1984) indicated that sodium silicate act as a physical barrier, which can retard both smoldering combustion and flaming combustion by preventing the flammable products from escaping and by preventing oxygen from reaching the substrate. These barriers also insulate the combustible substrate from high temperatures. Miyafuji and Saka (2001) indicated that this enhanced fire resistance was assumed to be due to chemical and physicochemical effects, such as dehydration and carbonization of wood by silica gel.



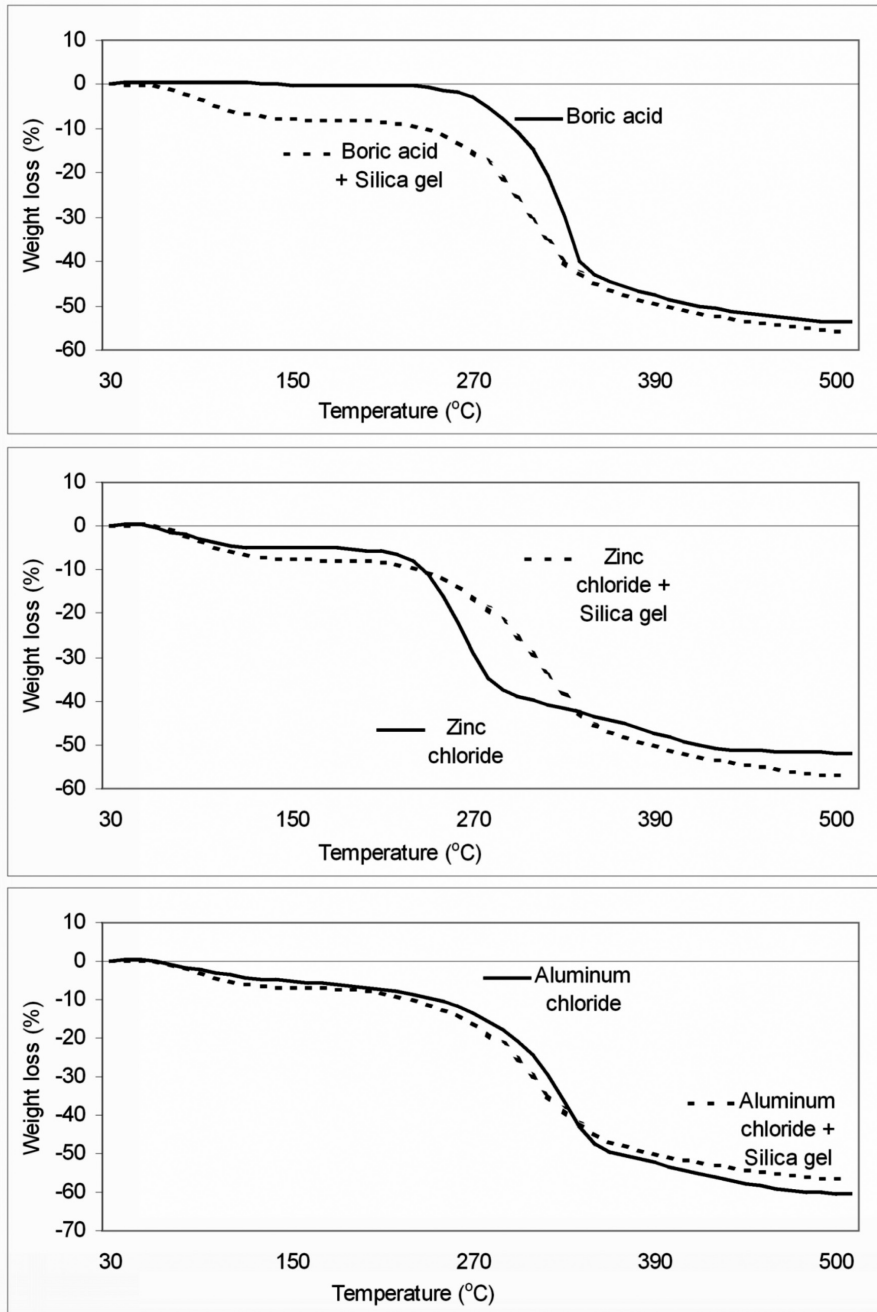


Fig. 1: TGA of treated Scots pine in argon at 10 °C.min<sup>-1</sup>



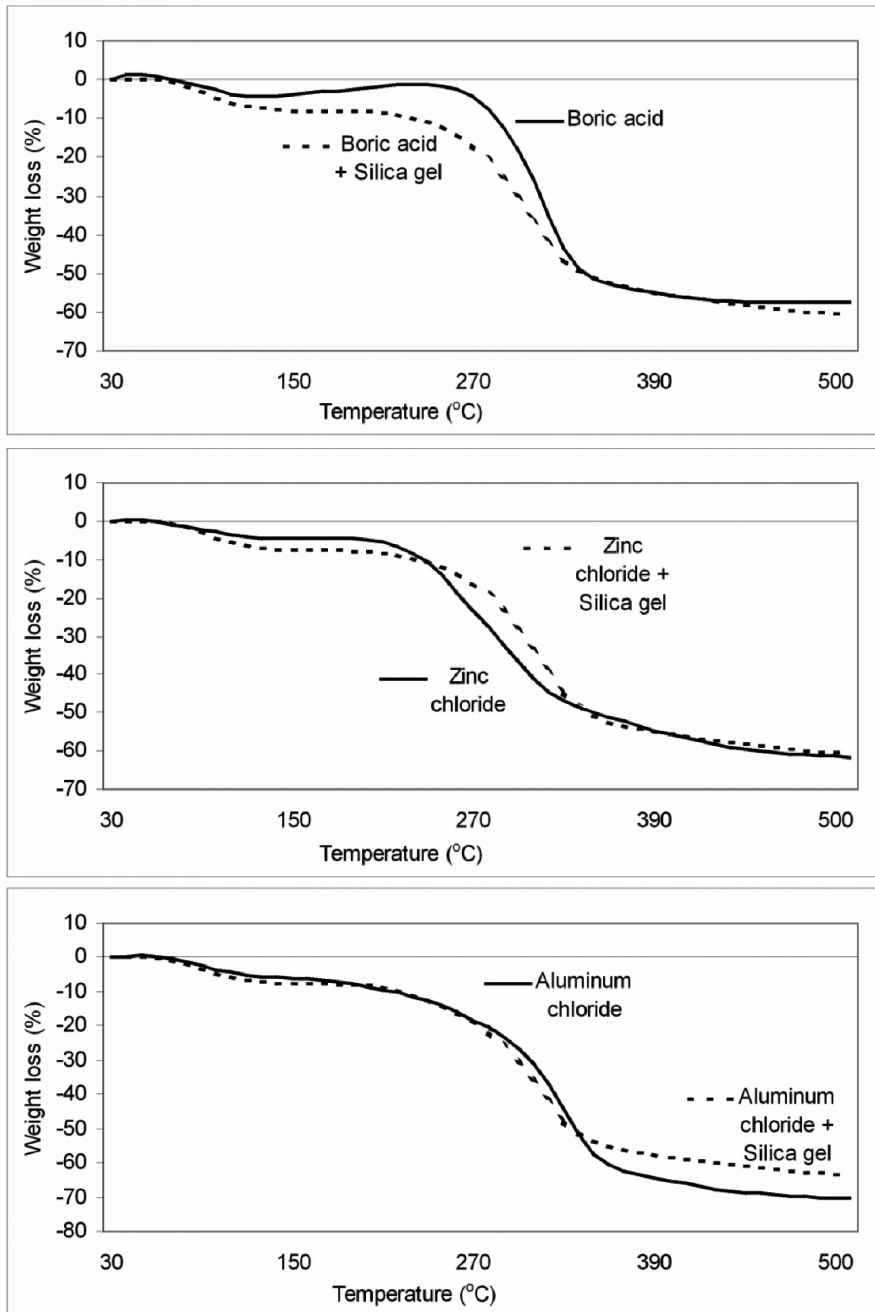


Fig. 2 : TGA of treated chestnut in argon at  $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$

## CONCLUSIONS

Silica gel treatment by using sodium silicate and sodium bicarbonate was applied in pre-impregnated wood to reduce leaching of the chemicals. It reduced amount of leachant in both Scots pine and chestnut. Amount of leachant in Scots pine was higher than that of chestnut due to Scots pine's lower density and higher permeability. Insoluble silica gel formed by precipitating silica particles with sodium bicarbonate in wood and it prevented the chemicals in the cell wall and lumens from water leaching. In addition silica gel contributed the fire retarding effect of impregnation chemicals due to its own fire retarding effect. Using silica gel lowered the total weight loss and temperature of rapid pyrolysis compared to impregnated wood without silica gel.

In conclusion, silica gel treatment can be used to decrease leaching of fire retardant water-soluble chemicals. By using this treatment water-soluble impregnation chemicals can be used in exterior applications.

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