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LEACHABILITY OF ACQ-D AFTER THREE DIFFERENT PRESERVATIVE TREATMENTS

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ABSTRACT

In this study, the ACQ-D preservative treatments of poplar wood were carried out using the method of living tree injection treatment and the leachability was examined by comparing with the traditional treatment methods, namely, immersion and pressure treatment. In addition, the effects of injection experiment on the soil environment and preservative stability in wood were examined. The results showed that the effect of injection experiment on soil environment around the treated trees was insignificant and the preservative stability of the injection treatment was the best among three methods. ACQ-D preservatives leached easily as the increase of concentration. The retention of preservative after pressure treatment was greater than that of the injection and immersion treatment, but after the leaching tests, three methods had the same preservation grade.

KEYWORDS: Living trees injection treatment, immersion treatment, pressure treatment, retention rate, preservative retention.

INTRODUCTION

Driven by concerns of environmental protection and human health, interest has risen in the development of clear wood preservative treatments to build an environmental-friendly society. Wood has been used as a construction material for thousands of years. Like other cellulose materials, wood suffers from attacked by insects or fungi. Therefore, it is necessary to pre-treat wood with preservatives before using it as building components during their services (Macchioni et al. 2016, Zhang et al. 2016). Wood preservation refers to the treatment of wood products with chemical agents to improve the ability to resist deterioration (Yu et al. 2009).

Coal tar or olive oil were the main preservatives used in the early stage of wood preservation. The simple preservation process mainly included immersion and brushing during the aged years (Gilbert et al. 1990). Vacuum pressure treatment of wood began in the 19^{th} century, while the use of preservatives was still limited to creosote and coal tar, and the treated wood became darker and smelly. Wood preservatives containing chromium and arsenic (copper, chromium and arsenic) were once the most widely used wood preservatives in the world, but their toxicity to human beings and the environment threatened them, so they were banned at the end of 2003 (Helsen and Bulck 2005). At present, copper-based wood preservatives such as amine-soluble quaternary amine copper (ACQ) have become the most widely used wood preservatives to replace copper, chromium and arsenic (CCA). However, it is likely that the ACQ-treated wood can be well protected from the attacks of insects/fungi to extend its service life. Currently, copper still is the major component in inorganic preservatives for wood preservative treatments.

As a type of wood water-borne preservative, active ingredients in ACQ are soluble in water, so the main drawback of ACQ is its poor leach resistance. Although, copper plays an essential role in protecting wood from attack of fungi, bacteria, insects and antifouling properties. The leaching of ACQ could cause human health problems, such as, liver disease, vomiting, stomach problem and muscle fatigue (Janin et al. 2011). Copper in leached ACQ could also be related to Parkinson disease, being suffered by many aged people in the World. In addition, ACQ does not contain chromium and other chemical components, so it lacks Cr^{6+} which can oxidize various lignocellulosic groups. Thus, it cannot provide more fixation sites for divalent copper ions, which can lead to the loss of divalent copper ions as active ingredients (Li et al. 2006). Although Cu^{2+} is relatively non-toxic to mammals, it is harmful to aquatic organisms. Studies have found that fish disappear completely when the lake water concentration exceeds 60 $ug \cdot L^{-1}$ of Cu^{2+}, because this concentration has exceeded the lethal dose of some juveniles (Temiz et al. 2006). Cu^{2+} also affects microbial populations in soil and sediments. High concentration of copper can inhibit the growth and photosynthesis of algae, affect the permeability of protoplasmic membranes, and make potassium ions lose from cells (Goven et al. 1994). Therefore, to solve the leach of active ingredients in water-borne wood preservatives can not only improve the preservation effect of wood preservatives, but also reduce its impact on the environment and human health. The leach resistance of preservative components is closely related to their degree of fixation in treated wood. This is because the unfixed preservative components in the treated wood are still water-soluble and easily lost to the outside world. The substances produced by a series of chemical reactions with wood components are insoluble in water, so they have better leach resistance (Sun et al. 2010).

As one of the most extensively used wood preservatives currently, alkaline copper quaternary (ACQ) comprises quaternary ammonia and copper oxide (Hasan et al. 2010). There are two common methods to penetrate the ACQ preservative into wood, namely, immersion and pressure treatments. The immersion treatment is to immerse wood in preservatives under the atmospheric pressure/temperature condition, which is a simple operation with low cost. The pressure treatment is to force preservative into the wood through an external pressure, which can deposit high preservative loading in wood after this treatment (Archer et al. 2006). The wood preservation treatment through the preservative injection of living trees is a method to improve the treatment efficiency and reduce the leaching of preservatives (Hill et al. 2004, Günther 1974) . This method is to drill or saw holes at the proper parts of the base of the tree, injecting preservative into holes, and bring preservative to the whole tree by means of sap flow of living trees. This method has advantages of simple process, no need factory equipment, less energy consumption and greatly reduced production costs. In plant transplantation and breeding, the injection method is an effective means to rescue the dangerous trees and supplement water and fertilizer of transplanted trees. It can effectively reduce the mortality during transportation and after being planted, accelerating their rapid recovery. It is also a means to quantitatively study some physiological processes of plants. Injection method can also be used for pest control, early fertilization, and growth promotion.

The leaching of copper is a major problem to copper-based wood preservatives, including ACQ-D. Under the atmospheric temperature and pressure, the fixation rate of copper preservatives is low and the amount of fixation is limited. Studies have shown that the degree of fixation of copper in preservative-treated wood was affected by factors such as temperature, time and relative humidity (Tascioglu et al. 2008). Therefore, different wood treatments processes such as wood surface treatment, heat treatment and microwave treatment could improve fixation rate (Hansson and Morén 2013). The leaching of preservatives will not only pollute the environment, but also directly affect the preservation efficiency (Tao et al. 2013). In this study, three wood preservative treatment methods, i.e., living tree injection, immersion and pressure treatment were used to preserve-treat poplar wood. The leachability of treated wood was tested, and the change of preservative loading before and after the leaching test was compared. The results can provide a theoretical basis for the research and production of poplar wood preservation.

MATERIALS AND METHODS

Materials

Ten years old living poplar trees (*Populus* L.) were cut from the Nanjing Forestry University Xiashu Forest Farm, Nanjing, Jiangsu Province, China. Tree height was about 10 m with diameter of about 0.3 m.

Poplar wood samples had an air-dried density of 0.43 $g \cdot cm^{-3}$ and processed into a size of 20 × 20 × 300 mm (radial × tangential × longitudinal), which were used for immersion and pressure treatments.

According to the standard of Wood preservatives - Ammonical copper quat (SB/T 10432-2007), the preservative used in this study was Didecyl dimethyl ammonium chloride (DDAC) with basic copper carbonate and ethanolamine. There are four types of ACQ according to the proportion of raw materials, components and the different solvents, namely ACQ-A, ACQ-B, ACQ-C and ACQ-D, as shown in Tab. 1.

Tab. 1: ACQ Wood preservative active ingredients.

Active ingredient	Index			
	ACQ-A	ACQ-B	ACQ-C	ACQ-D
Cu (Record with CuO) (%)	45.5-54.5(50)	62.0-71.0(66.7)	62.0-71.0(66.7)	62.0-71.0(66.7)
Quaternary ammonium salt (Record with DDAC) (%)	45.5-54.5(50)	29.0-38.0(33.3)	_	29.0-38.0(33.3)
Or Quaternary ammonium salt (Record with BAC) (%)	_	_	29.0-38.0(33.3)	_
- DDAC is Didecanyl dimethyl ammonium chloride. - BAC is Dodecyl dimethyl benzyl ammonium chloride. - The values in parentheses are representative values.				

The difference between ACQ-A and ACQ-B is that the proportions of CuO and DDAC in the formulation are different. The content of CuO in ACQ-B is much higher than that in ACQ-A, while the content of DDAC is lower than that in ACQ-A. Its main purpose is to reduce the cost of preservatives and improve the effectiveness of preservatives. The disadvantage of ACQ-A and ACQ-B is that the ammonia used as solvent is volatile, which makes that the treated wood has ammonia smell, affecting the operation of workers and polluting environment. Therefore, the ACQ preservative has been greatly improved by using ethanolamine instead of ammonia as a solvent to solve the problem of ammonia volatilization. The improved ACQ preservative is called ACQ-C or ACQ-D, using ethanolamine as a solvent. In this study, the ACQ-D was prepared and diluted with distilled water to three concentrations, namely, 1.5 wt.%, 1 wt.% and 0.5 wt.%.

Injection treatment of living poplar tree

Three small injection holes were drilled around the tree at a distance of 0.20 m from the ground. The angle between the injection hole and the longitudinal axis of the tree was 45°. A nail was nailed 1 m above the injection hole, and the bag containing preservative was hung on the nail as shown in Fig. 1. The valve on the bag was adjusted to drip the preservative liquid at the slowest rate, then the injection needle was inserted into the hole and started injection treatment until the tree stopped absorbing the preservative. Three concentrations (1.5 wt.%, 1 wt.% and 0.5 wt.%) of ACQ-D preservative were injected into trees, respectively. After the preservative injection, the section part of the tree with ACQ-D distribution was cut into 20 × 20 × 20 mm samples. Six samples were taken from each tree and weighed as T_1, respectively, while the samples without injection treatment were marked as T_1'.

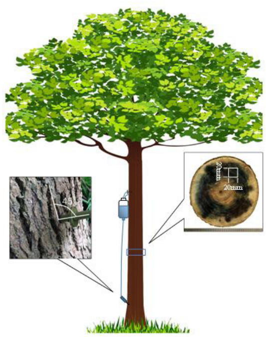

Fig. 1: Process of injecting ACQ-D and sample collecting.

Immersion treatment of poplar wood samples

The test poplar tree for the immersion treatment was selected from the same location and had the same age and similar size with the injection-treated trees, but grew up without any preservative treatment. The wood samples were cut from the tree and placed in the treatment tank, the ACQ-D preservative was added and glass blocks were placed on the test wood samples to make the samples completely immersed in the liquid preservative, and the opening of the tank was sealed with a plastic film. After 72 h immersion, the test wood samples were taken out and the wood surface was wiped with filter paper. Six test wood samples were treated with each ACQ-D concentration. After the immersion treatment, at the distance of 120 mm in the longitudinal direction of each test wood sample, three samples (20 × 20 × 20 mm) were continuously taken out and weighed as T_2, respectively. The samples without immersion treatment were weighted and marked as T_2'.

Pressure treatment of poplar test wood

Test wood samples were cut from the non-preservative-treated poplar tree and placed in a sealed treatment tank, then, the tank was vacuumed to 0.08-0.09 MPa for 30 min and added ACQ-D preservative, pressurized to 0.8 MPa and kept for 3 hours. After the pressure released, the tank was vacuumed again to 0.08-0.09 MPa for 10 min to drain out the excess preservative in the samples. After the pressure treatment, the test wood samples were taken out, and wiped the wood surfaces with filter paper. Six test wood samples were treated with each ACQ-D concentration. After the pressure treatment, at the distance of 120 mm in the longitudinal direction of each test sample, three samples (20 × 20 × 20 mm) were continuously taken and weighed as T_3, respectively. The samples without pressure treatment were weighted and marked as T_3'.

Environmental quality assessment of living tree injection treatment

Before and after the preservative injections, the soil around the tree roots was taken to detect the contents of heavy metals, e.g., copper, in which, sixty soil samples were obtained for each tree. Based on the standard of determination of available in forest soil (LY/T 1260-1999), the copper content in the soil was measured accordingly. According to the standard of Environmental quality standard for soils (GB15618-1995), the soil quality was examined before and after the preservative injections. Ten gram soil sample was dried in an oven with a temperature of 103°C for 24 h, placed in a bottle with 50.0 mL of 0.1 mol.L-1 hydrochloric acid and shaken for 1.5 h with a vibration machine, then the clear liquid was achieved by filtering the soil. Using an atomic absorption spectrometer, the copper concentration was examined and the soil copper content was calculated by Eq. (1):

$$M_{Cu} = c \times r \quad (1)$$

where: M_{Cu} is the calculated soil copper content, (mg·kg^{-1}); c is the copper concentration measured by the Atomic absorption spectrometry, (ug·mL^{-1}); r = (milliliter quantity of extracting agent)/(soil weight)$^{-1}$.

ACQ-D Leaching rate tests

Based on the standard Method for accelerated evaluation of preservative leaching (AWPA E11-16) and the Standard laboratory method of determining the leachability of wood preservatives (GB/T 29905-2013), for each preservative treatment case, six preservative-treated samples were immersed in a 500 ml beaker with distilled water that was stirred using a magnetic stirrer. The distilled water in the beaker was substituted by fresh water every 6 h, 24 h, 48 h and then fresh-substituted every 48 h for 14 days. Using the Atomic absorption spectrometry, the copper content of each test was determined. The ACQ-D leaching rate was calculated according to Eq. (2):

$$L = \left(\frac{0.18 \times c_1}{c \times m_G \times 1000}\right) \times 100\% \quad (2)$$

where: c_1 is the copper concentration in the determined filtrate, (mg·L^{-1}); c is the mass fraction of components to be measured in ACQ-D preservative (66.7% for ACQ-D) (%); m_G is the quantity of absorbed ACQ-D of sample in preservation treatment (T_i-T_i', i = 1, 2, 3), (g)

ACQ-D loading measurement

Based on the standard methods for the analysis of waterborne wood preservatives (GB/T 23229-2009), before and after the leaching tests, the different preservative loadings were examined. The powder samples were obtained by grinding the wood specimens with different treatments and oven-dried at 103°C for 24 h. The 0.5 g powder sample in the beaker that had 30 ml distilled water and 5.0 g concentrated sulfuric acid was heated to 75°C, held this temperature and shacked for 30 min for dissolving the powder sample. Then the solution with dissolved powder was poured into a 200 ml flask with distilled water for filtration. The filtrate elements from the filtration was examined using the Atomic Absorption Spectrometry and the preservative loadings of copper in wood specimens were estimated according to Eq. (3):

$$R_{Cu} = 0.2 \times c_2 \times d/W_1 \qquad (3)$$

where: R_{Cu} is the total copper loading of the preservative, (kg·m^{-3}); c_2 is the concentration of preservative in the filtrate that was measured using the Atomic Absorption Spectrometry, (mg·L^{-1}); d is the density of wood specimen, (g·cm^{-3}); W_1 is the amount of powder, (g).

EDX analysis

The leaching test samples (20 ×20 × 20 mm) were cut into two parts (20 × 20 × 10 mm each). One part of the sample was examined by EDX before the leaching test and the other one was examined by EDX after the leaching test to observe the effect of the leaching test on the preservative loading. Three kinds of preservation-treated samples with different concentrations were sliced by the slicing machine. The transverse section was observed and the thickness of the section was 20 um. All the sections to be observed were sprayed with a layer of metal by a vacuum coater to increase conductivity. Then the section was observed by the Quanta 200 Scanning electron microscopy energy dispersive spectrometer (EDX). The acceleration voltage was 15 kV and the working distance was 13 mm.

RESULTS AND DISCUSSION

Environmental quality assessment of living tree injection treatment

The flow of preservative in the tree trunk is mainly driven by transpiration of leaves. Since wood is a porous material, there are many small gaps among wood cells. Thus, the ACQ-D preservative went upward to the tree-top direction at daytime due to the transpiration, while it also flowed downward to the root direction at night time due to the condensation, which might cause the tree roots to contain a certain amount of preservatives. As a result, the ACQ-D preservatives could pollute the soil around the treated tree. Thus, it is necessary to test whether this injection treatment would cause environmental pollution. Based on the standard of soil environmental quality (GB/T 15618-1995), there are three grades of soil according to their pH values. In this study, the pH values of the soil around the preservative injected trees were range from 6.71 to 7.38, which was specified to be the acidic grade. Based on the standard of GB15618-1995, it was classified into type II soil, which designated that the soil quality was basically not harmful and pollution for plants and the environment.

As shown in Fig. 2, such as 1.5 wt.% ACQ-D treated samples, the experimental results indicated that, before the preservative injection, the soil copper contents were ranged from 41.6 to 67.1 mg·kg^{-1}, while after the preservative injections the copper contents were between 61.1 and 89.1 mg·kg^{-1}. For 1 wt.% and 0.5 wt.% ACQ-D treated samples, the copper contents were 55.5-81.2 mg·kg^{-1} and 43.9-79.7 mg·kg^{-1}. A slight increase in soil copper content was observed before and after the injection treatment. However, soil copper contents after the injection treatments were still lower than the acceptable value (i.e. 100 mg·kg^{-1}) for type II soil specified by the standard of GB15618-1995, which indicated that the preservative injection insignificantly affect the soil environment. This acceptable value (i.e. 100 mg·kg^{-1}) was also confirmed by Yang et al. (2007), which suggested that the low concentration of copper (< 100 mg·kg^{-1}) in soil would benefit the growth of trees.

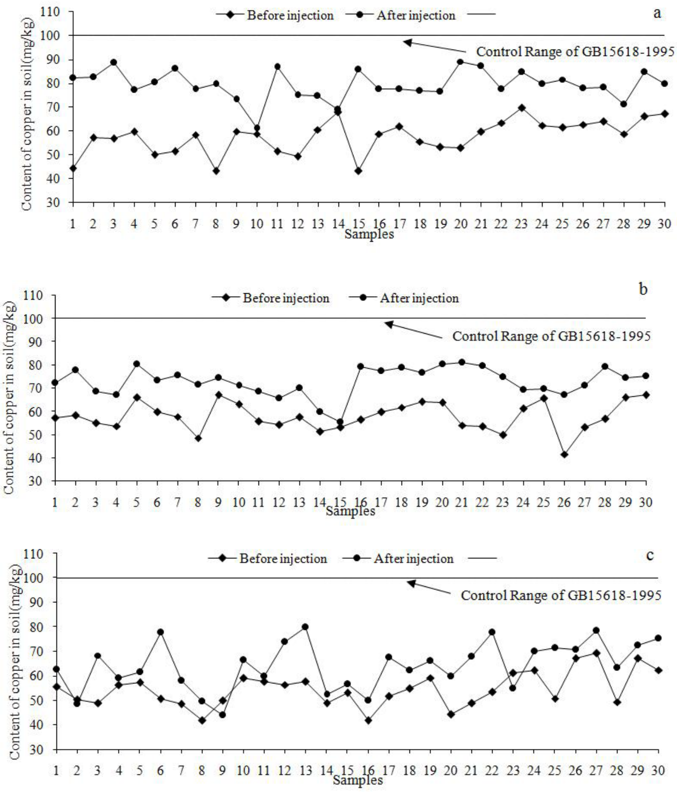

Fig. 2: Changes of copper content in soil before and after injection treatments: (a) 1.5 wt.% ACQ, (b) 1.0 wt.% ACQ and (c) 0.5 wt.% ACQ.

Leaching of ACQ-D with different preservation treatments

The leaching rates of ACQ-D with time using the three preservative treatment methods, namely, injection, immersion and pressure treatments are presented in Fig. 3. The curves of the treated wood during the leaching process could be divided into three different stages. In the early stage (0-24 h), the leaching rate of ACQ-D all increased significantly throughout this stage, which was the main period of leaching, showing that the large amount components of ACQ-D leached from the treated wood. The reason was that ACQ-D was a waterborne preservative, and the effective components of ACQ-D would leach with the water during the service of the wood products after the preservation treatment (Lin et al. 2009). In the second stage (24-96 h), the leaching rate began to decrease. In the last stage (after 96 h), the leaching rate was progressively reduced and approached to zero as shown in Fig. 3. The reason may be described as follows. The fixation state of ACQ-D could be divided into physical fixation and chemical fixation. Through the physical fixation, such as the dipole-dipole and ion-dipole interaction between cellulose and copper in ACQ-D (Yu et al. 2011), the preservatives in the wood were easy to leach with the water evaporation and the change of environments. The copper in ACQ-D reacted with hemicellulose and lignin in a chemically bonded manner. This fixation state made the preservative more stable in the wood and had a better antiseptic effect (Zhang and Kamdem 2000).

For three different preservative treatment methods, the leaching rate of ACQ-D after injection treatment was lower than that of the immersion treatment and pressure treatment. At the same time, the concentration of the preservative also affected its fixation in wood. As shown in Fig. 3, the preservative would be easier to leach with the increase of concentration. This result also had a good agreement with the results of published articles (Liu et al. 2019, Yu et al. 2015).

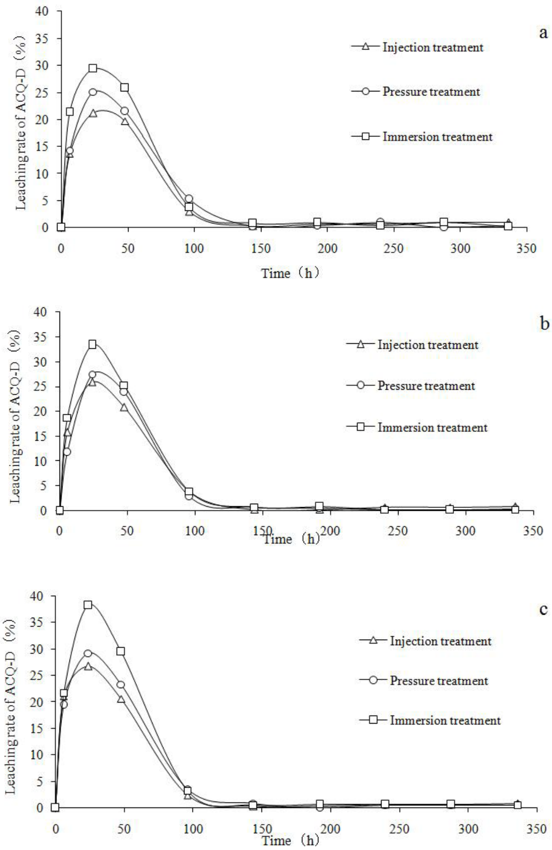

Fig. 3: Leach rate of ACQ-D after three different preservative treatments: (a) 0.5 wt.% ACQ, (b) 1.0wt.% ACQ and (c) 1.5 wt.% ACQ).

Retention of preservative in wood

Preservative retention is an important index for evaluating wood preservation consequence and also the basis for classifying the use grade of wood preservation. The preservative retention of three different treatments before and after the leaching tests are shown in Tab. 2. As shown in the table, it can be seen that the higher the concentration, the greater the preservative retention can be achieved, which was caused by the active ingredients contained in ACQ-D. Before leaching tests, such as 1.5 wt.% concentrations of ACQ-treated samples, preservative retention of pressure treatment was the highest, which can reach 4.89 kg·m^{-3}. According to the standard of GB/T 31761-2015 Alkaline copper quat (ACQ) of preservative pressure-treated wood, the wood treated by this method can meet the requirements of Grade C4. The preservative retention of injection treatment and immersion treatment were 3.78 kg·m^{-3} and 3.85 kg·m^{-3}, respectively, which could meet the requirements of Grade C3. However, preservative retention of the pressure-treated wood after leaching test was reduced to 3.07 kg·m^{-3}, and the loss amount of preservative was the most one among three methods. The preservative retention of immersion and injection treatments after leaching tests were 3.11 kg·m^{-3} and 3.16 kg·m^{-3}, respectively, which could still meet the requirements of Grade C3. The same trends could be found for the samples treated with 1 wt.% and 0.5 wt.% concentrations of ACQ-D, but preservative retention decreased slightly. Therefore, it could be seen that after leaching tests, preservative retention for three treatment methods reached the same grade, and injection treatment method could make the ACQ-D more stable in wood.

Tab. 2: Preservative retention of different preservative treatments.

Preservation treatment	Concentration (wt.%)	Retention of preservative before leaching (kg·m^{-3})	Retention of preservative after leaching (kg·m^{-3})
Injection treatment	1.5	3.78	3.16
	1	2.96	2.35
	0.5	1.67	1.42
Immersion treatment	1.5	3.85	3.11
	1	2.81	2.11
	0.5	1.51	1.09
Pressure treatment	1.5	4.89	3.07
	1	3.66	2.47
	0.5	2.38	1.67

EDX analysis

Copper is the major component in ACQ-D preservative to protect wood against the attacks of insects and/or fungi. The copper content examination can be helpful for evaluation of the efficiency of wood preservation treatment. This experiment mainly investigated the leaching of preservative after different preservation treatments, the EDX examination of only 1.5 wt.% concentration preservative after treatment is illustrated as an example (Fig. 4).

As sown in Fig. 4, after the preservation treatment, the ACQ-D preservative entered the wood cell wall and the distribution of copper was not even, in which, some parts were concentrated. Previous research also confirmed this result (Liu et al. 2019). It can be found that the distribution of copper in the cell wall changed after leaching test. In addition, after leaching test, the content of copper decreased, especially under the pressure treatment. The atomic percentage of copper in the cell wall was examined by EDX (Tab. 3) and it was shown that the content of copper decreased from 51.1% to 17.8%. Compared with the pressure treatment, the copper was more stable after the injection treatment, followed by the immersion treatment. This trend was basically consistent with the results of the preservative retention tests.

Leaching of copper is a major problem to copper-based wood preservatives, including ACQ-D. At room temperature and under normal pressure, the fixation rate of copper wood preservatives was slow and the amount of fixation was limited. Studies have shown that the degree of fixation of copper in preservative wood is affected by factors such as temperature, time and relative humidity (Ye and Morrell 2015). Therefore, different post wood treatments such as wood surface treatment, heat treatment and microwave treatment can effectively improve fixation rate (Ahmed et al. 2013) .

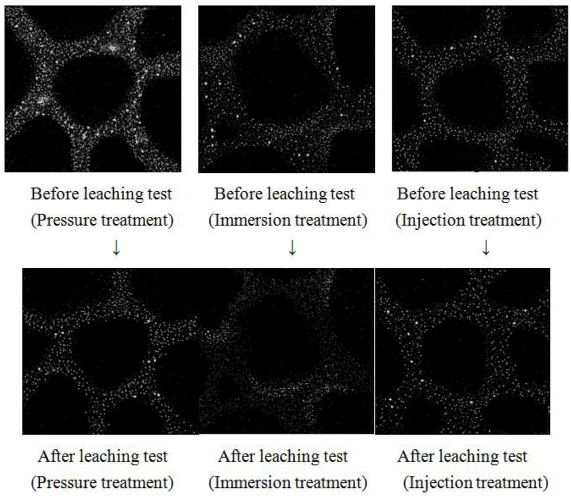

Fig. 4: EDX imagies of 1.5 wt.% ACQ-D treated samples.

Tab. 3: Percentage of Cu element content before and after leach test.

Concentration of ACQ-D		Pressure treatment	Immersion treatment	Injection treatment
1.5%	Before leaching	51.1 %	31.2 %	36.7 %
	After leaching	17.8 %	15.6 %	16.9 %
1%	Before leaching	28.6 %	19.5 %	21.6 %
	After leaching	13.1 %	12.2 %	14.8 %
0.5%	Before leaching	22.3 %	10.6 %	11.7 %
	After leaching	8.6 %	6.3 %	7.2 %

CONCLUSIONS

Poplar wood and trees were treated by three different preservation treatment methods. In the preservative leaching tests, the leaching rate of ACQ-D after injection treatment was lower than that of immersion and pressure treatments. The higher the concentration of the solution, the easier it would leach. Before the leaching test, the preservative retention of poplar wood treated by the pressure treatment was the highest, but after the leaching test, the preservative retention of samples treated by three preservation treatment methods reached the same grades. After the EDX analysis, it was found that the distribution of copper in cell wall changed after the leaching test. In addition, after the leaching test, the content of copper decreased, especially for the pressure treatment. The examination of the soil around the injection-treated tree before and after treament indicated that the living tree preservative injection treatment did not cause significant quality change for the soil environment.

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