

Effect of pyrolysis on solvent extractability of toxic metals from chromated copper arsenate (CCA)-treated wood

Tomo Kakitani^a, Toshimitsu Hata^{a,*}, Takeshi Kajimoto^b, Yuji Imamura^a

^a Wood Research Institute, Kyoto University, Uji, Kyoto 611-0011, Japan

^b Industrial Technology Center of Wakayama Prefecture, 60 Ogura, Wakayama-shi 649-6261, Japan

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Abstract

Solvent extraction was conducted to investigate the behavior of toxic metals in chromated copper arsenate (CCA)-treated wood and its pyrolysis residue. Four kinds of solvents, along with sulfuric and phosphoric acid as mineral agents, and citric and oxalic acid as complexing agents, were used. The CCA elements from the wood treated without pyrolysis were easily extracted. However, only a small amount of CCA elements were extracted from the pyrolyzed residue, even under very low pH conditions. The toxic elements in the pyrolyzed wood showed high stabilization against the solvent extraction. Pyrolysis strongly immobilized the toxic elements in the residue. Therefore, pyrolysis is not suitable as a pretreatment.

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1. Introduction

In recent years, the disposal of chromated copper arsenate (CCA)-treated wood wastes has become a serious environmental problem throughout the world. Combustion and landfill have often been used as practical methods to dispose of this type of wood waste. However, combustion and landfill of CCA-treated wood wastes suggested may result in the release of environmental pollutants. For example, some studies result that the combustion of CCA-treated wood wastes causes the release of arsenic into the atmosphere [1–3]. On the other hand, when the wood is landfilled, determining if the toxic metals have diffused from the waste wood into the soil and groundwater is difficult. Therefore, before waste wood is disposed in a landfill, toxic metals should be removed from the wood. Thus, developing a technique for removing toxic metals from CCA-treated waste wood before disposal in a landfill is imperative.

Among possible methods for purifying CCA-treated waste, solvent extraction has received much attention in recent years [4–8]. The advantage to this technique is that the purified product can either be landfilled or safely used as a building, manufacturing material or energy source without environmental risk. Kanjo et al. [6] reported that 0.25N sulfuric acid was a suitable solvent, and that the ideal temperature range required for the complete removal of the CCA elements was 90–100 °C. Phosphoric acid has been reported as one of the most effective solvents to recover CCA elements from the hazardous ash of the treated wood [9]. Clausen et al. [4,5,10] effectively used oxalic acid as a complexing reagent for recovering the toxic reagents. However, the complex characteristics of the CCA elements in the wood prevent the use of these techniques in commercial applications. In addition, because the purpose of the CCA treatment itself is to prevent weathering, the bond between the CCA salts and the wood is very strong, and the solvent extraction of CCA elements requires a great deal of time.

In previous studies, we investigated whether pyrolysis prior to solvent extraction would be a useful technique for decomposing the strong bonding with wood and/or immobilizing CCA elements in the residue [11]. In the present study,

* Corresponding author. Tel.: +81-774-54-0631;

fax: +81-774-38-3678.

E-mail address: n50120@sakura.kudpc.kyoto-u.ac.jp (T. Hata).

we applied pyrolysis before solvent extraction in order to investigate: (1) the behavior of toxic metals in CCA-treated wood and in the pyrolysis residue during solvent extraction; (2) the effect of pyrolysis on the extractability of the CCA elements; and (3) the mechanism of changes in the mobility of the toxic metals in the pyrolysis residue.

2. Experiment

2.1. Sample preparation

The sample used in this experiment was western hemlock (*Tsuga heterophylla*) treated with type III CCA reagent (chromium as CrO₃, 45–51%; copper as CuO, 17–21%; arsenic as As₂O₅, 30–38%; JIS-K1570, 1998) supplied by Koshii Mokuzai Co. (Osaka, Japan). After impregnation of the CCA solution, the treated wood was dried at room temperature for 3 weeks. The CCA-treated wood pieces were then broken into chips and milled to a powder with a particle size of under 20 mesh. The powder was then mixed sufficiently to minimize the non-uniform distribution of the CCA salts in the sample. In addition to these treatments, post-heat treatment (drying at 60 °C for 2 weeks) was also conducted in order to complete the reaction of the chemicals in the wood.

2.2. Pyrolysis methods

Twenty five grams of CCA-treated wood powder was pyrolyzed in an N₂ atmosphere using an electric furnace, 0.75 m high and 0.80 m wide, equipped with a 100 ml volumetric pear-shaped glass flask reactor. The pyrolysis conditions were fixed at a temperature of 300 °C and time of 60 min. We have determined that the loss of CCA elements is negligible under the conditions of 300 °C and 60 min. The heating and N₂ gas flow rates were adjusted to 4 °C/min and 12.5 ml/s, respectively. After the reaction, the reactor was removed from the furnace and allowed to cool to room temperature naturally.

2.3. Solvent extraction

Solvent extraction was conducted for both un-pyrolyzed and pyrolyzed CCA-treated wood powder. The samples were transferred to a glass reactor, and solvents were gently stirred into the samples. The solvents selected in this experiment were sulfuric and phosphoric acid as mineral agents, citric and oxalic acid as complexing agents, and distilled water as a control. The ratio of the solids (wood powder) to the liquid (extraction solvent) was fixed at 1 g to 100 ml. The solvent concentrations were adjusted to 0.5 or 1.0N, and the extraction temperatures to 25 or 75 °C. The duration of extraction was 1, 2, 3, or 6 h. Mixtures of sample and solvent were removed from the reactor at 1, 2, 3, and 6 h, then rinsed repeatedly using 300 ml of dis-

tilled water that was filtered through glass-fiber filter paper (Advantec-GA100; Toyo Roshi Kaisha Ltd., Japan) using an aspirator.

2.4. Chemical analysis

After the solvent extraction, the amounts of arsenic, copper, and chromium in the samples of wood powder and pyrolysis residue were measured by X-ray fluorescence (XRF) after dissolving them in HNO₃. The 0.1 g samples of oven-dried wood or pyrolysis residue were transferred to a flask, and 65% HNO₃ (a pure reagent for toxic metal analysis; Nacalai Tesque, Kyoto, Japan) was added. The flask was heated on a sand bath without loss of CCA elements during the dissolution process, and nitric acid was added repeatedly until the sample was completely dissolved. The CCA elements in the liquid specimen were analyzed using XRF spectrometry (JSX-3220 Element Analyzer; JEOL Ltd., Tokyo, Japan) and the calibration curve method, in which the concentration of each element is determined according to the intensity of the fluorescent radiation in comparison with that for standard solutions of arsenic, copper, and chromium. All measurements were performed in triplicate, and the results are presented as the mean value.

3. Results and discussion

3.1. Behavior of CCA elements during extraction of treated wood with distilled water

Table 1 shows the CCA elements content in the un-pyrolyzed and pyrolyzed wood powder after extraction with distilled water. The CCA elements in both the un-pyrolyzed and pyrolyzed samples showed high resistance to extraction with distilled water, reflecting the high

Table 1
Percentage CCA elements contents after solvent extraction of un-pyrolyzed and pyrolyzed wood with distilled water

| Time (h) | 25 °C | | | 75 °C | | |
|---------------------------|--------|--------|--------|--------|--------|--------|
| | As (%) | Cu (%) | Cr (%) | As (%) | Cu (%) | Cr (%) |
| Distilled water (control) | | | | | | |
| 1 | 91.9 | 93.2 | 108.0 | 75.0 | 95.7 | 93.9 |
| | 91.4 | 95.4 | 95.7 | 91.1 | 98.5 | 101.0 |
| 2 | 57.0 | 79.2 | 88.8 | 50.1 | 59.2 | 84.6 |
| | 104.5 | 100.6 | 108.0 | 84.4 | 87.2 | 101.2 |
| 3 | 83.5 | 103.0 | 83.6 | 63.3 | 88.4 | 87.4 |
| | 95.9 | 94.5 | 92.9 | 88.9 | 99.1 | 91.3 |
| 6 | 81.2 | 77.6 | 90.6 | 63.7 | 83.5 | 89.6 |
| | 102.6 | 110.9 | 98.7 | 99.7 | 110.4 | 115.6 |
| 24 | 77.7 | 98.2 | 94.1 | – | – | – |
| | 93.1 | 98.1 | 93.6 | – | – | – |

Upper value: un-pyrolyzed wood; lower value: pyrolyzed wood.

performance of CCA-treated wood in resisting weathering. After 24 h extraction, 22.3% arsenic, 1.8% copper, and 5.9% chromium were extracted from un-pyrolyzed sample, and those from pyrolyzed wood were 6.9% arsenic, 1.9% copper, and 6.4% chromium. Thus, the amount of arsenic leached from the pyrolyzed sample was roughly one-third of that leached from the un-pyrolyzed sample, indicating that pyrolysis markedly lowers the extractability of arsenic.

3.2. Behavior of CCA elements during the solvent extraction of treated wood

Table 2 shows the CCA elements contents in the samples after extraction with 0.5N sulfuric, phosphoric, citric, or oxalic acid. There was a minor increase of CCA elements in the first hour of leaching, and thereafter, the CCA elements in the wood decreased or remained constant. Some of the elements show percentages greater than 100%. This is due to the loss of fine particles, which is a result of the decomposition of wood powder by chemical or mechanical processing during extraction. In the first hour of leaching the decomposition of wood can proceed more rapidly than dissolution (or complexing) of metals. However, in the second hour of leaching, dissolution (or complexing) of metals proceeds more rapidly than the decomposition of wood. Therefore, some of the CCA elements in wood show a minor increase in the first hour of leaching.

As seen from the table, higher extraction temperature and longer duration of extraction tended to lead to enhanced removal of elements. Among the three elements measured, chromium had the lowest extraction rate. The high resistance to extraction in chromium may be due to the strong bonding between lignin and chromium [12–15]. On the whole, the 0.5N solvents used in this experiment were relatively inefficient at removing all of the elements in 6 h.

As regards the difference between the un-pyrolyzed and pyrolyzed samples, the results in Table 2 demonstrate that pyrolyzed wood had dramatically lower leachability than un-pyrolyzed wood. This phenomenon was observed not only when using sulfuric and phosphoric acids for the extraction, but also when using citric and oxalic acids. The rates of extraction in un-pyrolyzed wood by sulfuric, phosphoric, citric, and oxalic acid ranged from 75.7 to 99.5% by treatment at 75 °C for 6 h. On the other hand, those in pyrolyzed wood ranged from 4.0 to 39.2% under the same conditions. Thus there was a clear difference in the extractability of CCA elements between un-pyrolyzed and pyrolyzed wood, indicating the drastic changes in the extractability of toxic elements after pyrolysis.

Table 3 shows the CCA elements contents in the samples after extraction with 1N sulfuric, phosphoric, citric, or oxalic acid. As was observed in Table 2, higher extraction temperature and longer duration of extraction led to greater leaching of the elements. In addition, the higher concentration of extractant removed the elements more efficiently than

Table 2
Percentage CCA elements contents after 0.5N solvent extraction of un-pyrolyzed and pyrolyzed wood

| Time (h) | 25 °C | | | 75 °C | | |
|-----------------|--------|--------|--------|--------|--------|--------|
| | As (%) | Cu (%) | Cr (%) | As (%) | Cu (%) | Cr (%) |
| Sulfuric acid | | | | | | |
| 1 | 55.0 | 28.9 | 71.8 | 0.0 | 11.6 | 18.3 |
| | 97.8 | 79.3 | 108.9 | 115.1 | 92.2 | 112.2 |
| 2 | 22.0 | 29.2 | 47.5 | 18.8 | 37.2 | 27.2 |
| | 82.1 | 59.4 | 98.1 | 87.1 | 59.7 | 95.2 |
| 3 | 11.6 | 22.1 | 24.2 | 0.8 | 0.6 | 21.5 |
| | 102.3 | 83.3 | 101.6 | 108.9 | 107.9 | 103.5 |
| 6 | 4.3 | 14.5 | 27.3 | 1.6 | 0.8 | 7.8 |
| | 84.5 | 66.9 | 93.3 | 65.9 | 63.91 | 81.7 |
| Phosphoric acid | | | | | | |
| 1 | 56.0 | 34.9 | 76.2 | 16.5 | 24.7 | 42.1 |
| | 110.2 | 92.7 | 113.3 | 59.8 | 61.0 | 73.4 |
| 2 | 37.1 | 9.0 | 61.3 | 10.5 | 29.2 | 33.7 |
| | 93.8 | 70.8 | 93.7 | 62.6 | 48.4 | 76.3 |
| 3 | 42.7 | 24.1 | 69.7 | 15.3 | 27.6 | 21.2 |
| | 85.7 | 61.6 | 86.2 | 78.0 | 65.0 | 80.7 |
| 6 | 36.0 | 31.5 | 65.2 | 7.3 | 19.9 | 24.3 |
| | 85.9 | 78.5 | 81.9 | 61.3 | 60.8 | 80.7 |
| Citric acid | | | | | | |
| 1 | 46.1 | 14.7 | 63.2 | 17.9 | 12.0 | 44.7 |
| | 96.5 | 74.1 | 101.8 | 81.0 | 70.4 | 92.1 |
| 2 | 34.7 | 2.4 | 53.2 | 13.2 | 22.7 | 39.0 |
| | 80.7 | 77.7 | 80.8 | 84.6 | 46.7 | 79.2 |
| 3 | 32.1 | 7.4 | 63.8 | 4.6 | 23.0 | 23.0 |
| | 99.2 | 70.1 | 102.4 | 71.9 | 61.2 | 82.4 |
| 6 | 49.6 | 20.9 | 71.6 | 0.5 | 18.0 | 9.1 |
| | 80.1 | 57.3 | 82.0 | 72.8 | 70.9 | 96.0 |
| Oxalic acid | | | | | | |
| 1 | 30.0 | 18.5 | 55.8 | 12.5 | 27.7 | 28.8 |
| | 108.3 | 81.9 | 120.1 | 72.0 | 73.5 | 88.4 |
| 2 | 24.1 | 13.8 | 47.8 | 10.6 | 24.5 | 14.4 |
| | 91.0 | 51.0 | 94.6 | 59.1 | 71.8 | 76.0 |
| 3 | 12.1 | 14.2 | 19.4 | 0.0 | 10.6 | 9.5 |
| | 97.4 | 86.0 | 101.4 | 48.8 | 85.7 | 78.9 |
| 6 | 4.2 | 26.1 | 21.4 | 0.0 | 2.1 | 19.2 |
| | 99.4 | 63.6 | 97.5 | 67.1 | 62.6 | 68.1 |

Upper value: un-pyrolyzed wood; lower value: pyrolyzed wood.

the lower concentration used in Table 2. Here, the amount of leached elements after pyrolysis was also markedly lower than that of the un-pyrolyzed sample. In addition, the mobility of the CCA elements in the residue was lower even under very low pH conditions.

With respect to the effects of different solvents on the extractability of CCA elements, both sulfuric and phosphoric acid extraction resulted in similar amounts of removed arsenic, copper, and chromium. On the other hand, citric acid extracted copper preferentially, and oxalic acid extracted chromium preferentially. This is probably due to the for-

Table 3
Percentage CCA elements contents after 1N solvent extraction of un-pyrolyzed and pyrolyzed wood

| Time (h) | 25 °C | | | 75 °C | | |
|------------------------|--------|--------|--------|--------|--------|--------|
| | As (%) | Cu (%) | Cr (%) | As (%) | Cu (%) | Cr (%) |
| Sulfuric acid | | | | | | |
| 1 | 19.3 | 28.8 | 54.3 | 1.7 | 16.0 | 16.4 |
| | 100.6 | 95.7 | 112.5 | 89.9 | 48.4 | 87.1 |
| 2 | 18.5 | 32.2 | 60.0 | 9.4 | 9.1 | 33.8 |
| | 109.2 | 105.3 | 106.1 | 56.4 | 49.2 | 83.6 |
| 3 | 6.4 | 17.8 | 30.3 | 0.3 | 20.0 | 22.4 |
| | 103.8 | 93.8 | 99.4 | 79.7 | 82.9 | 85.6 |
| 6 | 12.9 | 21.0 | 16.7 | 2.1 | 0.0 | 23.0 |
| | 75.9 | 79.5 | 90.3 | 68.1 | 60.9 | 72.2 |
| Phosphoric acid | | | | | | |
| 1 | 16.5 | 33.0 | 35.9 | 3.7 | 0.0 | 24.9 |
| | 100.3 | 78.6 | 93.1 | 72.4 | 71.7 | 85.1 |
| 2 | 6.1 | 6.7 | 26.3 | 1.9 | 3.8 | 18.7 |
| | 99.7 | 95.1 | 95.9 | 73.7 | 63.5 | 91.6 |
| 3 | 6.5 | 18.1 | 30.9 | 7.1 | 7.5 | 18.9 |
| | 99.9 | 76.2 | 106.7 | 67.6 | 47.4 | 84.4 |
| 6 | 5.8 | 1.7 | 27.2 | 5.5 | 17.4 | 14.6 |
| | 96.2 | 61.9 | 103.0 | 62.9 | 68.5 | 83.6 |
| Citric acid | | | | | | |
| 1 | 39.5 | 16.7 | 62.0 | 9.1 | 30.5 | 29.7 |
| | 110.6 | 81.7 | 112.7 | 83.0 | 76.5 | 97.5 |
| 2 | 33.7 | 5.8 | 68.6 | 9.5 | 12.0 | 19.9 |
| | 108.8 | 70.4 | 106.7 | 77.7 | 70.4 | 76.4 |
| 3 | 31.1 | 19.2 | 60.9 | 12.6 | 31.1 | 30.2 |
| | 106.0 | 93.5 | 111.0 | 93.0 | 87.6 | 93.1 |
| 6 | 37.0 | 29.7 | 50.3 | 5.6 | 21.8 | 13.8 |
| | 101.7 | 88.9 | 90.5 | 70.9 | 70.8 | 83.9 |
| Oxalic acid | | | | | | |
| 1 | 13.5 | 26.9 | 46.0 | 3.4 | 15.7 | 8.8 |
| | 108.1 | 71.0 | 98.1 | 63.3 | 63.8 | 65.5 |
| 2 | 13.2 | 13.4 | 31.9 | 12.9 | 21.6 | 18.0 |
| | 88.8 | 55.9 | 99.2 | 64.5 | 90.1 | 84.1 |
| 3 | 7.5 | 15.5 | 25.5 | 0.0 | 10.1 | 4.3 |
| | 99.7 | 85.2 | 95.7 | 47.0 | 56.9 | 68.7 |
| 6 | 1.3 | 11.4 | 17.3 | 0.0 | 7.1 | 10.0 |
| | 90.5 | 79.0 | 99.0 | 42.8 | 65.8 | 64.0 |

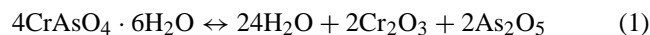
Upper value: un-pyrolyzed wood; lower value: pyrolyzed wood.

mation of soluble complexes such as copper–citrate and chromium–oxalate. From the results of the 1N extraction, sulfuric, phosphoric ($pK_a = 2.12$), and oxalic ($pK_a = 1.23$) acid removed almost all the elements in 6 h. However, citric ($pK_a = 3.15$) acid was less effective than the other solvents, which was attributed to its higher lower dissociation constant. It is, however, interesting to note that the extractability of copper in the pyrolyzed samples was lower than that of the other elements only in the case of oxalic acid extraction, which was attributed to the formation of copper–oxalate salt with less solubility. The formation of copper–oxalate may

have been accelerated by the higher reaction temperature and a self-preservation mechanism in the carbon structure of carbonized wood.

3.3. Pyrolysis effect

From the results of the solvent extraction of the pyrolysis residue, some characteristic tendencies were observed. The solubility of all CCA elements in the pyrolysis residues became markedly lower than those of the un-pyrolyzed sample. However, when comparing the solubilities of the three elements, only copper maintained a relatively high solubility. Arsenic was the easiest of the three elements to extract from the un-pyrolyzed sample. After pyrolysis, copper, rather than arsenic, showed the lowest resistance to leaching among the three elements. This finding suggests that the characteristics of arsenic and chromium were changed during pyrolysis. The decomposition of $CrAsO_4$, the main compound of CCA salts, during pyrolysis has been proposed to proceed as follows [16]:



Focusing on the changes of the states of chromium, chromium trioxide can be formed during pyrolysis in Eq. (1). If chromium trioxide is formed in the residue by heating, it is very hard to extract because of its high stability and insolubility in water, acid, and bases. Therefore, it was considered that the pyrolysis effect expressed in chromium was caused by the formation of chromium trioxide.

Considering the pyrolysis of As_2O_5 , the reduction and oxidation may occur as follows:



It is possible to have arsenic reduced from the pent- to tri-valent state in the reductive atmosphere, while the arsenic valences change from (V) to (III). The solubility of arsenic trioxide is lower than that of pentoxide. When arsenic trioxide is formed, the extractability of the arsenic is lowered. Furthermore, arsenic pentoxide can react with heavy metals, such as chromium and copper in this experiment, during pyrolysis, forming stable metal arsenates such as copper arsenate or chromium arsenate. In our previous study of measuring the metal compounds in pyrolysis residues by transmission electron microscopy, the CCA compounds and their reaction products, such as $Cr_2As_4O_{12}$ and As_2O_3 , were identified by selected area electron diffraction [11]. Therefore, we consider that the pyrolysis of CCA-treated wood promotes various kinds of complicated reactions and can form chromium trioxide, arsenic trioxide, and stable arsenic metal complexes in the residue. However, it is difficult to determine the detailed mechanism of the immobilization of toxic metals, since the role of the carbon structure of carbonized wood may play a complex role in the process.

4. Conclusion

The effect of pyrolysis on the extractability of CCA elements was studied. The results showed that the resistance of CCA elements to extraction changed drastically after pyrolysis. The toxic metals in CCA-treated wood were highly stabilized by heating at 300 °C for 60 min. Immobilization of toxic elements in the residue was promoted by pyrolysis, resulting in the transformation of toxic metals to various types of stable compounds. Based on these results, the landfilling of CCA-treated wood or of its pyrolysis residue is a poor disposal method, a conclusion also reached by Helsen and Van den Bulck [16]. Landfilling only postpones the collection of the toxic metals, and will lead to many future problems when the toxic elements finally diffuse. Further, although our results confirmed that the CCA elements in the pyrolysis residue were highly stable against leaching or extracting, the arsenic trioxide formed in the pyrolysis residue is more toxic than arsenic pentoxide. Thus, the landfilling of pyrolysis residues is also a poor solution because of their high toxicity. The further development of a suitable method of extracting solvents from waste wood may be the best solution to the problem. To the end, additional investigations into the mechanism of immobilization will be needed. We are currently preparing the next study in our series on the development of an effective solvent extraction method for waste wood.

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