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Influence of Time Variation of CCA and ACQ in Water on Fish Using Charcoal Adsorbability

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The effect of charcoal adsorbability on the contents of heavy metal ions, including Cr, As, and Cu, in water, as well as the fish liver, gill and scales after the time variation of wood preservatives CCA and ACQ in water were evaluated. The time–dependent pH value of charcoals in water with different preservatives was 7.97–8.59. Bamboo charcoal had better adsorption for the Cu content, but for the Cr and As contents it had poor adsorbability. Bamboo activated carbon had preferable adsorption for both Cr and Cu contents, yet it had insignificant adsorbability for the As content. The Cr ion content in the fish scales was with a decreased tendency and the Cu ion content in the fish liver was decreased due to the charcoal adsorbability. However, the Cr ion content in the fish liver and gills was no significant difference before and after a period of time. For the As ion content in fish liver, gills, and scales, there were no obvious change. According to the water quality result, the Cu ion content was decreased slightly, but the As ion content was increased slightly and higher than Cr ion content. In the scope of this study, the charcoal adsorbability can prevent water environments from acidifying, and the Cr and Cu contents can be adsorbed, as well as the adsorbability of bamboo activated carbon was better than that of bamboo charcoal.

Key words: Charcoal Adsorbability, Chromated Copper Arsenate (CCA), Ammonical Copper Quats (ACQ), Time Variation, Fish

INTRODUCTION

In recent years, as common people pay increasing attention to leisure life, the government actively builds recreational facilities following popular will. The outdoor facilities by streams, lakes, coasts, and in local scenic spots often use wood as the main structures, such as timber piles, wooden bridges, wooden trestles, wooden tables, chairs, walls, and bowers. Wood is easily attacked when it is exposed to the environment of biological deterioration, such as soft rot fungi, cellulose degradation fungi (brown rot fungi) and lignin degrading fungi (white rot fungi), crustaceans, and mollusks (Connell, 1991; Ibach, 1999), because the weather is with higher temperature and humidity in Taiwan. The wood strength is likely to be damaged, eventually damaging the structure, and the service life of wooden buildings and recreational facilities is shortened (Weis *et al*., 1995; Caquet *et al*., 1996; Brown and Eaton, 2001).

Many wood preservatives have been developed and applied for the prevention and control of biological deterioration over years. Chromated Copper Arsenate (CCA) wood preservative is one of the most famous water–borne preservatives in the world, and its main constituents include copper (Cu), chromium (Cr), and arsenic (As) salts (Ibach, 1999). CCA can be stably preserved in wood by chemical fixation, but it is likely to be affected by the pH of the wood, the structure and the content of lignin, and the extracts from wood (Lin, 1995). The fixing mechanism of CCA is long lasting, yet it still runs off (leaches from) the wood after a long time (Lin *et al*., 2016), and the leached metal ions have long–term chronic and acute toxic effects on animals and plants (Weis J. S. and P. Weis, 1992a, 1992b; Weis *et al*., 1995; Weis P. and J. S. Weis, 1999). CCA has been prohibited in Germany, Japan, and Indonesian since 2000, and prohibited in the USA and other European countries from 2004. The use of CCA has been forbidden in Taiwan since January 1st, 2016; however, some parts of outdoor facilities are still made of CCA–treated wood at present. In addition, some preservatives, free of Cr and As or with low pollution preservatives, are developed in such cases, in order to replace CCA. Ammonical Copper Quats (ACQ) is used most universally, as its compounds only keeps the Cu compound, and is free of Cr and As, which are carcinogenic heavy metal elements, thus, it is safer than CCA in utilization and operation at present.

Charcoals have particular pore structure, surface functional group, chemical stability, mechanical strength, acid resistance, alkali resistance and heat resistance. They can provide strong adsorption characteristics, and have been used extensively in water purification (Uchimura *et al*., 2000; Peng and Lin, 2015; Lin *et al*., 2015; 2016a). Moreover, the charcoals, after being refined by activation, become activated carbon that still retains the charcoal's characteristics. The specific surface area and total pore volume of activated carbon are obviously higher than those of charcoal (Chang *et al*., 2000; Lin *et al*., 2014). Previous work (Lin *et al*., 2016b)

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have established some of referable results for the adsorbability of bamboo charcoal and Bey–Charng charcoal on the contents of Cr, As and Cu ions, in water, as well as the fish. The adsorbability of bamboo charcoal for the Cu content is better than that of Bey–Charng charcoal. Furthermore, the heavy metal contents, organic carbon, and total nitrogen content in water–based environment with charcoals were lower than those without charcoals.

Therefore, the purpose of this study was to use the adsorbability of bamboo charcoal and activated carbon (refined from bamboo charcoal) to investigate the effect of time–dependent water–borne wood preservatives CCA and ACQ in water–based environments regarding the heavy metal contents of Cr, As, and Cu in water, charcoal, fish liver, gills, and scales. It is expected to have more diversified use and utility value for charcoal adsorbability, and hopefully can be provide a refer results for Taiwan's wood preservation and bamboo charcoal industries.

MATERIALS AND METHODS

Test materials

Water–borne wood preservatives

Wood preservatives included CCA (Chromated Copper Arsenate) Type 3 and ACQ (Ammonical Copper Quats) Type 1. Both preservatives were provided by the KunnYih Co. Ltd, Yilan, Taiwan.

Fish

Taiwan porgy (*Oreochromis hybrids*, Tilapia spp.), also known as Cichlidae, originated from Africa and their body length were about 5 to 10 cm before the tests, was provided by Yihua Fry Unisexual Cultivation Farm, Taiwan.

Charcoal

The bamboo charcoal was made from Moso bamboo (*Phyllostachys heterocycla* Milf) carbonized at 700°C, purchased from Yuen Hung Industry Co., Ltd., Taichung, Taiwan. The bamboo activated carbon was prepared from bamboo charcoal, as the precursors, at 850°C with CO_2–activation (Lin *et al*., 2014).

Source of water specimen

The tap water from National Chiayi University, Taiwan, was maintained for one week before time–dependent testing of the water–based environment.

Test methods

Time–dependent testing for charcoal, fish and preservatives in water–based environment

Taiwan porgy, wood preservatives (1 g), charcoal (bamboo charcoal and bamboo activated carbon) 2000 g (Huang, 2010), and water specimen were put in a rectangular fish bowl (60×45×45 cm^3; about 67.5 L for testing water). The test periods were 1, 2, 3, and 4 weeks, respectively. The charcoals, Taiwan porgy (fish liver, gills, and scales), and water specimen were taken out after each test period to investigate the heavy metal contents of Cr, As and Cu.

Measurement of the characteristics of charcoals

The iodine value of charcoals was measured according to JIS K 1474 (1991), and the detail experimental steps were refer to (Lin *et al*., 2016b). The true density was measured by a true density determinator (UL TRAPYCNOMETER 1000), and the experimental details of the density refer to (Hwang *et al*., 2013). Charcoal characteristic measurements, covering BET specific surface area (Gregg and Sing, 1982), average pore diameter (Yun *et al*., 2001), were carried out.

Measurement of preservatives pH and compound contents

The preservatives pH value and contents (kg/m^3) of Cr (counted as CrO_3), As (counted as As_2O_5), and Cu (counted as CuO) compounds in CCA and the content of Cu (counted as CuO) compound in ACQ were measured according to CNS 14495 (2000) with Atomic Absorption Spectrometry (Varian Model Spectr. AA 220).

Cr, As, and Cu elements analysis of charcoals

The Cr, As, and Cu element contents (%) in charcoals after each period were tested by fluorescence X–ray Harmful Element Spectrometer (XGT–1000WR), and according to WEEE/RoHS/ELV green environmental standards.

Cr, As, and Cu ions analysis of water

A 500mL water specimen was taken, and the pH, Cr, As, and Cu ions in water were measured. The pH was tested by a Cyber Scan pH510 pH meter; the Cr, As, and Cu ions were measured by Flame Atomic Absorption Spectrometer (FAAS). The equation of the concentration of heavy metals ion (ppm) is $A=A'\times F\times(V/W)$; A: concentration of heavy metals in the specimen (mg/kg); A': concentration of heavy metals (mg/L) in the specimen solution obtained by the calibration curve; V: final constant volume (L) after specimen pretreatment (L); W: weight of the original specimen (kg, wet weight, or dry weight); F: dilution factor.

Cr, As, and Cu ion concentrations testing of fish liver, gill and scales

The Taiwan porgy were dissected, and about 0.50 g (fine weighing to 0.01 g) of liver, gills, and scales was put in a microwave digestion bottle, respectively, mixed with 5 mL aqua fortis and 3 mL 35% H_2O_2, the digestion bottle was locked and put in the microwave digestor for microwave digestion, system set output power was 50%, heating time was 45 min, the digestion bottle was then cooled for future use, and the Cr, As, and Cu ion concentrations (ppm) in the digest are measured by Atomic Absorption Spectrometer (Varian Model Spectr. AA 220).

Statistical analysis

The test results are represented by a mean (standard deviation), and the time–dependent specimens are compared by Duncan's multiple range tests. If the ρ value is smaller than 0.05, it is indicated that a significant difference is among the time–dependent specimens.

RESULTS AND DISCUSSION

Information of charcoals and wood preservatives

Table 1 shows the basic characteristics of bamboo charcoal and bamboo activated carbon (activated carbon). The two kinds of charcoals were alkalescent, and the pH was 7.8 and 8.0, respectively. The iodine value of bamboo charcoal was about 118 mg/g, and that of activated carbon was 799.2 mg/g. The true density of bamboo charcoal was 1.96 g/cm^3, and that of activated carbon was 2.06 g/cm^3. The BET specific surface area of bamboo charcoal was about 342 m^2/g, and that of activated carbon was about 907 m^2/g. The average pore diameter of bamboo charcoal was 2.38 m, and that of activated carbon was 2.29 nm, which are of mesopore (IUPAC, 1972). The basic characteristics of charcoals were the same tendency as previous reports (Lin *et al.*, 2014; Lin *et al.*, 2016b).

The basic properties of wood preservatives are shown in Table 2. The CrO_3, As_2O_5, and CuO of CCA (wt %) were 20.67, 46.29, and 33.04%, respectively; the CuO of ACQ was 53.75%, conforming to CrO_3 45–51%, As_2O_5 33–38%, and CuO17–21% of CNS14495 CCA Type 3 and CuO 53–59% of CNS14495 ACQ Type 1. The pH of CCA was acid 2.8; and the preservative ACQ was close to alkaline 9.5.

pH value of water–based environment with wood preservatives

Figure 1 shows the time–dependent pH of an aquatic ecological environment with different preservatives and charcoals. The pH value of the original water was 7.95–8.22. After 4 weeks' testing, the pH of CCA without charcoal (CCABlank) was 8.22–8.38, that of ACQ (ACQBlank) was 8.13–8.30; the pH of that with charcoal was mostly 7.97–8.59, that of CCA with bamboo charcoal (CCABC) increased from 8.13 in Week 1 to 8.59; that of ACQ (ACQBC) was 8.10–8.43. The pH of water with charcoal was higher than that without charcoal, and the pH of CCA with activated carbon (CCAAC) and ACQ with activated carbon (ACQAC) was mostly 7.97–8.30. It is indicated that water with charcoal becomes an alkalescent ecological environment after a period of time, and it has a certain effect on improving water acidification. Shiah *et al.* (2003) reports that due to the charcoal containing abundant natural mineral substances, such as Ca, Mg, Fe, K, Mn, P metal ions etc., which are dissolved in water; therefore, the water becomes alkalescent water that is similar to natural mineral substance about 7.0 to 8.5. Besides, Shi (2003) and Lee *et al.* (2004) indicate that the pH value of water is very important for the environment, because the toxic metal elements in nearby soil and shore–side may dissolve, thus, killing aquatic animals, such as fish, if the pH of water is too low.

Cr, As and Cu contents of charcoals, water and fish

Cr ion content in CCA

Figure 2 (left column) analyzes the Cr ion concentration and Cr element contents in CCA in the time–dependent water–based environment with charcoals. The upper left figure shows the Cr ion (ppm) in water measured by FAAS, the Cr ion concentration content in water of the blank group (BlankW) and control group with CCA only (ControlWCCA) were 0.001–0.005 and 0.013–0.015 ppm, respectively, the Cr ion measured in water with bamboo charcoal (ControlWBC) and activated carbon (ControlWAC) was higher than BlankW and

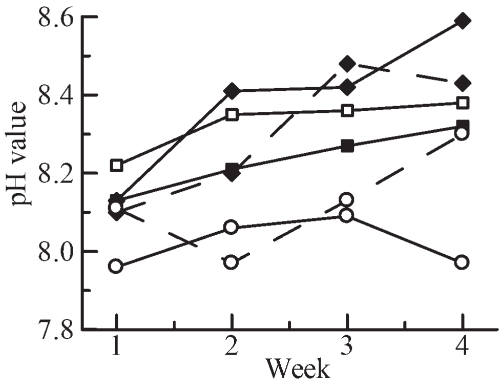

Fig. 1. pH value change of charcoals in water-based environment with different wood preservatives for the 4 weeks period

Legend: —□— : CCABlank, —■— : ACQBlank
—◆— : CCABC, —○— : CCAAC
– ◆ –: ACQBC, – ○ –: ACQAC

Notes: CCA: Chromated Copper Arsenate; ACQ: Ammoniacal Copper Quats; BC: Bamboo charcoal; AC: Activated carbon

Table 1. Basic characteristics of charcoals

Specimens	pH value	Iodine value (mg/g)	True density (g/cm^3)	BET surface area (m^2/g)	Average pore Diameter (nm)
Bamboo charcoal	7.8 (0.2)	118.3 (2.30)	1.96 (0.24)	342.87	2.38 (0.21)
Activated carbon	8.0 (0.5)	799.2 (3.15)	2.06 (0.15)	907.32	2.29 (0.15)

Table 2. Basic properties of wood preservatives

Wood preservatives	pH value	Content of preservatives (wt %)		
		CrO_3	As_2O_5	CuO
CCA[1)]	2.8 (0.20)	20.67 (1.02)	46.29 (1.88)	33.04 (2.01)
ACQ	9.5 (0.15)	–	–	53.75 (2.03)

[1)] CCA: Chromated Copper Arsenate Type 3; ACQ: Ammonical Copper Quats Type 1

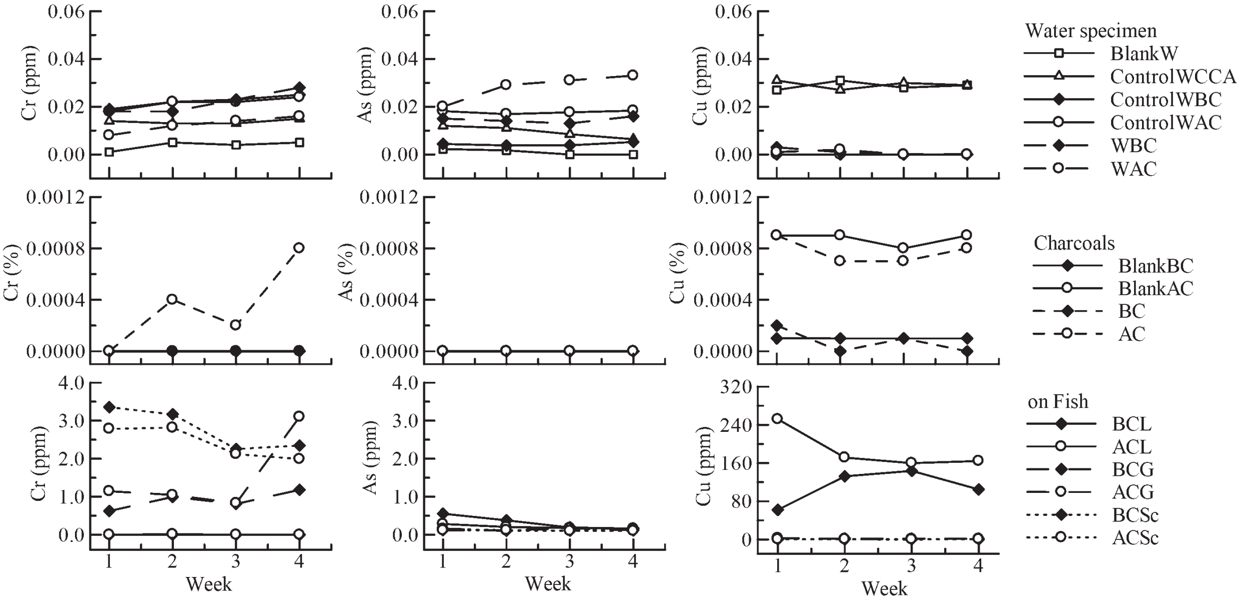

Fig. 2. Cr, As and Cu contents analysis of water, charcoals and fish in water-based environment with CCA for 4 weeks period
Notes: CCA: Chromated Copper Arsenate; ACQ: Ammonical Copper Quats; W: Water; BC: Bamboo charcoal; AC: Activated carbon; L: Liver; G: Gills; Sc: Scales

ControlWCCA, which was 0.018–0.025 ppm. The average Cr ion content in water with bamboo charcoal (WBC) was 0.018 ppm in the first two weeks, which increased to 0.028 ppm gradually in Week 3. The Cr ion of activated carbon (WAC) increased from 0.008 to 0.016 ppm with time. However, the difference of time–dependent in the Cr ion concentration content was insignificantly ($\rho<0.05$) in accordance with Duncan's multiple range tests.

The middle left figure of Figure 2 shows the Cr element content (%) in charcoals, as measured by an X–ray harmful element analyzer. The Cr content adsorbed by bamboo charcoal (BC) increased with the test period, and there was little adsorption effect till Week 4. The Cr content adsorbed by activated carbon (AC) increased from 0.0000 ppm at the beginning of the test to 0.0008 ppm at the end of the test, indicating activated carbon adsorbed Cr ion slowly. From the results of Table 1, the activated carbon was with high specific surface area (about 907 m^2/g) and its average pore diameter was 2.29 nm (a mesopores definition from the IUPAC, 1972). This indicates that the activated carbon can be used as an adsorbent for separating and purifying gaseous or aqueous solution systems as a catalyst supporter, or for recovering precious metals (Manocha, 2003 ; Yorgun *et al.*, 2009; Sun and Jiang, 2010; Peng and Lin, 2015).

The lower left figure of Figure 2 shows the Cr ion contents (ppm) in the fish liver, gills, and scales, as measured by AA. The Cr ion content in fish liver with charcoal (BCL) was almost zero, while the Cr ion in fish gill with bamboo charcoal (BCG) increased with time, from 0.623 ppm to 1.178 ppm. The Cr ion content in fish gill with activated carbon (ACG) increased from 1.145 ppm at the initial stage of testing to 3.099 ppm. Weis *et al.* (1995) indicates that the larvae of organisms in aquatic environments absorbs metal ions in an aquatic environment, and changes the metal ions in vivo into appearance when growing the hard appearance (e.g. fish scales, oyster shell and so on). Therefore, the Cr ion contents in scales with bamboo charcoal (BCSc) and in scales with activated carbon (ACSc) in Week 1 were 3.355 and 2.785 ppm, respectively, and the contents were apparently lower than 2.344 and 1.990 ppm in Week 4. It is inferred that the Cr ion content in fish scales is reduced when the charcoals are used in the water–based environment, and/or it might be correlated with the growth of the fish body.

As ion content in CCA

Figure 2 (middle column) analyzes the As ion in CCA in the time–dependent ecological water environment with charcoals. The upper middle figure showed the As ion content measured in the water, where the WAC had the highest As ion content among various groups of water, which was 0.020–0.033 ppm, and then 0.016–0.018 ppm of ControlWAC, and 0.013–0.016 ppm of WBC. The ControlWBC and BlankW had As ion content lower than 0.005 ppm among all water specimens, and the As ion content in various water specimens increased slightly after a period of time, as well as the As ion content in various water specimens were higher than the safety standard of 0.01 ppm of the Environmental Protection Administration (2003). It is indicated that the As ion content was increased slightly and higher than Cr ion content (the upper left figure of Figure 2).

The middle column with middle figure of Figure 2 shows that various charcoals had no adsorbability for As element content. This is because the charcoal has the best adsorption effect on Pb ion among heavy metal ions, and then, on Cu, Cr, and Ni, with the worst effect on Cd and As (Tsai, 2006). The lower middle figure of Figure 2 shows the measured As ion contents in fish liver, gills, and scales. The As ion contents in BCL and ACL were 0.161–0.553 ppm and 0.161–0.281 ppm, respectively, while that in BCG, ACG, BCSc, and ACSc were lower than 0.193 ppm. Therefore, the As ion contents in fish liver, gills, and scales decreased slightly with time. It might be said that the slight decrease of As ion content is concerned with the growth of the fish body, but not for charcoal adsorbability. In other words, the leaching of As ion from the CCA–treated wood nearby streams, lakes, and coasts, is need to be paid special attention to.

Cu ion content in CCA

Figure 2 (right column) analyzes the Cu ion in CCA in the time–dependent ecological water environment with charcoals. The upper right figure shows the Cu ion content in water. The Cu ion content of BlankW and ControlWCCA was 0.027–0.031 ppm, whereas, ControlWBC and ControlWAC did not contain any Cu ion in water, and for WBC and WAC the Cu ion content was 0.000–0.003 ppm. After 4 weeks' testing, the measured Cu ion contents of ControlWBC, ControlWAC, WBC, and WAC were lower than BlankW and ControlWCCA. The middle right figure of Figure 2 shows the Cu element content adsorbed by charcoal. The decrease of Cu content adsorbed by BC was from 0.0002 at the initial stage of testing to 0.000% at the end. AC had good adsorption effect in Week 1, which was about 0.0009%, and was able to reach 0.001%. While it decreased slightly in Week 2 and Week 3, the Cu content adsorbed by AC was still higher than 0.0007%, and the overall adsorbability of AC for Cu content was higher than that of BC by about 25%.

According to the lower right figure of Figure 2, in terms of the Cu ion content in fish liver, gills, and scales in the test period, the average Cu ion content of BCL and ACL was 61.93–251.75 ppm, which might be because the heavy metals were likely to accumulate in the liver. The Cu ion content of BCL increased from 61.93 ppm at the beginning to 143.63 ppm in Week 3, the Cu ion content of ACL reached its peak 251.75 ppm in Week 1, and then, decreased to about 160.00 ppm with time. The Cu ion contents of BCG, ACB, BCSc, and ACSc were lower than 2.20 ppm, which was apparently lower than the Cu ion of BCL and ACL. This is because the Cu ion in CCA can be adsorbed by the charcoals (the middle right figure of Figure 2), and the adsorbability of activated carbon was better than that of bamboo charcoal.

Cu ion content in ACQ

The upper figure of Figure 3 shows the Cu ion content in water. The control group with ACQ only (ControlWACQ) had the highest Cu ion content among various test groups of water, which was 0.523–0.622 ppm. The Cu ion content of the other groups of water was lower than 0.02 ppm. The ACQ only keeps the Cu compound, but the test conformed that the Cu ion content in various water specimens were lower than the safety standard of 1.0 ppm of the Environmental Protection Administration (2003). The upper right figure of Figure 2 was significantly different from the Cu ion content in the water of Figure 3. It might be because the CCA is a mixed compound and has the fixation of Cr ion, whereas, ACQ is a single compound, thus, the Cu ion is likely to leaching easily.

For the Cu ion adsorbed by charcoal in the middle figure of Figure 3, the Cu element content adsorbed by BC in Week 1 was about 0.0004%, which increased to 0.0005% in Week 2, and decreased slightly in Week 3 and Week 4, but there was no significant difference according to Duncan's multiple range tests. The Cu content adsorbed by AC was 0.0023% at the beginning of testing, which decreased to 0.0008% rapidly with time. However, the Cu content adsorbed by AC was the highest among various test groups. According to the Cu ion contents in fish liver, gills, and scales in the lower figure of Figure 3, ACQ was a Cu compound–based preservative, thus, the Cu ion remains in the fish liver, while the Cu ion contents of BCL and ACL increased from 142.132 and 93.571 ppm to 486.92 and 196.547 ppm, respectively. The contents of BCG, ACG, BCSc and ACSc were very low. It was considered that the charcoal had considerable adsorption effect on Cu ion in ACQ, the Cu ion content in water was relatively low, and the Cu ion content in fish liver decreased with time. Therefore, in the water–based environment of this study, the contents of BCG, ACG, BCSc and ACSc with AC adsorbability were all better than those with BC one.

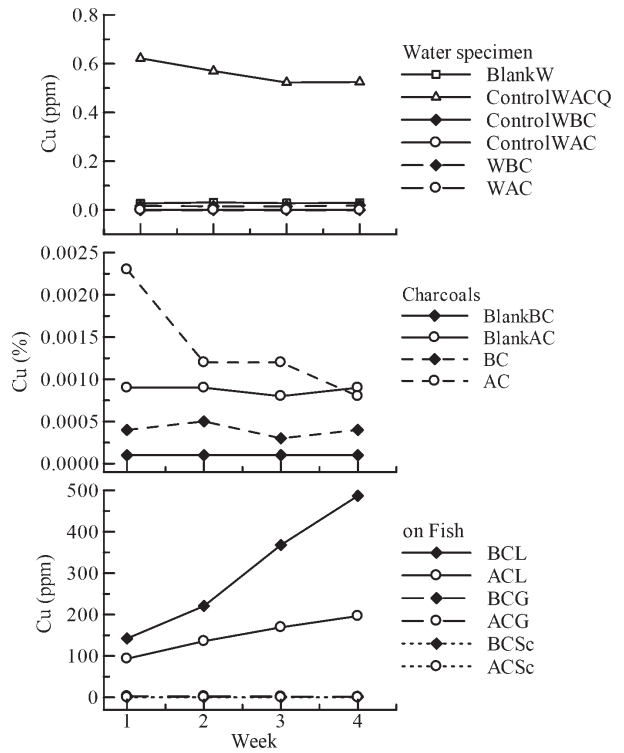

Fig. 3. Cu content analysis of water, charcoals and fish in water-based environment with ACQ for 4 weeks period

Notes: CCA: Chromated Copper Arsenate; ACQ: Ammonical Copper Quats; W: Water; BC: Bamboo charcoal; AC: Activated carbon; L: Liver; G: Gills; Sc: Scales

CONCLUSION

This study used wood preservatives CCA and ACQ as the heavy metal sources in water, and evaluated the adsorption effect of bamboo charcoal and activated carbon, refined from bamboo charcoal, on aquatic ecological environments according to the variation of heavy metal (Cr, As, Cu) contents in the water, charcoal and fish liver, gills and scales during time–dependent testing. The results showed that bamboo charcoal and activated carbon in water–based environments had significant effect on pH, and prevented water from acidifying. Moreover, the charcoals had better Cu ion adsorption than other heavy metals, and the activated carbon performed better than bamboo charcoal. According to analysis of the fish body, the Cu ion content was likely to accumulate in the liver, and the Cr ion in scales was higher at the initial stage of testing; however, due to charcoal adsorbability and the transformation of metal ions in the fish body into appearance with time, Cr ion gradually decreased.

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