Heavy metal levels in water and fish samples from coastal waters of Mahakam Delta, Kutai Kartanegara District, East Kalimantan, Indonesia

[1]Iwan Suyatna, [1]Sulistyawati, [1]Adnan Adnan, [1]Muhammad Syahrir, [1]Ghitarina Ghitarina, [1]Abdunnur Abdunnur, [2]Syahrul Saleh

[1] Faculty of Fisheries and Marine Science, Mulawarman University, Samarinda, East Kalimantan, Indonesia; [2] Agency of Marine Affairs and Fisheries of Kutai Kartanegara District, Tenggarong, East Kalimantan, Indonesia. Corresponding author: I. Suyatna, isuyatna@ymail.com

Abstract. Heavy metal levels in water and fish from Mahakam Delta Waters were measured during May to June 2014. The aims of the study were to describe the biophysical condition and to measure the metal levels both in water and fish. Three locations which were considered to have deposit of coal spill that occurred during transportation were determined as study locations. Water samples were collected from the bottom of the waters using Nansen water sampler. Meanwhile, fish samples were obtained using bottom minitrawler with the help of professional local fishermen. Results indicated that metals in water were almost below detection limit except for Fe (0.02 to 0.72 mg L^{-1}), Mn (0.01 mg L^{-1}), Cu (0.004 mg L^{-1}) and Zn (0.02 mg L^{-1}). Unlikely, metal levels in the six demersal fish species including grouper (*Epinephelus* sp.), snapper (*Lutjanus* sp.), croaker (*Otolithes* sp.), threadfin bream (*Nemipterus* sp.), common hairfin anchovy (*Setipinna* sp.) and mullet (*Mugil* sp.) showed much greater than those in waters. Three of ten estimated metals of Hg, As and Se in fish were below detection limit, while others were detected above the permissible limit of the national and international standards. Those metals were Cu, Zn, Mn, Fe, Ni, Pb and Cd with their levels varied from 1.26 to 44.71 mg kg^{-1}, 28.7 to 97.54 mg kg^{-1}, 6.40 to 69.51 mg kg^{-1}, 46.5 to 408.93 mg kg^{-1}, 0.17 to 1.62 mg kg^{-1}, 0.02 to 4.02 mg kg^{-1} and 0.01 to 1.03 mg kg^{-1} respectively. Generally, metal levels in studied fish exceeded the recommended permissible limit and the fish were considered to be unsafe for human consumption.

Key Words: coal spill, pontoon, Muara Badak, Pantuan, Muara Jawa.

Introduction. East Kalimantan, located in Borneo Island, is a province of Indonesia which has tens of years of history in commercial coal mining. Coal from two districts (Kutai Kartanegara and West Kutai) is continuosly transported by pontoons through Mahakam Delta waters before to be brought to other islands or exported to other countries. Consequently, during transportation, a large amount of coal is spilled and most likely it is deposited and distributed as sediments all over the waters. A hundred and ten samples of coal were analyzed by Zhang et al (2004) and resulted in 20 potentially hazardous trace elements (PHTEs) detected, namely: As, B, Ba, Cd, Cl, Co, Cr, Cu, F, Hg, Mn, Mo, Ni, Pb, Sb, Se, Th, U, V and Zn, whilst The Clean Air Act Amendments identified eleven trace elements and their compounds commonly found in coal as potential hazardous air pollutants (PHAPs): Be, Cr, Mn, Co, Ni, As, Se, Cd, Sb, Hg and Pb (Davidson & Clarke 1996). Populations of commercially edible important fishes commonly inhabit coastal environments which contain high levels of heavy metals which are discharged from industrial wastes or human activities (Duruibe et al 2007; Stancheva et al 2013). Mahakam Delta homes hundreds of commercial edible aquatic biota such as fish, mussels, shrimps and crabs which are an important component of the people diet in the region. During demersal fish samplings and shrimp trawlings operated by local fishermen at depth of two to 40 m, coals were often observed in the fish net (Suyatna 2010). The metals accumulated in fish not only have a bad influence on the biota but also affect the health of human beings (Desta et al 2012). Moreover, the transfer factors of all metals from water to the fish were greater then those from sediment (Abdel-Baki et al 2011). Thus, this preliminary study was carried out to determine the crucial metals levels in both water and fish from Mahakam Delta waters.

Material and Method. Geographically, Mahakam Delta is located on the East of Borneo between S 0°21' and 1°10' and E 117°15' and 117°40' (Sandjatmiko et al 2006).

Water and fish samples. Water and fish sampling were performed in May to June 2014. Bottom water samples were collected using Nansen bottle sampler from three locations, namely Muara Badak, Anggana and Muara Jawa (Figure 1) in which each location had three sampling sites and each site had three sampling points (or equal to 27 sampling points).

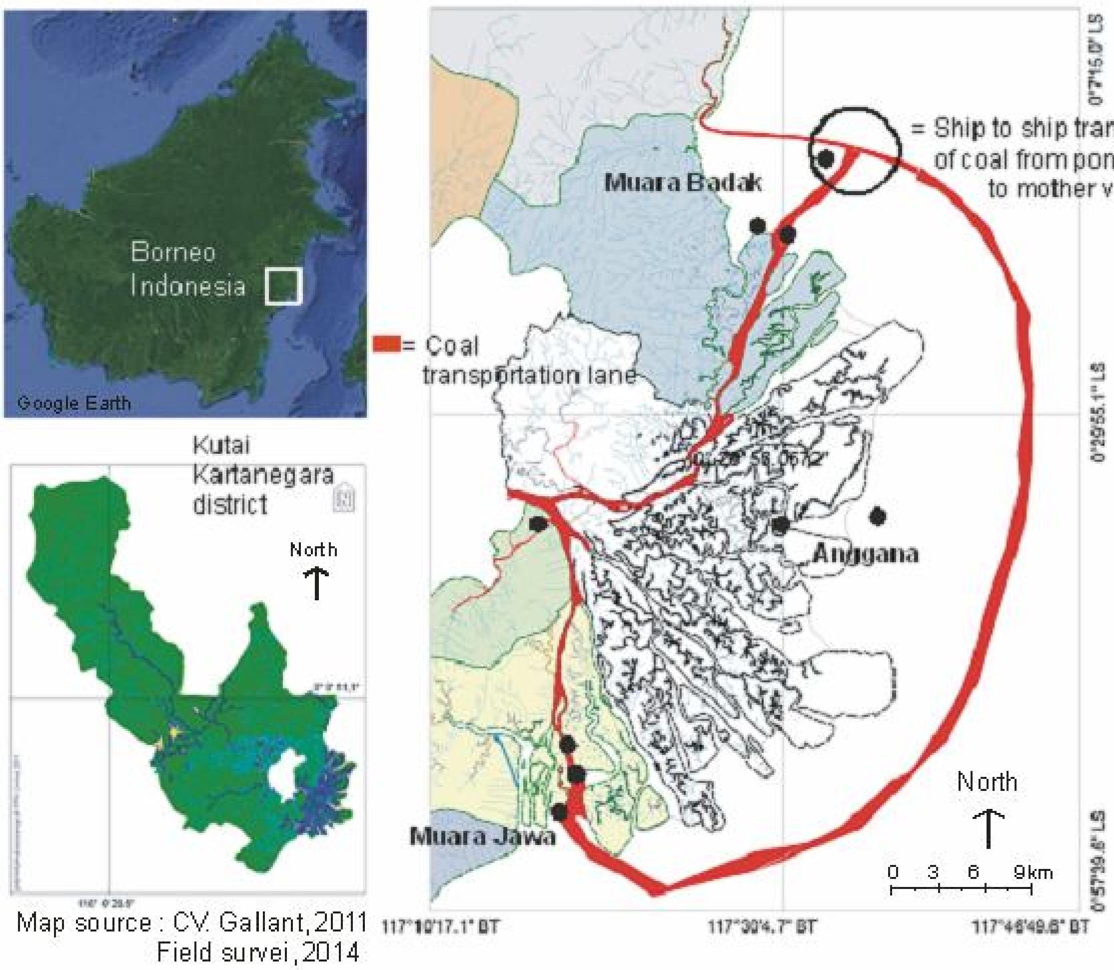

Figure 1. Map showing pontoons lane, area of ship to ship transfer of coal, locations and sampling points distribution of heavy metals study.

Samples of water were put into glass bottles of 2000 mL for physico-chemical parameters analysis; each water sample consisted of one preserved with HNO_3 and another without preservation. The samples then were stored in cool box for further treatments. Including heavy metals, the biophysical parameters of water analyzed were water temperature, water conductivity, water transparency, total suspended solid (TSS), pH, dissolved oxygen (DO), biological oxygen demand (BOD_5), chemical oxygen demand (COD_5), NO_2, NO_3, NH_3, H_2S and plankton (species and abundance). Some of the parameters were measured in situ using water checker U-10 Horiba made in Japan. The fishes were sampled around the locations, using a bottom minitrawler with the help of professional local fishermen. Six demersal species have been determined from each location, namely grouper (*Epinephelus* sp.), snapper (*Lutjanus* sp.), croaker (*Otolithes* sp.), thread beam (*Nemipterus* sp.), longfin anchovy (*Thryssa* sp.) and mullet (*Mugil* sp.). After catching fish were put into a cool box for maintaining their fresh condition. For histological analysis, some of live fish samples were sacrificed and dissected to gain tissue taken from organ of gill, liver and flesh while for metals determination of Cu, Zn, Mn, Fe, AS, Ni, Se, Hg, Pb and Cd, mix of all three of organs (gill, liver and flesh) from six demersal fish were dried by oven and then destructed with addition of chemicals of HNO_3 and HCl which results in a liquid. This liquid was then analyzed using AAS (Atomic Absorption Spectrophotometer).

Laboratory used. Laboratory of Water Quality, Faculty of Fisheries and Marine Science analyzed water quality parameters and prepared materials to be used for metals determination while laboratory of Toxicology prepared a five micron thickness of tissue of each fish organ sample and put it under a microscope of Olympus C-23 made in Japan to fabricate some photographs of histological condition. Determination of metals was carried out at East Kalimantan Province Laboratory of Health with Atomic Absorption Spectrophotometer on the basis of the standard of the SNI/Standar Nasional Indonesia, Indonesian National Standard (SNI 2009a, b) and American Public Health Association (APHA 2005).

Data analysis. Data were analyzed descriptively. Physico-chemical parameters of water and metal levels in water and in fish were referred to national and international water quality guidelines and literature data reported for water and fish. Qualitative histological alteration of tissue of studied fish was observed with referring to Handari (1983), Robert (1989), and Takashima & Hibiya (1995). Diversity index (Shannon and Dominance) of plankton were realized using software of the Palaeontological Statistics (PAST).

Results and Discussion. The results of the biophysical parameters of water analysis and diversity index of plankton samples from three locations are presented in Tables 1 and 2.

Generally, both physical and chemical properties of water were within the tolerable range, except TSS at certain sampling points of location Muara Badak (Table 1) which was above the permission limit. However, that is possible to accept because the turbid water of the big river of Mahakam highly affected the sea. The conductivity was detected very high as an effect of salinity like Ilelaboye & Olasoji (2015) reported that sea water has a high salt content affecting the conductivity of waters.

Shannon index (*H'*) value was moderate, and this was normal since the water samples analyzed were gathered from the bottom of the waters as Rahaman et al (2013) reported that species diversity index value of plankton depended on spatial variation including column of waters. All study locations seemed to be poor in species number and no any plankton species dominated.

Table 1

The results of the physico-chemical parameters analysis from all locations

Parameters	*Measure unit*	*Site*			*Permissible limit ($mg\ kg^{-1}$)*
Physical		*1*	*2*	*3*	
			Muara Badak		
Temperature	^0C	31.2	31.2	32	nat.
Conductivity	μS cm^{-1}	37600	37400	47000	15000-50000*
Transparency	cm	110	110	30	nat.[1]
TSS	mg L^{-1}	62	18	37	20[2]
			Anggana		
		1	*2*	*3*	
Temperature	^0C	29.1	28	28.6	nat.
Conductivity	μS cm^{-1}	228	52	5020	15000-50000*
Transparency	cm	18	16	25	nat.[1]
TSS	mg L^{-1}	28	15	17	20[2]
			Muara Jawa		
		1	*2*	*3*	
Temperature	^0C	28.9	28.8	28.8	nat.
Conductivity	μS cm^{-1}	8570	8430	9900	15000-50000*
Transparency	cm	80	85	45	nat.[1]
TSS	mg L^{-1}	5	6	7	20[2]
			Muara Badak		
Chemical		*1*	*2*	*3*	
pH		7.12	7.18	7.23	7-8.5 [2]
DO	mg L^{-1}	5.72	5.72	4.61	>5.0 [2]
BOD_5	mg L^{-1}	1.42	1.48	1.22	20.0 [2]
COD_5	mg L^{-1}	13.4	20.4	12.3	50[1]
NO_2	mg L^{-1}	0.09	0.08	0.01	0.06[1]
NO_3	mg L^{-1}	7.51	11.44	8.49	10.0[1]
NH_3	mg L^{-1}	0.12	0.08	0.08	0.5[1]
H_2S	mg L^{-1}	-	-	-	1.0 [2]
Salinity	‰	23.9	23.9	28.9	nat.[1]
			Anggana		
		1	*2*	*3*	
pH		7.32	7.21	7.11	7-8.5 [2]
DO	mg L^{-1}	4.09	4.23	4.22	>5.0 [2]
BOD_5	mg L^{-1}	1.24	1.32	1.22	20.0 [2]
COD_5	mg L^{-1}	5.9	8.2	7.1	50[1]
NO_2	mg L^{-1}	0.01	0.02	0.02	0.06[1]
NO_3	mg L^{-1}	8.39	8.49	8.35	10.0[1]
NH_3	mg L^{-1}	0.3	0.18	0.3	0.5[1]
H_2S	mg L^{-1}	-	-	-	1.0 [2]
Salinity	‰	0.5	0.5	0.5	nat.[1]
			Muara Jawa		
		1	*2*	*3*	
pH		7.02	7.05	7.06	7-8.5 [2]
DO	mg L^{-1}	4.08	4.12	4.09	>5.0 [2]
BOD_5	mg L^{-1}	1.21	1.24	1.12	20.0 [2]
COD_5	mg L^{-1}	12.4	12.2	14.1	50[1]
NO_2	mg L^{-1}	0.02	0.05	0.01	0.06[1]
NO_3	mg L^{-1}	5.71	7.54	5.73	10.0[1]
NH_3	mg L^{-1}	0.07	0.08	0.12	0.5[1]
H_2S	mg L^{-1}	-	-	-	1.0 [2]
Salinity	‰	0.5	0.5	0.5	nat.[1]

Note: - = below detection limit; [1]PP no. 82 (2001); [2]KLH no. 51 (2004); nat. = natural (according to water ecosystem); *Edwin et al (2016).

Table 2

The diversity index of plankton from locations

	Muara Badak			*Anggana*			*Muara Jawa*		
	Sampling point								
	1	*2*	*3*	*1*	*2*	*3*	*1*	*2*	*3*
Taxa_S	8	9	7	10	8	6	8	8	9
ind/l	1575	1386	2834	1197	756	693	945	1323	1386
H'	1.82	1.97	1.63	2.19	2.02	1.67	1.88	1.91	2.05
D	0.2	0.17	0.24	0.12	0.14	0.21	0.18	0.17	0.14

Metals in water. Among ten metals analyzed, four metals were within detectable level occurred in two locations and others were below detection limit. Cu and Fe were detected in Muara Badak, while Zn, Mn and Fe were observed in Muara Jawa, only Fe could be said beyond the limit (Table 3).

Table 3

Heavy metal levels detected in water at all locations and the permission limit

Metals	*Muara Badak*			*Anggana*			*Muara Jawa*			*Permissible limit (mg L^{-1})*
	Sampling point									
	1	*2*	*3*	*1*	*2*	*3*	*1*	*2*	*3*	
Cu	0.004	-	-	-	-	-	-	-	-	0.008[2]
Zn	-	-	-	-	-	-	0.02			0.010[2]
Mn	-	-	-	-	-	-	0.01	0.01	0.01	0.050[4]
Fe	0.02	0.42	0.19	-	-	-	0.08	0.73	0.49	0.300[1]
As	-	-	-	-	-	-	-	-	-	0.012[2]
Ni	-	-	-	-	-	-	-	-	-	0.050[2]
Se	-	-	-	-	-	-	-	-	-	0.050[3]
Hg	-	-	-	-	-	-	-	-	-	0.001[2]
Pb	-	-	-	-	-	-	-	-	-	0.008[2]
Cd	-	-	-	-	-	-	-	-	-	0.001[2]

Note: - = below detection limit; [1]PP no. 82 (2001); [2]KLH no. 51 (2004); [3]Governor decree no. 339 (1988); [4]WHO (2003).

Levels of Fe and Zn in study locations were higher than the national water quality guidelines. Saeed & Shaker (2008), Ozturk et al (2009), and Bazzi (2014) reported much greater of their findings of Fe levels as the followings: 0.28 to 2.39 mg L^{-1}, 0.008 to 1.98 mg L^{-1} and 7.06 to 8.67 mg L^{-1} respectively. Fe levels in water samples in the present study were much lower. Meanwhile, Abdel-Baki et al (2011) in their results of determination of metal levels study reported that water was the transfer factor of metal bioaccumulation in fish. Since the majority of water samples content metal levels in below detection limit, therefore waters from all study locations were considered safe from the heavy metal pollution.

Metals in fish. Different with the metal levels obtained in water samples, the metal levels in fish samples showed much greater than those in water samples. Seven metal levels (Cu, Zn, Mn, Fe, Ni, Pb and Cd) in fish samples were determined and exceeding the tolerable values standards as shown in the Tables 4, 5 and 6 for Muara Badak, Anggana and Muara Jawa, respectively.

Table 4

Metal levels detected in fish at location Muara Badak and the permission limit

Metals	*Sample from each sampling point*									*Permissible limit (mg kg^-1^)*
	Site 1			*Site 2*			*Site 3*			
	1	*2*	*3*	*1*	*2*	*3*	*1*	*2*	*3*	
Cu	1.23	1.05	1.33	1.81	1.76	1.66	1.50	1.46	1.23	< 20.0[7]
Zn	36.20	36.20	36.20	44.10	45.01	43.50	78.90	70.06	76.1	40.0[5]
Mn	4.00	4.02	4.02	19.30	18.90	19.00	4.70	4.65	3.80	1.00[9]
Fe	51.50	49.05	50.46	73.00	69.20	70.09	68.60	67.80	68.00	100.0[9]
Ni	-	-	-	0.66	0.57	0.66	-	-	-	0.12[8]
Pb	0.53	0.60	0.56	0.53	0.60	0.52	0.18	0.10	0.20	0.30[6]
Cd	-	-	-	-	-	-	0.01	0.01	0.01	1.00[10]

Note: - = below detection limit; [5]Hutagalung & Suwirna (1987); [6]Indonesia National Standard SNI 7387 (2009a, b); [7]BPOM No. 03725/B/SK/VII/89 (1998); [8]Lihan et al (2006); [9]WHO (1989); [10]Indonesian National Standard SNI 7388 (2009b).

Table 5

Metal levels detected in fish at location Anggana and the permission limit

Metals	*Sample from each sampling point*									*Permissible limit (mg kg^-1^)*
	Site 1			*Site 2*			*Site 3*			
	1	*2*	*3*	*1*	*2*	*3*	*1*	*2*	*3*	
Cu	1.35	1.26	1.36	1.93	1.89	1.93	1.33	1.33	1.33	< 20.0[7]
Zn	97.54	89.90	96.65	60.68	60.66	60.78	81.57	80.98	81.34	40.0[5]
Mn	6.48	6.46	6.40	62.51	60.07	64.05	69.51	67.89	68.06	1.00[9]
Fe	82.71	89.70	83.60	135.8	150.8	128.40	238.6	200.5	223.8	100.0[9]
Ni	0.70	0.70	0.68	1.56	1.50	1.55	1.60	1.58	1.62	0.12[8]
Pb	-	-	-	-	-	-	-	-	-	0.30[6]
Cd	-	-	-	0.05	0.01	0.04	1.03	0.98	1.01	1.00[10]

Note: - = below detection limit; [5]Hutagalung & Suwirna (1987); [6]Indonesia National Standard SNI 7387 (2009a); [7]BPOM No. 03725/B/SK/VII/89 (1998); [8]Lihan et al (2006); [9]WHO (1989); [10]Indonesian National Standard SNI 7388 (2009b).

Table 6

Heavy metal levels detected in fish at location Muara Jawa and the permission limit

Metals	*Sample from each sampling point*									*Permissible limit (mg kg^-1^)*
	Site 1			*Site 2*			*Site 3*			
	1	*2*	*3*	*1*	*2*	*3*	*1*	*2*	*3*	
Cu	2.51	35.78	23.50	44.71	20.35	12.80	1.58	1.45	1.56	< 20.0[7]
Zn	96.20	62.70	70.80	57.80	57.60	50.80	34.08	30.45	28.70	40.0[5]
Mn	11.12	26.94	23.65	16.89	15.76	16.09	15.20	15.02	15.23	1.00[9]
Fe	135.2	264.5	143.3	408.9	324.5	350.2	53.86	46.50	50.50	100.0[9]
Ni	0.33	0.90	-	1.31	-	-	0.17	-	-	0.12[8]
Pb	-	4.02	3.05	2.97	2.65	2.03	1.23	1.00	0.02	0.30[6]
Cd	0.03	0.11	0.09	0.20	0.20	0.17	-	-	-	1.00[10]

Note: - = below detection limit; [5]Hutagalung & Suwirna (1987); [6]Indonesia National Standard (SNI 7387:2009a): Maximum limit of polluted heavy metal in food; [7]BPOM No. 03725/B/SK/VII/89 (1998); [8]Lihan et al (2006); [9]WHO (1989); [10]Indonesian National Standard (SNI 7388, 2009b): Maximum limit of polluted heavy metal in food.

In general, people who regularly eat fish from certain polluted waters have higher levels of metals than those who do not. Five common toxic metals should be avoided is Hg, Pb, Al, As and Cd. The effect of these toxic metals may cause various problems from subtle symptoms to serious diseases. Muscles always possessed the lowest concentrations of all metals, liver was the target organ for Cu, Zn and Fe accumulation, Pb and Mn however, exhibited their highest concentrations in the gills, different species of fish showing inter-specific variation of metals, as well as variations between fish from the same species (El-Moselhi et al 2014).

Eventhough Hg, As and Se in the present study were not found from all study locations, however the majority of other metals in fish was above the level of permissible limit especially Zn, Mn and Fe, Fe was ranging from 46.5 mg kg^{-1} to 408.93 mg kg^{-1} much higher than the other two metals. After reviewing 17 results of assessment from various countries such as Turkey, Egypt and India related to the above metals, Fe level in fish were found to reach up to 5660.84 mg kg^{-1} accumulated in the tissue, muscle, liver and gills of *Sander lucioperca* (Basyigit & Tekin-Ozan 2013), 4569.9 mg kg^{-1} accumulated in the muscle, gills and liver of Nile tilapia, *Oreochromis niloticus* (Saeed & Shaker 2008) and 3473.95 mg kg^{-1} accumulated in the gills, liver, kidney, muscle and integument of *Channa punctatus* (Javed & Usmani 2014) respectively. Similar to our finding, Ayas et al (2007) and Edward et al (2013) recorded the order of metal level in fish from their study results of metal determination that Cd was the lowest. However, even in very low level, this metal is considered the most toxic element to human life that may cause a bone disease similar to rickets, cardiac enlargement, anemia, gonadal atropy, kidney failure, and pulmonary emphysema (Khallaf 1999 cited by Bahnasawy et al 2011). The present study has also observed that the levels of Zn and Mn in fish samples were much higher than the national standards. The levels of the metal ranged from 28.7 mg kg^{-1} to 97.54 mg kg^{-1} and 3.8 mg kg^{-1} to 69.51 mg kg^{-1} respectively. Kumar et al (2012) assessed the content of Zn and Mn in valuable coastal fishes and found the range of the metals from 3.00 mg kg^{-1} to 99.1 mg kg^{-1} and 0.5 mg kg^{-1} to 12.0 mg kg^{-1} respectively. Our finding of Pb and Ni levels were reaching to levels that are harmful to humans with their levels ranged from 0.02 mg kg^{-1} to 4.02 mg kg^{-1} and 0.17 mg kg^{-1} to 1.62 mg kg^{-1}, respectively. It was reported that Fe is often discharged into aquatic environments, ferrous iron (Fe^{2+}) being more toxic to fish than the ferric (Fe^{3+}) form and the highest bioconcentration of iron in fish tissues was found in the liver and gonads, decreasing in brain, muscle and heart (Authman et al 2015), and highest in gills (Akan et al 2012), while Zn is the second most abundant trace element after Fe found almost in every cell; Mn and Cu were found in skin, gills, gonads and in liver, respectively (Rajkowska & Protasowicki 2013), Cu accumulated in brain and eyes causing mense disease which is about a fatal disorder (Azaman et al 2015), Pb in fish from Cirata dam Bandung Indonesia exceeds the standard quality according to FDA RI (Junianto et al 2017) and generally the origin was mainly anthropogenic (Mollazadeh et al 2013). This metal and Cd were observed in muscle, gill, and liver of three fish species (Jia et al 2017), the values of heavy metals such as Ni (0.12 mg L^{-1}) was beyond the limit detected in tissues such as gills, liver, kidney, muscle and integument of fish (Javed & Usmani 2013); Cd was also detected in muscle tissue of snapper and mullet (Damodharan & Reddy 2013).

From Mahakam Delta waters, almost all metals assessed in fish samples exceeded the safety permissible level of human use except Hg, As and Se, they were not detected in fish from all locations. The metal levels obtained in fish samples were in the order of Fe > Zn > Mn > Cu > Pb > Ni > Cd. Most of the metals are present in edible portion of fish (Afshan et al 2014).

However, other seven metals exceeded the recommended permissible limit and so, fish from the studied locations are potentially harm to consumers.

Microscopic photographs of fish organ tissue of gill, liver and flesh from result of laboratory works showing histological changes are presented in the Figures 2-7.

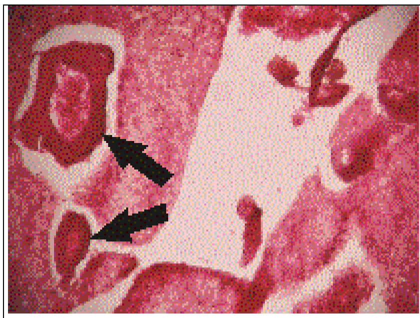

Figure 2. Liver tissue of croacker *Otolithes* sp. thickened (indicated by black arrow).

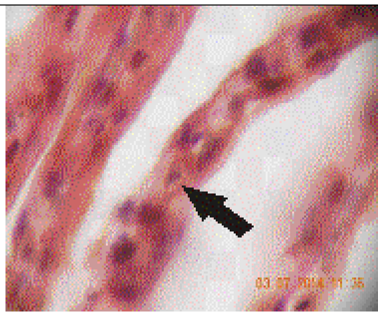

Figure 3. Gill pilaster cell of longfin anchovy *Thryssa* sp.

Figure 4. Liver of thread beam *Nemipterus* sp. experienced inflammation (indicated by black arrow).

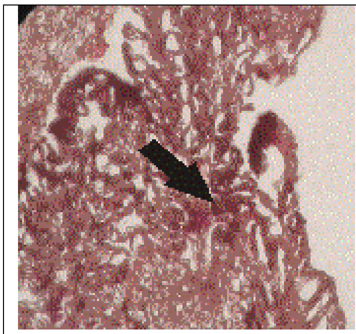

Figure 5. Gill of grouper *Epinephelus* sp. attacked by parasite (indicated by black arrow).

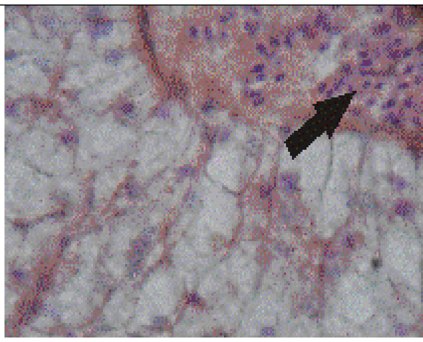

Figure 6. Color of erythrocite in blood vessel of mullet *Mugil* sp. (indicated by black arrow).

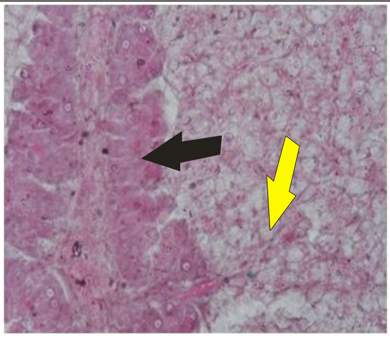

Figure 7. Endocrine pancreas (indicated by black arrow) and blood vessel (yellow arrow) of snapper *Lutjanus* sp. became pale.

Conclusions. On the basis of the results of ten metals analysis, in water four metals were detected and Fe was above the permissible limit, and in fish seven metals showed to accumulate in fish organ of gill, liver and flesh of six edible demersal fish samples from estuarine waters of Mahakam delta. Histologically, bioaccumulation of the metals in fish organ caused abnormalities on tissue and this metal accumulation might be one among factors causing fish easily attacked by parasites especially in wild population.

Acknowledgements. Authors acknowledge financial support from Agency of Marine Affairs and Fisheries of Kutai Kartanegara District. We also thank the Dean of Faculty of Fisheries and Marine Science for allowing us to use laboratory facilities. Finally, we appreciate East Kalimantan Province Laboratory of Health for the assistance of metals analysis of fish samples. Thank goes also to Rusdiansyah, Ahmad Mutakin, Tedy Hanjoko and Moh Raafi for their help during field and laboratory works.

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Received: 16 August 2017. Accepted: 08 October 2017. Published online: 31 October 2017.
Authors:
Iwan Suyatna, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: isuyatna@ymail.com
Sulistyawati Sulistyawati, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: busulis.bdp@gmail.com
Adnan Adnan, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: adnan_unmul@yahoo.co.id
Muhammad Syahrir, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: syahrir.ramang@gmail.com
Ghitarina Ghitarina, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: ghitarina@gmail.com
Abdunnur Abdunnur, Aquatic Resources Management, Faculty of Fisheries and Marine Science, Mulawarman University, Indonesia, East Kalimantan, Samarinda, Jalan Gunung Tabur Kampus Gunung kelua, 75123, e-mail: abdunnur67@yahoo.co.id
Syahrul Saleh, Agency of Marine Affairs and Fisheries of Kutai Kartanegara District, Jalan Ahmad Yani No. 50, Kelurahan Melayu, Tenggarong, East Kalimantan, Indonesia, e-mail: pesisirpulau@yahoo.com

How to cite this article:
Suyatna I., Sulistyawati, Adnan A., Syahrir M., Ghitarina G., Abdunnur A., Saleh S., 2017 Heavy metal levels in water and fish samples from coastal waters of Mahakam Delta, Kutai Kartanegara District, East Kalimantan, Indonesia. AACL Bioflux 10(5):1319-1329.