Heavy Metals Concentration in Muscle Tissue of Threatened Sharks (*Rhizoprionodon acutus*, *Sphyrna lewini,* and *Squallus hemipinnis*) from Binuangeun, Lebak Banten, Indonesia

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Abstract: *Metals accumulation in threatened sharks' meat represents a global health issue. The objective of the current study was to measure the concentration of six metals (Li, Ti, Ni, Cd, As, and Pb) using ICP-MS in threatened sharks' meat of* Rhizoprionodon acutus, Squalus hemipinnis, *and* Sphyrna lewini *from Binuangeun Fish Auction in Lebak, Banten, Indonesia. The results showed that the concentration of Ni, Li, Cd, and Pb was below the acceptable levels for food sources for human consumption, except for As concentration (more than 30 fold higher). This study showed high levels of Ti concentration in all threatened sharks' meat. Overall, this study shows that an accumulation of Ti in sharks' meat should be considered a risk to the health of seafood consumers.*

Keywords: *heavy metals;* Rhizoprionodon acutus*;* Sphyrna lewini*;* Squalus hemipinnis*; Binuangeun*

INTRODUCTION

Elasmobranchii can be found in many habitats and at various depths. They show a variety of foraging strategies and tactics, including planktonic filter-feeding and predatory species. *Elasmobranchii* (sharks and rays) seek the meat of cartilaginous fish used to make shark fin soup dishes throughout Southeast Asia in many regions, including Indonesia [1]. Sharks are commonly consumed in shark fin soup, filets (fresh meat), and liver oil in many countries such as Australia [2], Brazil [3], China [1], Japan [4], and South Korea [5]. Metal accumulation is an essential parameter in determining the food safety of shark meat due to the many harmful effects of metals [5-6]. Since shark meat has long been an important food source, much research has been done on heavy metal concentrations to ensure food safety [2,4,7-10].

Sharks can accumulate heavy metals such as Pb, Hg, Cd, and As when they ingest prey through the food web [11] and the feeding behavior of each species [12]. Because sharks exhibit characteristics associated with low metabolic rates and high longevity [13], they are more susceptible to the accumulation of these metals [2]. Heavy metals from shark meat can cause harmful effects on humans if consumed frequently in large amounts [14].

Sharks are secondary products in the Binuangeun Fish Auction that mainly focus on pelagic fishes (tuna, skipjack tuna, and mackerel tuna) and are dominantly caught by gillnet and longline fishing [15]. Types of sharks and rays recorded landed at Binuanguen Fish Auction in Banten such as *A. marmoratus*, *C. falciformis*, *C. longimanus*, *C. brevipinna*, *C. melanopterus*, *A. pelagicus*, *Squalus hemipinnis*, *I. oxyrinchus*, *C. sealei*, *R. acutus*, *H. griseus*, *S. lewini*, *R. australiae*, *R. penggali*, *Mobula* sp., *T. lymma*, *R. javanica*, and *N. caeruleopunctata* [15]. Sharks are sold directly at the Binuangeun Fish Auction and are commonly consumed as fishmeal without processing (salted, smoked, or dried). *Rhizoprionodon acutus* and *Squalus hemipinnis* are listed as vulnerable in The IUCN Red List of Threatened Species, while *Sphyrna lewini* is a critically endangered species [16-18]. Previous studies have demonstrated the ability of *Elasmobranchii* to accumulate high levels of metals such as As on *S. lewini* muscle tissues from the Gulf of California [19]; Cd and Pb on *R. acutus* from the Persian Gulf area [20], and Kuala Terengganu Malaysia [21]; Cd and As on *S. lewini* from Trinidad-Tobago [22].

The metal concentration in shark meat consumed in Indonesian areas is limited, especially from the Southern Java Sea. In this study, we evaluated the concentration of heavy metals (As, Cd, Pb, Ni, Li, and Ti) in three threatened shark species (*R. acutus*, *S. lewini*, and *S. hemipinnis*) that landed at Binuangeun Fish Auction, Banten, Indonesia. We also examined correlations between metal concentrations and biological aspects such as total length (TL) and total weight (TW) and metal-metal correlation in each species of sharks. By measuring metal concentrations in sharks and comparing them with national and international standards, risks to human health can be assessed, and it is possible to know the pollution status of *Elasmobranchii* from the Binuangeun area. Current findings may be helpful to future researchers.

■ EXPERIMENTAL SECTION

Materials

Sharks were purchased at Binuangeun Fish Auction in Lebak Banten, Indonesia (latitude: -6.838101°, longitude: 105.883637°) in September 2019. A total of sixteen *R. acutus*, eight *S. lewini,* and six *S. hemipinnis* were measured the morphometric with a rolling meter for the total length (TL) and digital balance for the total weight (TW) and directly frozen (-20 °C) before further analysis. In the laboratory, approximately 50 g of sharks' muscle was filleted from the dorsal area, collected in plastic bags, and kept in a freezer (-20 °C) until further analysis. Nitric acid 65% Merck (Darmstadt, Germany), mixed standard stock solutions (containing 100 mg/L of each element) were provided by Merck (Darmstadt, Germany). All reagents used were of analytical reagent grade, and the solutions were prepared using ultra-pure water (Milli-Q).

Instrumentation

Dried sample preparation using oven (Heraeus Instrument), volumetric flash 25 mL, glass funnel, petri dish, spatula, mortar and pestle, and a microwave digestion system (Multiwave 5000 Anton Paar with 41 rotor vessels). The metal concentration analysis using ICP-MS ICAP-RQ Thermo. All glassware was soaked in 10% of HNO_3 for 24 h and rinsed three-time using deionized water before being used.

Procedure

Sample preparation

Shark muscles were dried in an oven at 60 °C for 24 h for metals analysis and moisture content. Dried samples were homogenized using mortar and pestle. Digestion procedures were developed according to the procedures from Anton Paar [23] with modifications on temperature, the volume of acid, and time for digestion.

Metals analysis

Approximately 0.2 g (triplicate) of homogenized dried sharks' muscle samples were digested using Multiwave 5000 microwave digestion. Each sample was mixed with 5 mL of HNO_3 and heated gradually from room temperature to 100 °C in 10 min and held for 5 min, increased to 175 °C in 8 min, and held for 15 min to complete digestion processes. Samples were cooled to room temperature, deionized water was added to digested samples, and diluted until 25 mL using a volumetric flask. Blank digestion was also carried out the same as the sample digestion process consisting of 5 mL HNO_3. Heavy metals analysis was based on a previous study by Murugesan et al. [24]. Total concentration of 6 metals (arsenic [^{75}As], cadmium [^{111}Cd], lead [^{208}Pb], lithium [^{7}Li], nickel [^{60}Ni] and titanium [^{48}Ti]) were analyzed using ICP-MS. Analysis was carried out in Kinetic Energy Discrimination (KED) mode, the $^{140}Ce.^{16}O/^{140}Ce$ ratio of 0.017%, plasma power of 1550 W, auxiliary flow of 0.8 L/min, and nebulizer flow of 0.985 L/min. The digested solutions (blanks, samples, and Certified Reference Materials (CRMs)) were measured three times for each sample (Relative Standard Deviation (RSD) < 5%). The final concentration of metals was reported as µg/g wet weight. Wet weight concentration is calculated by multiplying the dry weight concentration with the moisture content factor. The accuracy and precision were verified using CRM DORM-4 provided by the National Research Council of Canada (NRCC) (n = 3).

Statistical analysis

Statistical correlation and graphical plot were based on the open-source R program (package R.4.1.2 for Windows) [25]. The normality data was first confirmed by conducting a Kolmogorov-Smirnov test. Pearson's correlation coefficient analysis determines the significant relationship between total length/total weight and metals concentration in each species. Two-tailed p-value < 0.05 and p-value < 0.01 were considered significant.

■ RESULTS AND DISCUSSION

Metals Concentration on Each Species of Sharks

The morphometrics of sharks analyzed in this study is shown in Table 1. The results of heavy metal concentration (µg/g wet weight) analysis in the meat of *R. acutus*, *S. hemipinnis*, and *S. lewini* are shown in Table 2. The *R. acutus* and *S. hemipinnis* samples were mature based on the size described in the IUCN report [17-18], while *S. lewini* size shows in the juvenile stage [16]. The highest concentrations of As were found in the meat of *R. acutus* (9.60 ± 8.01 µg/g) and *S. hemipinnis* (9.57 ± 3.95 µg/g), and the lowest values were found in *S. lewini* (3.93 ± 1.08 µg/g). The highest concentrations of Ti were found in *S. lewini* (0.619 ± 0.398 µg/g). Pb concentrations were up to ten times higher in *R. acutus* (0.060 ± 0.037 µg/g) than in *S. lewini* (0.006 ± 0.008 µg/g). Ni concentrations were observed equally in *R. acutus* and *S. lewini* (0.037 ± 0.044 µg/g and 0.037 ± 0.019 µg/g, respectively). Li concentrations were found around 0.009–0.018 µg/g in all shark meat. Cd concentrations were observed only on *R. acutus* (0.004 ± 0.001 µg/g), while *S. hemipinnis* and *S. lewini* below LOD. Measurement of CRM (DORM-4) shows promising results with a range from 81.28–107.73% of the recovery (Li: 100.41%; Ni: 81.28%; As: 92.52%; Cd: 81.60% and Pb: 107.73%, respectively), indicating the digestion method suitable to apply on shark muscle samples.

In this study, the concentration of As showed a significantly higher concentration in demersal sharks (*S. hemipinnis*) [18] than in pelagic sharks (*R. acutus* and *S. lewini*) [16-17]. The As concentration in *R. acutus* is lower than the previous study reported by Kim et al. [5] from Jeju island, the Republic of Korea, and by Boldrocchi et al. [26] from Djibouti, Indian Ocean areas, but shows a higher concentration of As compared to study reported by Muhammed and Muhammed [22] from Trinidad and Tobago areas. The As concentration in *S. lewini* is threefold lower than the previous study reported by Berges-Tiznado et al. [19] and Boldrocchi et al. [26] from Djibouti, Indian Ocean areas. Differences in these findings in this species may be related to differences in As availability at different sites and biological parameters involved in As metabolism (length, age, weight, reproductive stage) [19]. Feeding behavior and the food sources of the shark species were other factors that metals can uptake in shark metabolism [19] and trophic position on the food web [27]. *S. lewini* has been classified as a general opportunistic predator [16],

Table 1. Total body length, body weight, and sex of sampled sharks

Scientific name	Common name	N	Total weight (g)	Total length (cm)	Sex	IUCN status
Rhizoprionodon acutus	Milk shark	16	195–450	34.5–46.8	M: 11 F: 5	Vulnerable [17]
Squalus hemipinnis	Indonesian shortnose spurdog shark	6	485–1190	49.5–62.3	M: 1 F:5	Vulnerable [18]
Sphyrna lewini	Hammerhead shark	8	825–1335	56.4–68.0	M:4 F:4	Critically endangered [16]

Table 2. The concentration of metals (μg/g wet weight) in sharks' species from Binuangeun, Lebak, Banten, Indonesia. The average concentration is shown in brackets

Species	Li	Ti	Ni	Cd	As	Pb
R. acutus	0.004–0.108 (0.018)	0.048–0.579 (0.295)	0.001–0.139 (0.037)	0.002–0.006 (0.004)	1.57–23.49 (9.60)	0.009–0.140 (0.060)
S. hemipinnis	0.005–0.018 (0.009)	0.287–0.604 (0.384)	< LoD	< LoD	3.73–13.50 (9.57)	0.003–0.063 (0.036)
S. lewini	0.08–0.023 (0.014)	0.310–1.570 (0.619)	0.011–0.062 (0.037)	< LoD	3.00–5.85 (3.93)	0.001–0.021 (0.006)

LoD: limit of detection

directly relating to the abundance of prey in the study area [19,28]. Another factor is the migratory pattern of the shark while in the juvenile and adult stages or during the nurseries stage [29-30].

Pb, Cd, and Ni concentration in *R. acutus* and *S. lewini* from Binuangeun show a lower trend than the previous result on Ong and Gang [21] from Kuala Terengganu, Malaysia, Boldrocchi et al. [27] from Djibouti, Indian Ocean areas, and Adel et al. [20] from the Persian Gulf. This study's Cd, Ni, and Pb concentrations were lower than Hauser-Davis et al. [31] on blue sharks (*Prionace glauca*) from the Western North Atlantic Ocean. The concentration of Cd and Pb in *S. lewini* reported by Mohammed and Mohammed [22] from Trinidad and Tobago shows a higher concentration in all muscles (hypaxial muscle, epaxial muscle, and dorsal fin) compared to this study. These metals have been the most studied in life because their effects can severely alter metabolic processes and even lead to death at low concentrations [32-33]. This study reported new data related to metals on *S. hemipinnis* because there is no publication about metal accumulation in Indonesian sprout nose dogfish from Indonesia. The concentration of Cd and Pb on demersal sharks in this study was lower than the study reported by Bilbao et al. [10] from Macaronesian Archipelago.

Unfortunately, there are no studies of titanium accumulation by sharks in the study area. The concentration of Ti in this study was shown to be lower compared to the study reported by Hauser-Davis et al. [31] on *P. glauca* from the Western North Atlantic Ocean and Ju et al. [34] on megamouth sharks (*Megachasma pelagios*) from Taiwan. However, higher than a study reported by Bouchoucha et al. [35] from the Western Mediterranean Sea on commercial fishes. Ju et al. [34] reported that a high concentration of Ti on *M. pelagios* is probably because of the feeding habits of planktivorous compared to carnivorous sharks. Titanium has been reported to be associated with marine microalgae (diatoms and dinoflagellates) in high concentrations (more than 1,000 μg/g dry weight) [36]. Titanium (Ti) is a contaminant of increasing concern, exhibiting increased bioavailability in aquatic environments in recent decades due to increased use in personal care products (toothpaste, conditioner, shampoo, and conditioner) in the form of TiO_2 nanoparticles [37]. Some previous studies had reported Ti could be biomagnified through food webs and bioaccumulated in several aquatic organisms [38-43]. The International Agency for Research on Cancer has already classified titanium as "possibly carcinogenic to humans" (Group 2B) because it may pose significant risks to human health [44]. Future assessments will be conducted to clarify the accumulation of Li and Ti on marine fishes. The concentration of metals varies according to the habitats, feeding behavior, and species-specific [4-5,10,45].

Metals Correlation on Each Species

We observed specific metal-metal correlation in sharks meat shows different responses due to species-specific metabolism, and no significant correlation was observed between total length or total weight and concentration of metals in the meat of all shark species. Total weight of *R. acutus* was shown significantly positive correlation ($p < 0.001$) with total length (r^2 = 0.91). A significant negative correlation ($p < 0.001$) was observed between Ti-Cd (r^2 = -0.79) and As-Cd (r^2 = -0.69, $p < 0.01$) on *R. acutus*. A significant positive correlation ($p < 0.05$) on *R. acutus* was observed between Li-Ti (r^2 = 0.43), Li-As (r^2 = 0.51), Li-Pb (r^2 = 0.59), Ti-As (r^2 = 0.49), Ti-Pb (r^2 = 0.45) and As-Pb (r^2 = 0.50). A significant negative correlation ($p < 0.05$) on *R. acutus* was observed between Li-Cd (r^2 = -0.47) and Cd-Pb (r^2 = -0.52). *S. hemipinnis* and *S. lewini* were observed a significant positive correlation ($p < 0.001$) on TW-TL (r^2 = 0.94) and TW-TL (r^2 = 0.99), respectively.

Biplot PCA grouping of samples on threatened sharks from the Binuangeun area using the concentration of metals, TW, and TL as factors shows separation from *R. acutus* to *S. hemipinnis* and *S. lewini* (Fig. 1). *R. acutus* negatively correlates with Li, As, and Pb but positively correlates with Cd. *S. hemipinnis* and *S. lewini* show a positive correlation with Ti concentration, indicating that those sharks accumulate higher titanium than *R. acutus*. PCA can show factors correlating to metal accumulation in each species of threatened sharks from the Binuangeun area.

The multivariate analysis (PCA) can identify the differences in metals among shark species. Variable food sources could explain the variability in each species in their habitat [13,19]. The metals pattern of PCA is useful because it normalizes data, minimizes the effects of individual element concentrations, and more clearly divides samples into two or more groups based on species or sex identities. In this study, linear regression analysis did not reveal a significant relationship between shark length/weight and metal concentration in each species. However, length/metals or weight/metals relationships have been reported in the previous study [5,46]. We observed a significant relationship between habitat (coastal/pelagic sharks vs. deep-water/benthic sharks) and metal concentration. Titanium concentration showed a significant positive relationship with total length and weight (Pearson correlation, (TW) r = 0.40, p = 0.028, n = 28; (TL) r = 0.43, p = 0.016, n = 28) if analysis based on habitat (coastal/pelagic vs demersal/deep-water), while lead showed a significant negative relationship with TW and TL (TW r = -0.58, p = 0.0016, n = 25; TL r = -0.61, p = 0.0008, n = 25). In habitat, many factors influence the uptake of metals in sharks, such as a variety of food diets [2,19], the abundance of food sources [19], anthropogenic activities [7,26], and different trophic positions while juvenile stage vs. adult stage [27,29].

Heavy metals concentration such as Cd and Pb except As were lower than the permissible limit ([As] = 0.25 µg/g, [Cd] = 0.10 µg/g and [Pb] = 0.20 µg/g) from National Agency of Drug and Food Control (NADFC) or BPOM [47] or JECFA/WHO [48]. The concentration is of As in *S. lewini* four times (3.93 µg/g) higher than the NADFC permissible guideline, while in *R. acutus* (9.60 µg/g) and *S. hemipinnis* (9.57 µg/g), more than 38 fold than the rules. The TL of *S. lewini* did not exceed 70 cm, and it is evident that samples consist mainly of juveniles [16], but *R. acutus* and *S. hemipinnis* showed

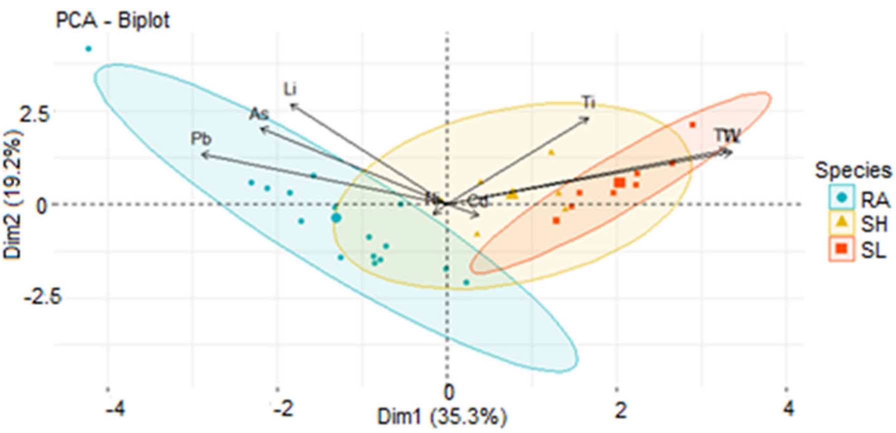

Fig 1. Biplot PCA metals concentration in threatened sharks from Binuangeun, Lebak, Banten, Indonesia. RA = *R. acutus*; SH = *S. hemipinnis*; SL = *S. lewini*

mainly adult samples [17-18,49]. In the shark fishery, small sizes are preferred for meat due to the low concentration of urea (the way it decomposes affects the smell and taste of meat), low commercial price, and other potentially toxic elements [19]. As in shark meat (commonly marine fish), the concentration of arsenic is primarily in the form of an organic compound (85 to 90%) and will secrete from the human body by urinary excretion [50]. Even though inorganic As is the dominant species in freshwaters and marine, it will rapidly bio transform to organic As such as methyl arsenate or dimethyl arsenate by an aquatic organism. The organic arsenic is bioaccumulated in biota and biomagnified through the food web, but the concentration of inorganic As does not increase through trophic levels [19,26-27]. The consumption of shark's meat in Binuangeun areas must be considered the As concentration level because arsenic correlates to Li and Ti, especially on *R. acutus* (Fig. 1). However, whether the same biomagnification pattern of As is different species or different adult-juvenile populations is still unknown. This is evidence that more studies are required to elucidate whether arsenic is increasing in the aquatic food web, including all the shark species from Binuangeun areas.

■ CONCLUSION

This study showed a significant concentration of metals in threatened sharks' meat from Binuangeun Fish Auction, Lebak, Banten, Indonesia. The concentration of total As in milk shark and dogfish shark meat was found to be higher compared to a scalloped hammerhead shark. The presence of a high concentration of Ti in *S. lewini* and *S. hemipinnis* through this study requires investigation regarding the form of Ti that accumulated in sharks' meat.

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■ AUTHOR CONTRIBUTIONS

Conceptualization, S.S. and N.A.; sample analysis, S.S.; statistical analysis, S.S. and D.S.; writing-original draft preparation, S.S.; writing-review and editing, S.S., N.A. and S.W. All authors have read and agreed to the published version of the manuscript.

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