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Water Quality Assessment Using Activated Carbon from Cocoshells in Lake Mainit, Philippines

Kristine Georgia Y. Po[1], Christine Joy L. Ocon[1], Chris Rolan P. Dayuno[1], Jenny C. Cano[1*], Angelus Vincent P. Guilalas[1], Arjune A. Lumayno[2], Nathaniel D. Tiu[1], Jessa S. Cabaña[3]

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ABSTRACT

Mining activities pose environmental impacts especially when the operation is near bodies of water, thus, affecting water quality. As these environmental impacts are growing, there is a pressing need for increased intervention studies to improve water quality. This study aimed to evaluate the effectiveness of granulated activated carbon made from coconut shells in reducing heavy metal levels and enhancing the water quality of Lake Mainit located at Agusan del Norte and Surigao del Norte, Philippines. Silica sand, pumice stones, and white marble chips were added to a glass tank with the granulated activated carbon made from coconut shells. The water sample underwent various laboratory tests. The atomic absorption spectrometry flame technique was used to analyze the heavy metals lead and cadmium. Gravimetric method was employed in total suspended solids and total dissolved solids, and nephelometric method for turbidity. Pre-treated water sample analysis regarding lead, total dissolved solids, and turbidity are within the permissible limits, however, total suspended solids and cadmium concentration surpassed the allowable limits for Class A waters. Removal efficiency in terms of heavy metal concentration and the significant difference of parameters between the water sample before and after intervention were calculated. Results showed that after the intervention, activated carbon made from coconut shells were able to reduce the cadmium level present in the water sample. It also improved the quality of water within permissible limits. Hence, the activated carbon made from agricultural waste such as coconut shells has considerable potential to provide better water quality.

INTRODUCTION

Water is life as it is necessary for all known forms of life. The importance of water in the global economy cannot be taken for granted. Agriculture consumes around 70% of the freshwater utilized by humans (Khokhar, 2017). Moreover, water is a great solvent for a wide range of mineral and organic chemicals, and it is frequently utilized in industrial processes, as well as cooking and cleaning. One of the industries that needs so much water is the mining industry. Each ton of coal produced in a coal mine necessitates around 250 gallons of freshwater. Freshwater accounts for around a quarter of overall water consumption in coal mining, with the balance being recycled water (Moon, 2017). The mining sector significantly impacts water resources, draining and contaminating water supplies through outflows or leakages from tailings or waste rock impoundments. To conserve water, typically mining industries treat the water used in order to reuse it for other needs, employing a number of possible treatment methods.

In the Philippines, Lake Mainit as the country's fourth biggest lake located at the province of Surigao del Norte and Agusan del Norte has a surface size of 173.40 square kilometers. The lake is also the country's deepest, with a maximum depth of 223 meters (Lotha, 2017). There are 28 river tributaries comprising the lake and just one exit river that goes into Butuan Bay. Accordingly, this freshwater lake is classified as Class A in the country (DENR, 1990). This rich and diversified wetland habitat supports a vibrant freshwater fishery and the livelihood of over 3,000 fishermen who use a wide range of fishing gear (Biña-de Guzman *et al.*, 2013). In recent years, environmental and human factors have endangered the lake's biodiversity and productivity. While large-scale mining was no longer taking place near the lake, ongoing small-scale gold mining in municipalities of Alegria, Kitcharao, and Jabonga posed a similar risk of siltation and heavy metal pollution of the lake. From a study, heavy metal levels, such as cadmium, lead, and mercury, were measured and results showed that there's really presence of these heavy metals in the said lake which has been a growing concern of its residents (Ebol *et al.*, 2020).

For ideal water filtration systems, activated carbon is an important component. Activated carbon (ACb) is a type of carbon that has been treated to provide a vast surface area. Tiny, low-volume pores provide a surface for adsorption or chemical reactions. The enormous surface area of ACb contributes to its efficacy. One gram of ACb has a surface area of about 3,000 square meters, and one teaspoon has almost the same area as a football field (CBTECH, 2018). Moreover, ACb is highly helpful for a broad range of applications, including water treatment, due to its huge surface area. Meanwhile, physical adsorption is the primary method by which ACb removes a material. Intermolecular forces pull molecules into the millions of holes and pockets on the surface of ACb when liquid or air comes into contact with it (Emis, 2010).

[1] College of Engineering, Saint Paul University Surigao, 8400 Surigao City, Philippines
[2] College of Education, Culture & Arts, Saint Paul University Surigao, 8400 Surigao City, Philippines
[3] Chemist, Taganito Mining Corporation, Claver, 8410 Surigao del Norte, Philippines
[*] Corresponding author's email: jenny.cano@spus.edu.ph

Furthermore, ACb is made from high-carbon-content environmental sources such as bamboo, coconut husk, willow peat, wood, coir, lignite, coal, and petroleum pitch (Ahmad & Azam, 2019). The Philippines is a country abundant of agricultural resources such as coconuts. In 2018, the country is the second-largest producer of coconut goods in the world (Tacio, 2019). The coconut shell is a promising source of ACb when it comes to treating water. A study shows that when compared to commercial activated carbon (coal, lignite and peat-based carbons), coconut shell activated carbon had a greater lead adsorption capability. Coconut shell carbon is often cleaner than coal-based carbon and can be used in drinking water applications as a safe, high-quality carbon (Hatch, 2001). Moreover, it was shown that the coconut shells were effective in reducing the Methyl Tertiary-butyl Ether (MTBE) to less than 1 ppb (parts per billion). It also has enhanced the odor and flavor of the water (Mohd Samdin *et al.*, 2013). And in China, Potassium Hydroxide-prepared coconut shells produced satisfactory results in adsorption removal of lead (Song *et al.*, 2013).

These abovementioned facts motivated the researchers to further compare and analyze the potentials of agricultural resources as water filtration medium for water polluted with heavy metals. This study can be the basis of further exploring the means for filtering water using renewable resources that can be found in abundance.

Objectives

This study aimed to answer the following research questions:

1. What is the quality of water sample before the intervention in terms of Total Dissolved Solids (TDS), Total Suspended Solids (TSS), turbidity and heavy metal (Lead and Cadmium) concentrations? What is the quality of water after the treatment has been implemented? How is the water quality evaluated using the standards set by Department of Environment and Natural Resources?
2. Is there a significant difference between the pre-test and post-test results?

LITERATURE REVIEW

Surface and groundwater resources can be depleted by mining. Groundwater withdrawals might harm or destroy streamside habitats hundreds of kilometers away from the mine. Due to interaction between water and various types of minerals, a mine creates vast volumes of highly concentrated effluent. In addition to rainfall drainage, the source of these effluents may be traced back to several mining activities. Wash waters, flow process acids, water leaching, flotation, concentration, and effluents from refineries and gas scrubbers can all contribute to mining effluents. Rain infiltrating the mine's tailings can trigger oxidation, hydrolysis, washing, and other processes, resulting in highly polluted effluent (Safe Drinking Water Foundation, 2017).

Small-scale and large-scale mining exist in the region of Caraga, Philippines to exploit gold and other mineral resources. However, these mining activities pose a threat to the water bodies of the communities. In the study, locations of three mining activities were identified: (1) near Tagbuyawan, Mainit, there was a small-scale gold processing area, (2) upstream of the Magpayang, Mainit, there is a large-scale gold mine, and (3) upstream near San Pedro, Alegria, there is a small-scale gold mining operation. They have been equally detrimental in terms of siltation and in heavy pollution that might lead to pollution of lake (Ebol *et al.*, 2020). Water treatment should be taken importance in strategic water planning to minimize water pollution and maximizing the resources available.

Coconut shell is an agricultural waste that is widely available in tropical places around the world. Charcoal is commonly made from coconut shells. Charcoal yields 25–30% of the dry weight of shells used in the classic pit method of manufacture. The calorific value of the coconut shell is 20.8MJ/kg, and it can be utilized to make steam, energy-rich gases, bio-oil, charcoal, and other products. It is a 100-percent organic, natural, and biodegradable material that is a by-product of the coconut fiber extraction process (Zafar, 2022). Apart from its other qualities, coconut shells have been demonstrated to be exceptionally successful at removing pollutants, tastes, and smells from drinking water. Coconut shell carbon has a greater volume of micropores for filtering than coal, wood, or lignite carbon. As reported, coconut shell activated carbon is an effective adsorbent to remove Bismuth from aqueous solutions with good adsorption capacity (Sartape *et al.*, 2012). Coconut shell was also proven to be useful when being used to remove another heavy metal, Zinc, from an aqueous solution. It shows satisfactory results with its adsorption capacity (Behnamfard & Salarirad, 2013).

Coconut shells have been extensively studied as a raw material for the production of activated carbon and other adsorbent materials. Due to their availability, low-cost, and renewability, they have gained attention as an eco-friendly alternative to traditional adsorbents. Several studies have investigated the use of coconut shell-based adsorbents for removing heavy metals from contaminated water. For instance, a study by Odisu *et al.* (2022), results showed that the activated carbon made from coconut shells within the temperature range of 450^oC to 500^oC with 0.1M KOH as activating agent was able to remove 50% of the iron concentration. When 0.1M ZnCl was used as activating agent within the temperature range of 400^oC to 450^oC, the activated carbon made from coconut shells was able to reduce the zinc, iron and lead concentration to 0.01mg/L. Nickel concentration was reduced down to 0.01mg/L when 0.2M ZnCl was used as activating agent for temperature range of 400 ^oC to 500 ^oC. Their research has demonstrated that coconut shells and other organic wastes that are transformed into activated carbon may be utilized as cement waste water adsorbents instead of currently available commercial adsorbents. Because of its high surface area and deeper pore spaces, it has also been confirmed that the combination of physical and chemical treatment of the adsorbent may improve the adsorption capacities. The adsorption capabilities within the scope of the experimental inquiry demonstrated sensitivity to temperature, with an inverse relationship for activated carbon manufactured from coconut shells.

Similarly, another study conducted by Emahi *et al.* (2019) investigated the effectiveness of activated carbon made from coconut shells in removing mercury and arsenic from "artificially" contaminated water versus the coconut shells in its raw form. $CaCl_2$ was used to activate the coconut shell-derived activated carbon, which was subsequently rinsed, deionized, and dried for three hours at 110°C in the oven. According to an investigation using atomic absorption spectroscopy, coconut shell-derived activated carbon produced better results. Its removal efficiency is 67% for arsenic and 53% for mercury, compared to 65% and 49% for raw coconut shells. Additionally, physicochemical characteristics including pH, conductivity, color, turbidity, TDS, and TSS were measured. In general, these factors were enhanced as well.

The study of Packialakshmi *et al.* (2021) shows that the activated carbon made from coconut shells can also be used as an adsorbent for treating wastewater discharged from electroplating industries.

The factors such as adsorbent effective particle size, contact duration, and dose influence the treatment efficacy of zinc and potassium, which have been recognized as possible contaminants in waste water. Increased dose and specific surface area with smaller particle size improves treatment effectiveness up to a certain point, after which the fraction adsorbed remains constant. It is clear that a dose of 15 g/l for 90 minutes is sufficient to remove 90% of phosphate and 97% of zinc from 150 μm particle size. For the removal of zinc and potassium, the adsorption equilibrium values were successfully matched using the Freundlich isotherm. The baseline investigation demonstrated activated carbon from coconut shell's capability for reducing zinc and potassium from industrial waste water.

As reported, heavy metals can be removed using activated carbon made from coconut shells. With an initial concentration of 2 ppm, a pH of 5, and adsorbent dosage of 10 g/L, 70 percent of copper is removed (Aparna & Aryasree, 2019). Activated carbon is powdered charcoal that has been purified (Jackson, 2020). It's made from high-carbon-content waste in the environment (Ahmad & Azam, 2019). Carbonaceous material generated from charcoal is used to make activated carbon. Pyrolysis of organic compounds of plant origin produces activated carbon. Coal, coconut shells, and wood, as well as sugarcane bagasse, soybean hulls, and nutshells, are among the materials used (Ahmad & Azam, 2019). It is subjected to physical or chemical treatment to create micro fissures, greatly enhancing its adsorptive surface area. Activated carbon is highly recommended for air and water purification, as well as precious metal recovery and removal. According to a study entitled, Performance of activated carbon in water filters, activated carbon is particularly successful in reducing pollutants; its adsorption capacity achieves the most positive findings for TSS, Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and the pH has increased from 6.12 to 7.13 (Siong *et al.*, 2013).

MATERIALS AND METHODS

The aim of this study was to present an evaluation and analysis of the granular activated carbons from coconut shells in treating polluted water through adsorption method. In order to achieve this, an experimental research design using the one-group pretest-posttest design was utilized by the researchers. This is a method in which measurements are obtained before and after a treatment. It has been used since the 18th century in many fields such as medicine-nursing, health, mental health, as well as education and has been used commonly up to the present since it is a fast and convenient method (Zavvar Mousavi & Seyedi, 2010). In this study, water sample as the dependent variable was tested before and after the treatment with the activated carbon bed filter as independent variable.

This study utilized the following materials: (1) mechanical grinding machine to turn coconut shells into fine particles, (2) mesh for obtaining the size of ground, (3) furnace for carbonization and drying out of excess water, (4) Gravimetric Methods for TSS and TDS, (5) Nephelometric Methods for turbidity, and (6) the Atomic Absorption Spectrophotometry (AAS) Flame Technique for heavy metal analyses.

Materials needed to prepare activated carbon were collected, such as the granular activated carbon made from coconut shells and the water sample from Lake Mainit. The granular activated carbon made from coconut shells were acquired from Cocoprime Activated Carbon Corporation in Aurora, Zamboanga del Sur. Materials needed for the construction of filter bed system were purchased such as glass and polyvinyl chloride (PVC) for the tank. Silica sand, pumice stones and white marble chips were also purchased to serve as sand and gravel in the filter bed system. Testing of the water sample was done before the treatment in order to measure various set of parameters such as TDS, TSS, turbidity, and heavy metal concentrations in regards with Lead and Cadmium.

Preparation of Activated Carbon

The following procedures shows the method of preparing activated carbon by the manufacturer: the coconut shell should be separated and cleaned from coconut fibers and soil. Next, dry the coconut shells under the sun. Put them in a burning sink or drum at 300-500^{0}C for 3-5 hours. To become chemically activated, soak the carbon in chemical solution (calcium chloride or zinc chloride at 25%) for 12-18 hours. Using distilled or clean water, wash the carbon. To drain, spread on a tray at room temperature. Dry for 3 hours at room 110^{0}C temperature in the oven. Crush or refine activated charcoal into a specified mesh using a crusher wood/iron.

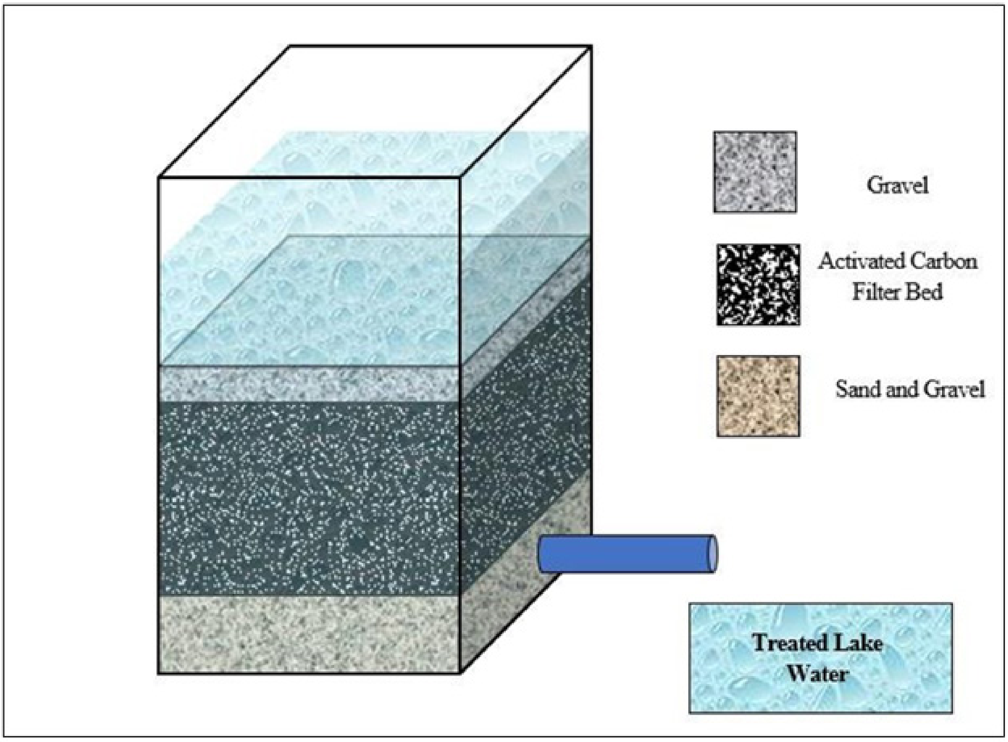

Figure 1: Activated Carbon Bed Filter System

Construction of Activated Carbon Bed Filter

As shown in Figure 1, a tank with the dimension of 30 × 30 × 50 cm (length × width × height) was made out of glass then a faucet of polyvinyl chloride (PVC) is connected to serve as a drainage pipe. Gravel ranging in size from 16 to 32 mm was washed with distilled water to eliminate pollutants and impurities. It was installed above the 2 kg of activated carbon to operate as an energy dissipator. Sand in the 0.6–2 mm size range was also cleaned with distilled water to remove pollutants and contaminants. It was placed beneath the activated carbon layer to keep the particles from falling. A second layer of gravel is placed beneath the sand and in the outlet pipe zone to provide sufficient pore spaces for water to enter the perforated pipes that are connected to the drainage pipe.

During the treatment, ten (10) liters of water sample was poured to the activated carbon bed filter. Contact time starts respectively. Treatment was observed for 3 days. The researchers took samples at the 3rd day of contact time. After the treatment, the researchers utilized Multi-Parameter Water Quality Checker and Atomic Absorption Spectrophotometry (AAS) to evaluate and compare the water quality. TDS, TSS, turbidity and heavy metal concentration in water intake after the treatment were determined.

For data analysis, removal efficiency equation was used for the calculation of the removal percentage (R%) of each heavy metal detected by the following formula:

$$R\% = (C_i - C_e)/C_i \times 100$$

where Ci and Ce were the initial and final concentration of specific heavy metal in the solution respectively (mg L^{-1}). The researchers also utilized the country's administrative orders also known as DENR Administrative Order No. 2016-08 or the Water Quality Guidelines and General Effluent Standards of 2016 (DENR, 2016), DENR Administrative Order No.1990-34 or the Revised Water Usage and Classification Water Quality Criteria (DENR, 1990), and the DENR Administrative Order No. 1994-26A or the Philippine National Standards for Drinking Water (DENR, 1994). They provide criteria for classifying water bodies in the country; determining temporal trends and evaluating phases of water quality deterioration/improvement; and determining the need for adopting actions to avoid, control, or abate water pollution and to ensure clean water. The researchers used these administrative orders as basis for the comparison of the results measured after the intervention for different parameters. Samples were tested at the water laboratory facility of University of San Carlos, Talamban Campus, Cebu City, Philippines. Lastly, after checking the normality assumption of the data, paired t-test and effect size were used with 5% significance level to determine the significant difference between the pre-test and post-test results.

RESULTS AND DISCUSSION

For Cadmium and Lead, AAS Flame Technique was employed. AAS utilized the characteristic of metals to absorb light at a specific wavelength. Standard solution of different concentrations was read in AAS for the equipment to establish a linear relationship between absorption and concentration. The absorption of cadmium and lead present in lake water samples were determined and subsequently determined their concentration from the linear equation obtained from the standards. Gravimetric method was used for TSS and TDS. With this method, the total suspended solids were filtered out from the water sample and dried at 103°C-105°C to eliminate water then weighed. For total dissolved solids, the filtrate was evaporated and further subjected to a temperature of 180°C to isolate dissolved solids then weighed. Nephelometric method for turbidity measured the light from a source that was scattered when it passed through the water sample.

Table 1 presents the measurement of each parameter of water samples before and after the 3 days intervention. With the methods used, Cadmium was found to be undetectable (<0.008) after the intervention while Lead was undetectable (<0.01) before and after the intervention, as shown in Table 1. Furthermore, Table 2 shows that Cadmium and TSS levels were above the permissible limits during the pre-test, hence, failed to meet the minimum standards (DENR, 2016). After the intervention or the use of Activated Carbon made from Coconut Shells, these results were improved by decreasing the values within the permissible limits.

Table 1: Pre-test and Post-test Results of Physicochemical Parameters

Parameter/Unit	**Pre-Test**	**Post-Test**	**Method**
Cadmium (mg/L)			
Trial 1	0.013	Less than 0.008	AAS Flame Technique
Trial 2	0.014	Less than 0.008	
Trial 3	0.022	Less than 0.008	
Lead (mg/L)			
Trial 1	Less than 0.01	Less than 0.01	AAS Flame Technique
Trial 2	Less than 0.01	Less than 0.01	
Trial 3	Less than 0.01	Less than 0.01	
Total Suspended Solids (mg/L)			
Trial 1	55	1.5	Gravimetric
Trial 2	50	2	
Trial 3	70	1.5	
Total Dissolved Solids (mg/L)			
Trial 1	362	138	Gravimetric
Trial 2	378	148	
Trial 3	406	148	
Turbidity (NTU)			
Trial 1	3.2	0.22	Nephelometric
Trial 2	3.24	0.22	
Trial 3	3.2	0.25	

Table 2: Pre-test Results as Compared to DAO No.08 (2016)[1], DAO No.34 (1990)[2] and DAO No. 26A, (1994)[3] Permissible Limits

Parameters	**Maximum Level Allowable**	**Results**	**Remarks**
Cadmium1	0.01 mg/L	0.0163 mg/L	Failed
Lead1	0.01 mg/L	Undetectable	Passed
TDS2	1000 mg/L	382 mg/L	Passed
TSS1	50 mg/L	58.33 mg/L	Failed
Turbidity3	5 NTU	3.213 NTU	Passed

Table 3: Post-test Results as Compared to DAO No.08 (2016)[1], DAO No.34 (1990)[2] and DAO No. 26A, (1994)[3] Permissible Limits

Parameters	**Maximum Level Allowable**	**Results**	**Remarks**
Cadmium1	0.01 mg/L	Undetectable	Passed
Lead1	0.01 mg/L	Undetectable	Passed
TDS2	1000 mg/L	144.67 mg/L	Passed
TSS1	50 mg/L	1.66 mg/L	Passed
Turbidity3	5 NTU	0.23 NTU	Passed

Removal efficiency was calculated only for Cadmium since Lead was already below the detectable level based on the analysis. After conducting three (3) trials, the resulting 0.016333 average of the pre-test was used. Meanwhile, the result for post-test was within the undetectable level of below 0.008. It is safe to assume that the cadmium content is 0.007 for the sake of determining the removal percentage. Thus, the removal percentage was:

$$R\% = \frac{0.016333 - 0.007}{0.016333} x\ 100 = 57.06\%$$

As shown in Figures 2, 3, 4, and 5, the levels of Cadmium, TDS, TSS, and Turbidity were decreased after the intervention. Specifically, after the intervention, the Cadmium and TSS levels were below the 0.01mg/L and 50mg/L permissible limits, respectively; and the TDS and Turbidity levels were further decreased, below the 1000 mg/L and 5 NTU permissible limits, respectively (DENR, 2016; DENR, 1990; DENR, 1994).

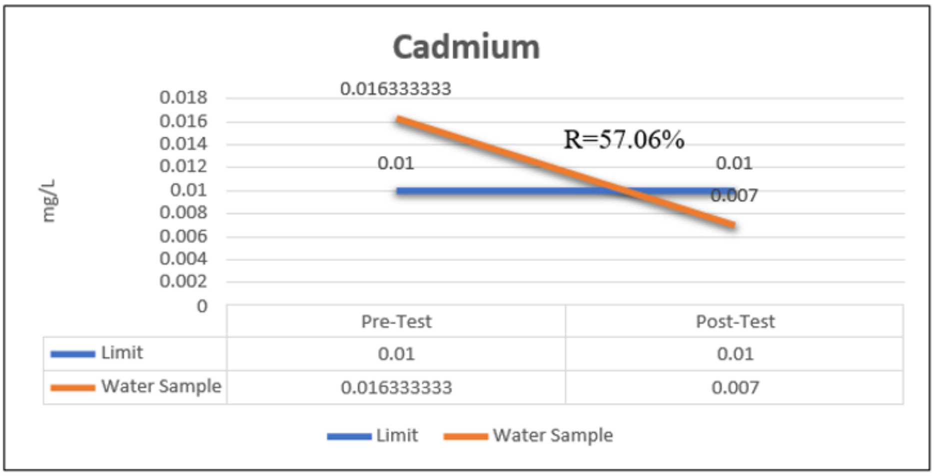

Figure 2: Analysis of Cadmium Levels

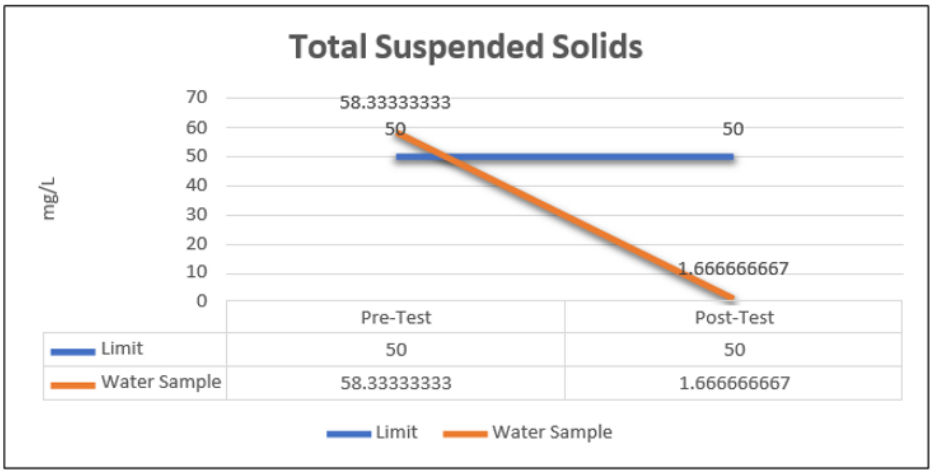

Figure 3: Analysis of Total Suspended Solids Levels

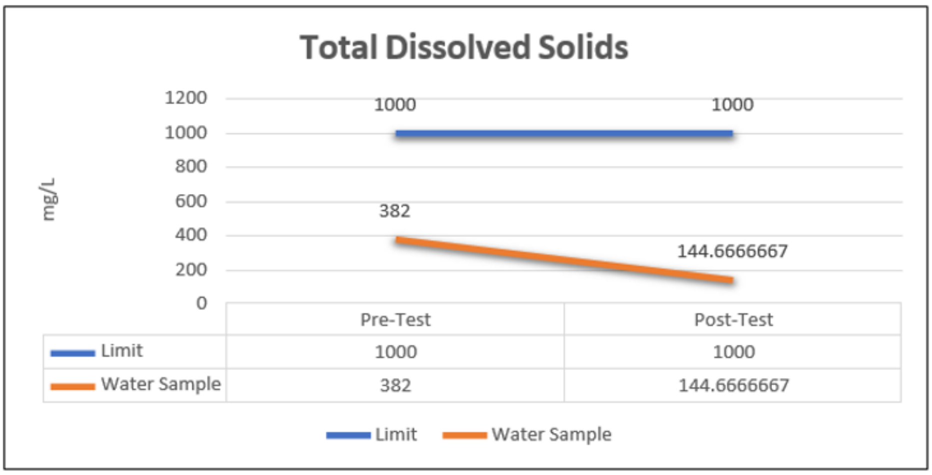

Figure 4: Analysis of Total Dissolved Solids Levels

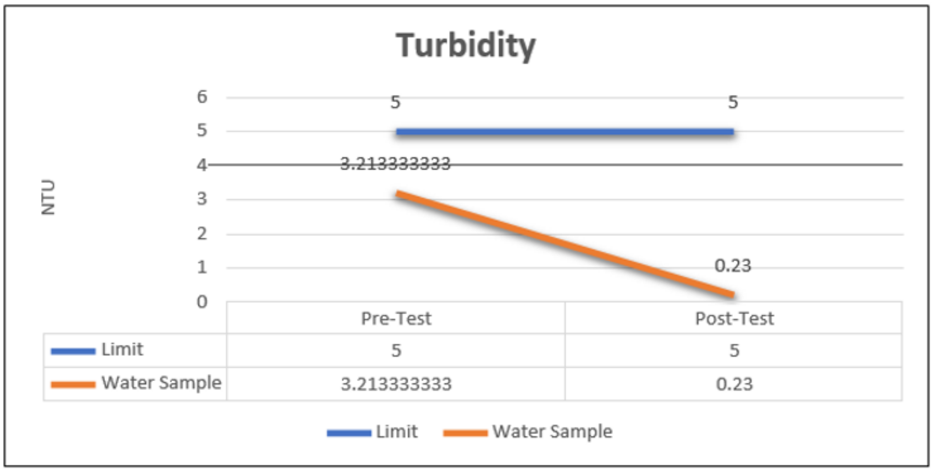

Figure 5: Analysis of Turbidity Levels

Table 4 shows the paired t-test results to determine if there's a significant difference between the pre-test and post-test results. Lead was ignored as it is undetectable in both before and after intervention. The results showed a significant decrease in the level of Cadmium, TSS, TDS, and Turbidity before intervention to after intervention. To verify these results, Table 5 shows the magnitude of the experimental impact using the effect size. The greater the association between two variables, the higher the effect size. The guidelines for interpretation of the value are 0.01 = small effect, 0.06 = moderate effect and 0.7 = large effect (Cohen, 1988). As the ETA squared calculated are 0.84, 0.98, 0.99 and 0.99, the intervention greatly improves the quality of water sample in terms of Cadmium, Total Suspended Solid, Total Dissolved Solid and Turbidity levels.

Table 4: Paired T-Test Results

Variables	Mean	Standard Deviation	t-value	p-value	Decision
Cadmium					
Pre-test	0.016333333	0.004933	3.277152121	0.015291278	Reject Null Hypothesis
Post-test	0.007	0			
TSS					
Pre-test	58.33	10.41	9.426278541	0.000353068	Reject Null Hypothesis
Post-test	1.67	0.29			
TDS					
Pre-test	382	22.27	17.86712783	0.000028832	Reject Null Hypothesis
Post-test	144.67	5.77			
Turbidity					
Pre-test	3.21	0.023	179	0.0000000029	Reject Null Hypothesis
Post-test	0.23	0.017			

Table 5: Effect Size Results

Variables	Eta Squared ($\eta 2$)
Cadmium	0.84
TSS	0.98
TDS	0.99
Turbidity	0.99

CONCLUSION

After conducting the analysis for the pre-treated water, it gave positive results in terms of Lead, Total Dissolved Solids and Turbidity. The pre-treated water from Lake Mainit gave unsatisfactory results in its Cadmium and Total Suspended Solid Levels. Surpassing level of Cadmium, a heavy metal, is alarming for the health of the residents. On the other hand, based on the analysis of treated water, the granulated activated carbon bed filter tank lowered the concentrations of some parameters, thus improving the water quality. With the considerable 57.06% removal efficiency of Cadmium heavy metals, statistically significant pre-test and post-test results, and large effect sizes, the granulated activated carbon bed filter tank made from coconut shells effectively reduces the present heavy metal in water and also in improving water quality.

RECOMMENDATION

This study used Granular ACb from Coconut Shells with specified size of 1.2 mm ACb and acquired from

Cocoprime Activated Carbon Corporation in Aurora, Zamboanga del Sur. The source of water sample was acquired from lake's surface specifically near Barangay San Juan, Alegria, Province of Surigao del Norte. Midstream and downstream water samples were not taken as subjects for this study due to the unavailability of assistance when the study took place in the middle of Covid-19 Pandemic. The weather condition was rainy and the place is experiencing Northeast Monsoon climate, so distribution of parameters may vary depending on the climate. pH was not measured as water samples have to be refrigerated due to shipping means and laboratory availability. The water samples underwent laboratory testing to assess and evaluate the TDS, TSS, Turbidity and heavy metals in Waterlab Facility of University San Carlos, Talamban, Cebu.

The used granular ACb after 3 days intervention did not undergo laboratory testing and measurement of the ACb's surface area was not determined due to unavailability of equipment in the country. Disposal time of ACb bed filter was not known.

With these constraints, it is highly recommended to other researchers to conduct a study that involves upstream, midstream and downstream water samples, to conduct in multiple locations around Lake Mainit, to consider different seasons of the year, to include pH as one of the parameters in the study, to find out the surface area of the granular ACb and lastly, to determine the period the ACb Bed Filter will be saturated for disposal. Also, other researchers may consider testing the efficiency of the filtration system for verification on some known water with significant lead content as well as to other heavy metals like mercury.

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