Towards Sustainable Laboratory Practices: Eco-Friendly Treatment of Decentralized Wastewater

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Abstract— **Wastewater from laboratories are less considered due to their low quantity and decentralization even though it gives detrimental impact to the human and environment. Investigate the contaminants in laboratory wastewater and find an efficient and economical treatment for small scale laboratories. Prototype model of horizontal flow constructed wetland system with bio-geo filters designed, constructed and operated with locally available utilized materials as filter media. Isotherm and kinetic studies carried out for Cinnamon and Neem biochar and clay composite. Laboratory wastewater diluted to desirable pH with pipe borne water was allowed to pass through the treatment system. Higher removal efficiencies of 80% BOD_5 and 86% COD observed in the treatment system may due to the presence of biofilm in final treatment tank and clay: neem biochar composite 1:1 as filter media. Operating the treatment system of horizontal flow constructed wetland with bio-geo filters gives eco-friendly and efficient solution for small scale, decentralized highly contaminated laboratory wastewater with heavy metals.**

Keywords: Decentralized, Constructed Wetland, Heavy metal, Ecofriendly, Composite

Introduction

Wastewater is used water that has been categorized as industrial, commercial & domestic wastewater based on its users. Wastewater from the laboratories contains hazardous chemical wastes, reactive chemicals, corrosive waste, radioactive wastes, infectious wastes, infectious microorganisms, and toxic chemicals, etc., based on the function of the laboratory. Physical and chemical pollutants from laboratory wastewater contain heavy metals such as As, Cd, Hg, Pb, Cr, Mo etc, Soluble Organic materials, Inorganic Particles (sand, grit, metals, ceramics), Soluble inorganic materials (Ammonia, Cyanide, Hydrogen Sulphides), Toxins (Pesticide, Herbicide, Poisons), Pharmaceuticals, etc [6 , 22]. Biological pollutants from laboratory wastewater contain pathogens (Bacteria, Viruses, Protozoa, and Parasites) [2].

Laboratory waste generated from various commercial, industrial and other institutional sources causes an immense environmental problem. It affects the health and quality of life of people, particularly the residents in urban and sub urban areas. Heavy metals have negative effects on the growth of aquatic organisms and depress the number of living organisms in an aquatic eco system [19,25]. Amongst the pollutant's pesticides, heavy metals, and detergents are the major cause of concern for the aquatic environment because of their toxicity, persistency, and tendency to accumulate in organisms [30].

These heavy metals are usually present in trace amounts in natural waters but many of them are toxic even at very low concentrations [3]. Metals such as Arsenic, Lead, Cadmium, Nickel, Mercury, Chromium, Cobalt, Zinc, and Selenium are highly toxic even in minor quantities. Consumption of heavy metals through vegetables, corn products leads to kidney dysfunction, renal dysfunction, bone defects, etc. Diarrhea, typhoid, paratyphoid and other Salmonella type diseases are caused by bacterial pathogens. Large amounts of many inorganic substances can contaminate soil and water. Some are toxic to animals and humans, may accumulate in the environment. Acute poisonings from heavy metals in drinking water lead, potential long-term health effects.

In srilanka, Central Environmental Authority the implementing body of government has framed regulations for management of hazardous wastes.

"Guidelines for the Management of Scheduled Waste in Sri Lanka in accordance with the National Environmental (Protection & Quality) Regulation No. 01 of 2008" in practice and applicable for laboratory solid and liquid wastes. However, these regulations were not fully implemented yet due to several practical problems, especially lack of laboratory facility for identification and classification of waste in Sri Lanka and more significantly the absence of an environmentally sound final disposal facility in the country [4].

Most of the laboratories in Sri Lanka, onsite storing their hazardous wastages for a maximum period of 90 days or a maximum quantity of 10,000 kgs, permitted by the CEA. Collection and transportation of scheduled waste to offsite storage, treatment and /or disposal facility done by the registered and licensed person. Ultimately these scheduled wastages can undergo Recovery Co Processing and Recycling, Treatment and Disposal process based on its category. Further, this storage amount of wastewater for 90 days is considerably very low, toxic and harmful. Categorizing, storing, labelling and handing over to licensed collectors also quite time-consuming procedure. Some laboratories are operating owned treatment and disposal systems, and some are operating pretreatment system before discharge in to the central treatment plant. Meanwhile, wastewaters from few laboratories were released to the canals, rivers, sewer networks, municipal wastes, and other water streams without proper treatment.

Faculty of applied science, University of Vavuniya has constructed a new building for physics and chemistry laboratory. This treatment unit is constructed to treat the physical and chemical wastewater from the laboratory. Generally, laboratory wastewater treatment is small scale, decentralized systems with highly contaminated toxic, hazardous and inorganic substances. Wastewater from laboratories are less considered due to their low flow rate (Small scale), even though it gives more harsh impact on the human and environment. In order to find a solution for this, solutions like conventional treatment system cannot be suggested due to its corrosiveness of pipes, flammability, and chemical treatment process are very expensive and difficult to maintain. Constructed wetlands can do physical, chemical and biological treatment in one simple unit and it is suitable for decentralized systems. Horizontal flow constructed wetlands are very effective in removal of organics, suspended solids, microbial pollution and heavy metals. Constructing the treatment unit with locally available materials and plants leads construction of economical treatment unit. Besides their low operation and maintenance cost, horizontal flow constructed wetlands mostly do not use electric power and flow is supported only by gravity. As there is scant researches and solutions found specifically in this area, it will be a great proposal in removing harmful elements in laboratory wastewater in an environmentally friend manner. The objectives is to design and implement constructed wetland using clay- biochar composite to remove laboratory wastewater contaminants and To assess the quality and quantity of pollutants in laboratory wastewater.

MATERIAL AND METHODS

A. Methods

The wastewater samples from each sink of the laboratory connected to the storage tank through a constructed open channel. This composite sample underwent physical, chemical and biological characterization of wastewater. Purpose of preparing the composite sample is to get more reliable results than the grab sample. Type of constructed wetland, selection of filter media, plants based on raw laboratory wastewater characterization and literature survey. Treatment unit with filter media such as Calicut tile, 10mm aggregates, biochar and clay were constructed as layers. Filter media such as Calicut tile, 10mm aggregates, biochar, and clay were constructed as a layer in treatment units. Wetland plants were planted on each tank in order to remove pollutants through phytoremediation. The concentration of influent and effluent water was taken to determine and analyse the removal efficiency of the treatment system.

B. Materials

Collection of Raw Materials

Clay deposits are spread throughout Sri Lanka and many of these deposits contain a wide variety of clay mixtures. Murunkan clay has a greater percentage of Montmorillonite (MMT), layered clay having high cation exchange capacity and multi-cation adsorption ability [28]. Several studies confirm the presence of Montmorillonite clay in the Murunkan basin [10]. During the dry season, it was easy to collect the MMT rich clay sample from Gaint tank basin located in Murunkan area, Northern Province. As per the previous studies and literature survey, these samples were collected at a depth of 0.9m from the surface.

Cinnamon biochar collected at Down Draft gasifier from a hotel, which is used to produce syngas production of the boiler. Chopped cinnamon pieces with 20% of moisture are used as fuel for the Gasifier. By product of this Gasifier is called Biochar, used as one of the filter media of the treatment system. Neem biomass collected from Vavuniya, which under goes the thermochemical process in a brick furnace. Calicut tile was crushed in to small piece (approximately 12mm) and used as filter media. The layer depth of the Calicut layer was 10.16cm and it was washed thoroughly with pipe borne water prior to place in the treatment unit. Further, 10mm aggregates from the research laboratory also used to fill the tank bottom for 2".

Plant growth provides a vegetative mass that deflects flows and provides attachment sites for microbial development. In addition, plants stabilize substrates while enhancing its permeability, and plants add greatly to the aesthetic value of the wetland [8]. Wetland plants such as Cannas and Scirpus Grossus, were planted in tanks 1, 2, 3 & 4 respectively. Cannas plant roots generally penetrate 2 to 5" depth in its supporting media.

The final tank is full of slurry with murunkan clay, produces biofilm to facilitate the micro-organism presence during the operation phase. Cannas and Scirpus Grossus were collected from the Kelaniya Peliyagoda wetland area. These plants and specially its roots were thoroughly washed with pipe borne water prior to place in the treatment unit.

C. Preparation of clay biochar composite

Cinnamon and neem biochar grinded with motor and pestle and sieved through 150μm sieve. Clay from Murunkan basin also grinded and sieved through 150μm sieve. These are mixed by 1:5, 1:3, 1:1, 3:1, 5:1 mass ratios and prepared small threads and then allowed to undergo slow pyrolysis at 400°C in muffle furnace Model No Sx2-5-12.

D. Adsorption isotherm and kinetic studies

Adsorption kinetic studies and isotherm were done in order to analyze the hydraulic retention time and adsorption capacity respectively. 30g of clay-biochar composite pallets prepared (stated in materials) and stirred continuously with 1500ml laboratory wastewater (Measure initial COD). 100ml of samples were withdrawn at time 0, 3, 5, 7, 15, 30, 60, 120, 240, 480, 720, 1440, 2880 min. Then Supernatant was collected at a particular time interval and passed through a 0.45μm Nylon filtration membrane to find m/v ratio and COD test. Phosphorus variation with time and pH also studied.

E. Design Approach

The system was worked out to suit the topographical condition, less flow rate and pollutant concentrations of wastewater. The BGF design was done by using the design concepts of Subsurface Flow Constructed Wetlands. The selection of filter media, detention time, influent concentration; selection of plants plays an important role in the design approach of horizontal flow constructed wetlands.

COD is the key parameter in the raw water sample, and isotherm and kinetic studies were conducted to analyze its variation.

1). Hydraulic Design of Filter Media

Data such as Quantity of Wastewater inflow, Influent BOD_5, Characteristics of filter media, Root depth of proposed macrophytes are required for substrate design purposes. The BOD_5 removal in the wetland was described by First-order reaction model as given below [31],

$$Ce=Ci\ e^{-K_T t}$$

Influent BOD_5 value was taken from the composite sample collected from the laboratory. Effluent BOD value was considered as CEA's Effluent discharge standard "Tolerance limits for the Discharge of Industrial Wastewater in to Inland Surface Waters".

RESULTS AND DISCUSSION

A. Characterization of Laboratory Wastewater

Laboratory wastewater from each sink of the laboratory connected to the collection tank. Influent pH measured once a day from the influent collection tank. The EC values give measure of the total water-soluble ions. It can affect the plant growth negatively as it tends to decrease water uptake by plant roots resulting reduced nutrient intake [29].

Table 1 - Important Parameters of the Influent Wastewater

Parameter	Unit	Results
Ph @ ambient temperature		3.05
Electric Conductivity	ms/cm	1294
Biochemical Oxygen Demand (BOD5 in five days at 200C)	mg/l	16000
Dissolved Oxygen	mg/l	256
Total Dissolved Solids	g/l	773
Resistivity	MΩ	-100
Turbidity	NTU	940
Fluorite	mg/l	106
COD	mg/l	27,000

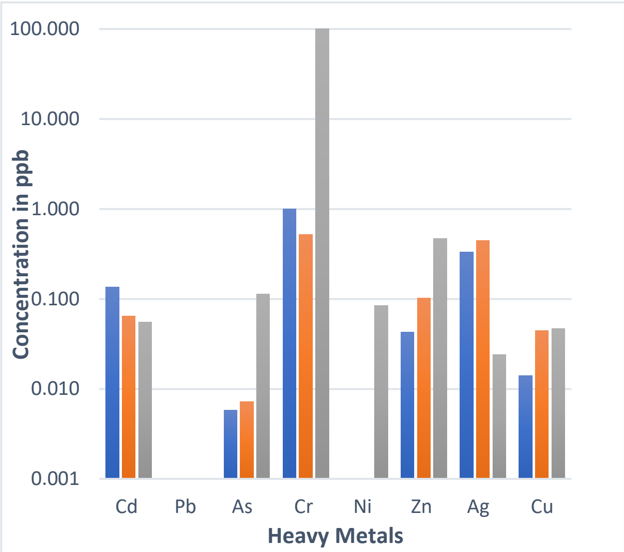


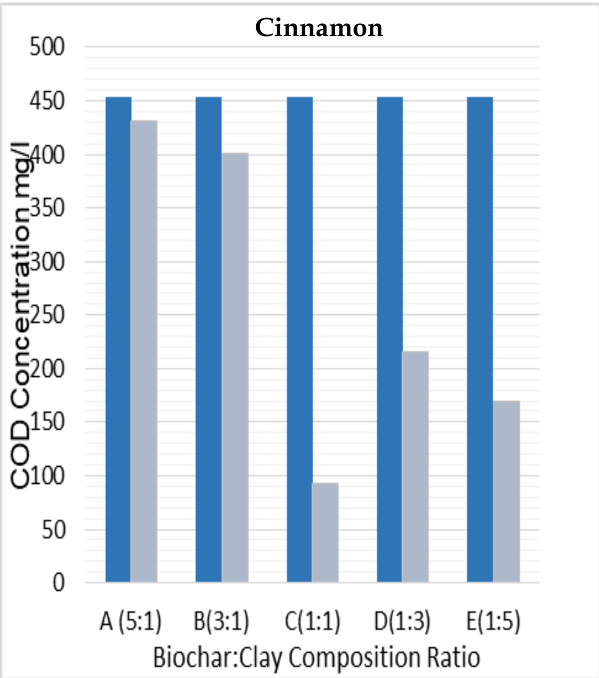
Fig 1 - Elements of Laboratory Wastewater

Key parameters such as Electric Conductivity, Phosphorus, BOD_5, COD in the influent wastewater is extremely high due to its harmful chemicals content. Suspended solids are mainly removed by physical mechanisms, such as sedimentation and filtration processes. Since it is the analytic laboratory wastewater, it consists considerable amount of suspended solids and higher amount of inorganic substances. Further analysis was done to identify the existing elements in the laboratory wastewater as depicted in fig 1.

B. COD removal efficiency of clay biochar composite

Clay biochar composites were prepared for various Clay: Biochar mass ratios then pyrolyzed at 600^0C for two hours in a muffle furnace (Witeg, Germany) and then immediately quenched in water at room temperature to activate surface pores. Then those composites were soaked in wastewater sample for 24 hours in ambient temperature. COD value before and after soaking period was determined in order to find the efficiency of the composite.

Fig 2 represents the COD Concentration variation for both type of composites. From the results we can get that among the Clay: Biochar mass ratios, 1:1 ratio composite for both Neem and Cinnamon shows greater efficiency in COD removal. Hence, Clay: Biochar = 1:1 ratio considered here for further adsorption and kinetic studies. Neem BC and Cinnamon BC clay samples compared in this study to find the best BC-Clay composite prior to place in the treatment system. Purpose of this laboratory scale experimental study is to find best composite ratio and locally available source materials.



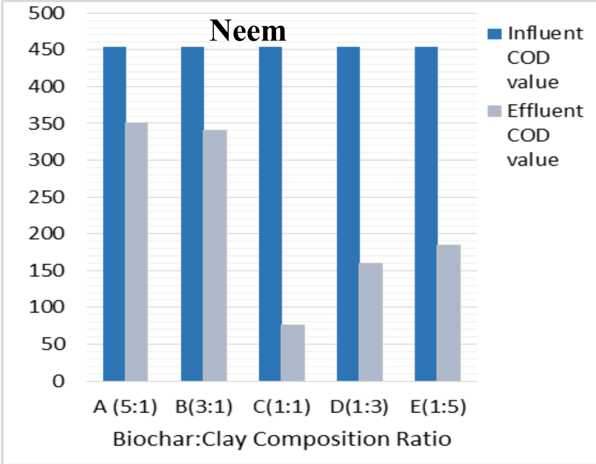


Fig 2 - Influent and Effluent Concentration of Different Composites for Cinnamon and Neem biochar composites

C. Adsorption Studies

The kinetic studies describe the COD removal rate with the changes in the contact time of on the solid-solvent interface [26]. The COD removal process from the solution to the adsorbents is simultaneously controlled by several processes, such as adsorption onto the surface, external diffusion, and/or pore diffusion, which can all be using adequate kinetic models [18].

As shown in the Fig 7, the adsorption rate of COD on to both Neem biochar and Cinnamon biochar composites was initially fast, followed by a slower adsorption until approaching the equilibrium. Fast sorption during the first stage may be due to a large number of vacant sites available for adsorption [34]. The adsorption by the Neem - BC composite was better than that by the Cinnamon – BC Composite.

Lagergren's pseudo-first-order model is expressed as $qt = qe\ (1 - e^{-k1t})$ where qt and qe (mg/g) are the adsorption capacity at time t and equilibrium, respectively, and $k1$ is the Lagergren rate constant of pseudo-first-order adsorption (min^{-1}). Plotting log ($qe - qt$) vs. t should give a linear relationship with slope qe and with intercept $k1$. Pseudo-second-order model is expressed as $q_t = \frac{k_2 q^2{}_e t}{1 + k_2 q_e t}$ where k_2 is the pseudo-second-order adsorption rate constant (g/mg·min). Fig 3 and Fig 4 gives the graphical representation of the pseudo-first-order model and Pseudo-second-order model experiment values respectively.

To obtain the adsorption isotherms for two kind of adsorbents Freundlich model was applied. The equation of the Freundlich isotherm was followed, $\log[x/m=\log[kf+1/n \quad \log c\ ]\ ]$, where KF represented the Freundlich constant and 1/n was a parameter related to the intensity of adsorption, which varied with the heterogeneity of the material. Relative

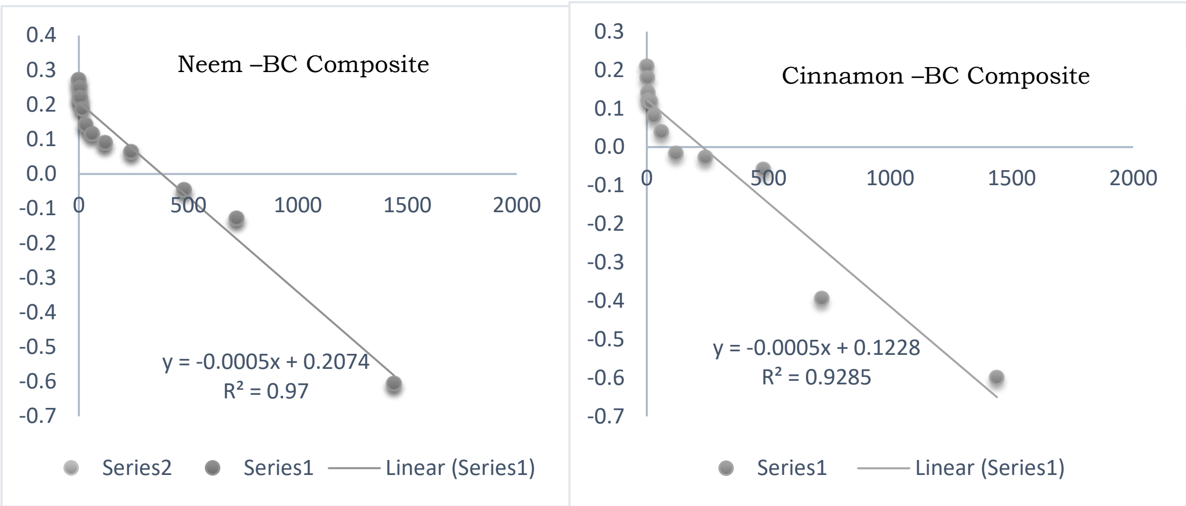


Figure 3 - Pseudo First Order Kinetic Model for Neem – BC and Cinnamon –BC composites

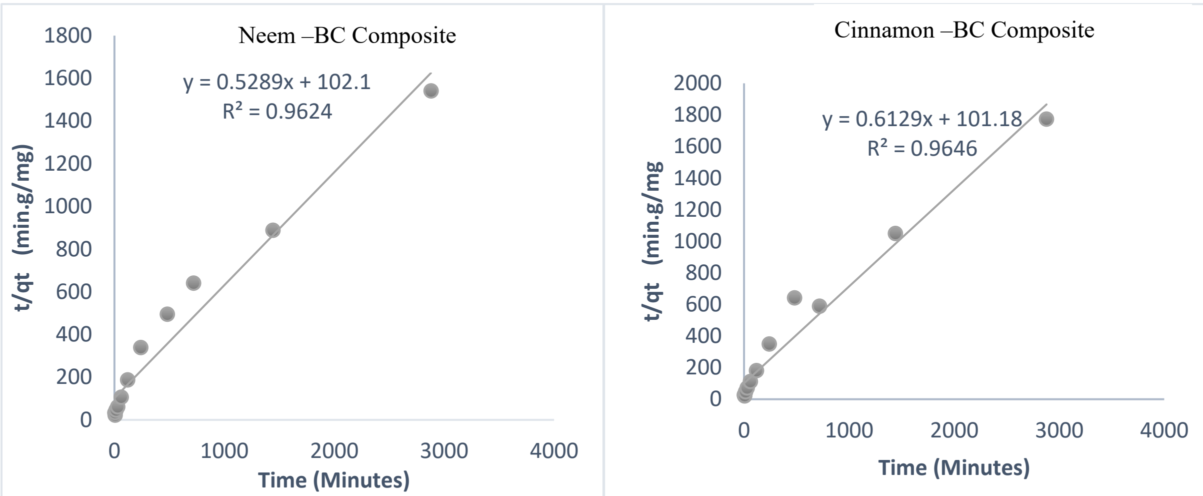


Figure 4 - Pseudo Second Order Kinetic Model for Neem – BC and Cinnamon –BC composites

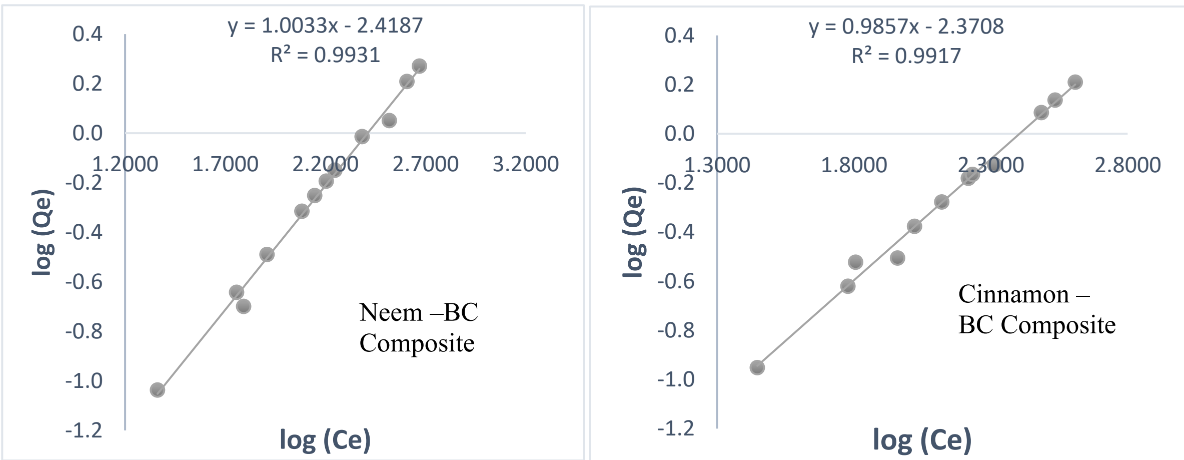


Figure 5 - Freundlich Isotherm model for Neem – BC and Cinnamon –BC composites

Table 2 -Kinetic model parameters for the adsorption of COD onto Neem-BC and the Cinnamon - BC

Model	Parameters	Unit	Neem	Cinnamon
Pseudo-First-Order Kinetics	Qe	mg/g	1.868	1.624
	K1	1/min	-0.0005	-0.0005
	R^2		0.97	0.9285
Pseudo-Second-Order Kinetics	K2	g/mg/min	0.00281	0.00375
	R^2		0.9624	0.9646
Freundlich Isotherm	n		0.9967	1.015
	R^2		0.9931	0.9917
	Kf	$(mg\ g^{-1})/(mg\ L^{-1})^n)$	0.00381	0.004258

parameters of the model can be calculated from the slope and intercept of the plots. Fig 5 shows the graphical representation Freundlich isotherm model.

Fitting order of kinetic model is determined to be Freundlich Isotherm > pseudo first order > pseudo second order for Neem Biochar and Freundlich Isotherm > Pseudo Second Order > Pseudo First Order for Cinnamon.

Sorption equilibrium data was tested on the Freundlich isotherm model in order to understand the COD removal mechanism. The data was best fitted with Freundlich model with R^2= 0.9931 for Neem – BC and R^2= 0.9917 for Cinnamon - BC (Table 2). Freundlich isotherm model describes adsorption occurring on heterogeneous and amorphous surfaces, which have different adsorption energies not restricted to monolayer adsorption or finite sorbent uptake capacity [17]. The Freundlich exponent, n indicates favorability of the adsorption. Since n < 1 (0.9967) for the composite which mad of Neem –BC, the COD adsorption can be suggested as a favorable process to heterogeneous biochar surface.

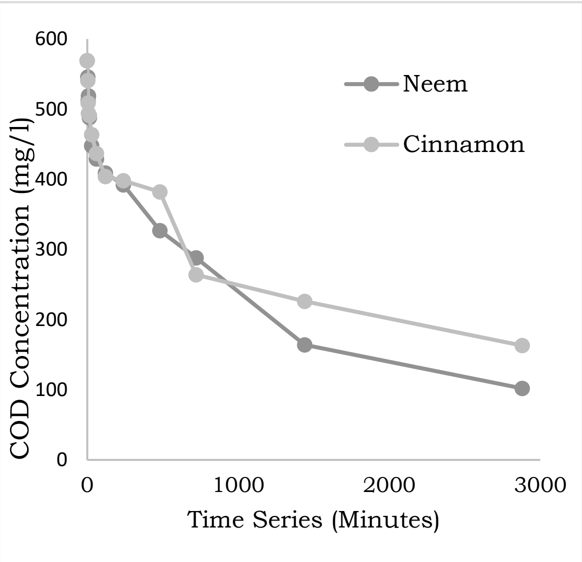


Figure 6 - COD concentration variation with time

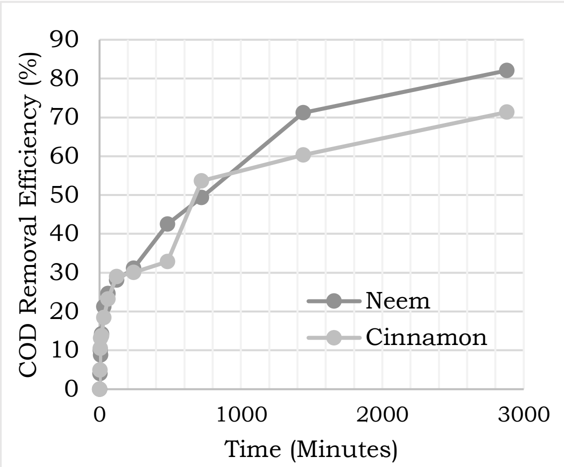


Figure 7 - COD Removal Efficiency with time

As shown in the Figure 6, the concentration of COD reduced along with increase of contact time. The removal efficiency has been increased with time for both of the adsorbent. But as shown in the Fig 7 adsorbent made from Neem biochar composite shows a higher removal efficiency than that from Cinnamon biochar composite.

D. Design Approach of the Treatment System

Horizontal flow subsurface constructed wetland treatment system is designed and constructed with locally available materials. Constructed wetland is selected due to its simplicity, inexpensiveness, eco-friendly and efficient treatment for heavy metals. Typically, filter media for wetlands include laterite soil, sand, gravel, etc. Gravel is used as the filter media as it provides a large surface area for biochemical processes. It provides high conductivity which is required to stabilize hydraulic retention time of wetland. Sand is considered as the most suitable filter media in relation to hydraulic and organic loading. In this research biochar, 20mm aggregates and Calicut tile are selected as filter media. Sand and coir mixed layer followed by 24mm thick laterite layer was designed to facilitate the vegetation roots growth and water supply for plants. Laboratory wastewater sample contains lots of pollutants at high concentrations, especially heavy metals. Sand and gravel cannot treat the wastewater in an effective manner. Calicut tile and 20mm coarse aggregates used as filter media which, helps to increase the permeability of wastewater treatment system. Since concentration of influent wastewater is severely high, filter media such as sand and aggregates cannot achieve the CEA's discharge standard of effluent into water bodies. Biochar has a relatively structured carbon matrix with a high degree of porosity and extensive surface area, suggesting that it may act as a surface sorbent which is similar in some aspects to activated carbon and thereby play an important role in controlling contaminants in the environment (Anthony O,2025). Much work has been done on Biochar is having a large surface area, charged surface, functional groups [21]. Hence, filter media such as biochar is used here to remove heavy metals from the wastewater.

Four rectangular tanks designed in continuous flow in the treatment system. Each tank with the dimension of 3m length, 0.75m wide, and 1m deep. First 3 tanks having the same filter media and cannas plant and 4th tank is with full of clay slurry with Scirpus Grossus plant. These plants collected from Kelaniya Peliyagoda wetland area & these roots were thoroughly washed several times prior to place in the treatment plant. Here main filter media is biochar with K 86%, Si-7.4%, P-2.7%, S-2%, Ti-0.6%, Mn-0.4% & Fe-2.0% and clay with Si 55.5%, Al-17.1%, S-0.3%, K-2.9%, Ca-3.68%, Ti-2.27%, Mn-0.33%, Fe 16.10%). Filter media biochar obtained from gasification process while clay was from basin of Gaint tank in Mannar.

E. Subsurface Horizontal Flow (SSHF) Constructed Wetland System

1) Hydraulic Detention Time

Data's such as quantity of wastewater inflow, influent BOD5, characteristics of filter media, root depth of proposed macrophytes are required for substrate design purposes. The BOD_5 removal in the wetland was described by first-order reaction model by Reed's equation, Ce=Cie-KT t [31].

First-order temperature dependent rate constant (K_T) was calculated using the following equation given by Ronald Crites (1994).

$K_T = K_{20} (\theta)^{(T-20)}$ [32]

K_{20}=1.35 [26]

Temperature=30°C, θ=1.06

Hydraulic detention time (t) $= \frac{ln C_i - ln\, C_e}{K_T}$ [20],

Relatively small hydraulic retention time of 2880minutes achieved from design calculation of treatment system, when compared with other systems.

Influent BOD_5 value was taken from the laboratory wastewater collection tank. Effluent BOD_5 value was considered as CEA's effluent discharge standard "Tolerance Limits for the Discharge of Industrial Wastewater in to Inland Surface Waters".

F. Characterization of Effluent Laboratory Wastewater

In this treatment system wastewater is fed in at inlet tank no 1 and then flows slowly through the porous medium under the surface of bed. Then it travels to tank no 2, tank no 3, and tank no 4 to outlet. During the passage the lab wastewater comes in contact with a network of aerobic, anoxic and anaerobic zones. Most of the bed is anoxic/ anaerobic due to permanent saturation of the beds. The aerobic zones occur around roots and rhizomes that leak oxygen to the substrate. Here, turbidity, EC, COD, TDS, BOD_5 reduces when wastewater passage through the treatment system. Higher P observed in tank no 1, 2 & 3, but considerable reduction observed in tank no 4, may be due to the MMT filter media and plant. Higher P observed in the tank 1, 2, & 3, may be due to the biochar filter media under anoxic/ anaerobic condition.

Table 3: Influent and Effluent of treatment system

Testing Parameter	Unit	Influent	Effluent
Turbidity	NTU	2.5	1.42
pH at ambient temperature		6.9	7.4
Biochemical oxygen demand ((BOD_5 in five days at 200cor BOD_3 in three days at 27^0c)	mg/1, max.	100	20
Chemical oxygen demand (COD)	mg/1, max.	1432	202
Dissolved phosphates (as P)	mg/1, max.	1.2	11.03
Total Kjeldahl nitrogen (as N)	mg/1, max.	1.7	0.93
Electrical Conductivity	µS/cm	640	241
Dissolved Oxygen	mg/1		
Total Dissolved Solids	mg/l	641	216
Salinity	g/l	0.7	0.4
Resistivity	MΩ	-1	-1
Temperature	° C	29.1	28.5

Laboratory wastewaters are small quantity and highly contaminated, composite sample of laboratory wastewater collected from collection tank and then allowed to pass through the treatment system. Effluent quality from each tank was tested for each batch of samples. Key parameters such as electric conductivity, Total Kjedahl nitrogen (as N), Biochemical Oxygen Demand (BOD_5 in five days at 20ºC), Dissolved phosphates (as P) and Chemical Oxygen Demand (COD) were reduced and effluent satisfies the discharge standard of "tolerance limits for the discharge of industrial wastewater in to inland surface waters". Reduction in COD may be due to the filter media of clay-biochar composite. More BOD_5 reduction observed in tank no 4 due to its filter media. Variation in electric conductivity gradually decreases from tank no 1 to 4. Dissolved phosphates (as P) and Total Kjeldahl nitrogen (as N) also reduces and satisfies the CEA's discharge standard [4].

Parameters such as BOD, COD in the influent wastewater is extremely high may be due to the long-term storage and degradation of harmful chemicals. Higher removal efficiency of 80% BOD and 86% COD observed in the treatment system and this may due to the Clay biochar composite filter media and biofilm filter media in the treatment system. Slightly higher pH increase observed during passage through the bed due to the filter media of Neem charcoal. Variation in electric conductivity gradually decreases from tank no 1 to 4. Variation of physical and chemical parameters in the treatment system depicted in Table 3.

Higher removal efficiency of 80% BOD and 86% COD observed in the treatment system. BOD and COD removal probably mainly occurred by adsorption with biochar and the anaerobic process. Slightly higher pH increase observed during passage through the bed due to the filter media of biochar due to its alkalinity nature.

CONCLUSION

Sending the composite sample of laboratory wastewater through the treatment system is an important practice and it leads ethical result to analyze. Treatment system was prepared with locally available utilized materials and results overall good removal efficiency in laboratory wastewater contaminants. Over the other pollutants, BOD_5 and COD results more than 80% of removal efficiency due to the clay: neem biochar 1:1 composite as a filter media. Thus, clay: neem biochar 1:1 composite, biochar plays the major role in removing contaminants especially the heavy metals from the laboratory wastewater and reason for 86% of COD reduction in the treatment system. Horizontal flow constructed wetland with locally, onsite available materials result good removal efficiency for key parameters such as BOD_5, COD. Meanwhile, this treatment unit is cost effective and eco-friend solution for decentralized, small scale high toxic chemical wastewater treatment.

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