Clay biochar composites in treating laboratory wastewater

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***Abstract*—Laboratory wastewater contains hazardous physical and chemical pollutants which results detrimental impact to the human, animals and environment. Horizontal flow constructed wetland with clay - biochar composite, Calicut tile, biochar, clay, wetland plants, 20mm aggregates by natural resources available at nearby places. Laboratory Wastewater collected from the collection tank diluted to desirable pH with pipe borne water was allowed to pass through the treatment system. Slightly higher pH increase observed during passage through the bed due to the filter media of biochar's alkalinity nature. Higher removal efficiencies of 80% BOD and 89% COD observed in the treatment system may due to the presence of biofilm in final treatment tank and clay: biochar composite as filter media. Gasified cinnamon biochar by-product of syngas production, plays a key role in reducing COD by 86% in the proto type model due to its distinctive physiochemical properties such as higher BET SSA, morphology of SEM analysis, lower H/C & higher O/C molar ratio in CHN analysis. Higher removal efficiency of BOD and COD observed for horizontal flow constructed wetlands constructed by utilizing onsite available materials and clay - biochar composites. The dispersed small laboratories can benefit more from running an economical and ecologically friendly treatment system with locally accessible natural materials.**

Keywords: Clay biochar composite, Constructed Wetland, Heavy metal, biochar

INTRODUCTION

Laboratory wastewater, often containing a complex mixture of recalcitrant and toxic organic and inorganic pollutants, requires effective treatment strategies to mitigate its environmental and health impacts [3,25]. Traditional wastewater treatment technologies frequently encounter limitations such as high energy consumption, risks of secondary pollution, and insufficient capacity to address complex contaminants [20,31]. Consequently, there is an urgent need for advanced, sustainable, and cost-effective methods for wastewater remediation [20,34]. Biochar, a carbon-rich material produced from biomass pyrolysis under oxygen-limited conditions, has emerged as a promising eco-friendly adsorbent for environmental remediation due to its unique physicochemical properties, including high specific surface area, porosity, and abundant surface functional groups [25,16,29,17]. It is also recognized for its low production cost and carbon sequestration potential [16,29]. However, pristine biochar often has limitations such as small pore size and low surface functionality, which can hinder its adsorption efficiency for various pollutants [24]. To overcome these limitations and enhance performance, biochar is frequently combined with other materials to form composites [24,32,28]. Clay minerals, being inexpensive, non-toxic, and naturally abundant adsorbents, offer complementary properties to biochar [13,9]. The integration of biochar with clay minerals results in clay-biochar composites, which leverage the advantages of both components, leading to improved adsorption capacities, enhanced surface active sites, and better structural stability [13,9]. This introduction explores the rational design, characteristics, and application of clay-biochar composites as an advanced and sustainable approach for treating laboratory wastewater, highlighting their role in addressing contemporary water pollution challenges.

Biochar and its composites, particularly with clay, represent an innovative intersection within these methods, primarily acting as advanced adsorbents for physical treatment, though they can also be engineered for chemical and biological enhancements [24,13,32,28] . Clay-biochar composites are highly effective in removing heavy metals such as cadmium (Cd(II)), chromium (Cr(VI)), and copper (Cu(II)) [17]. The increased cation exchange capacity and surface area of the composites facilitate the binding of these metal ions [17].

In srilankan context, Laboratories 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. 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. 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. This paper aims to investigate an ecofriendly solution for small scale decentralized wastewater treatment system.

MATERIAL AND METHODS

A. Methods

There are 4 no of 20L plastic containers and, 3 no of 5L plastic containers are stored in the environmental laboratory. These samples were poured and mixed together in another 500-liter capacity vessel. This composite sample underwent physio chemical parameter analysis. 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. 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

The MMT rich clay sample from the Gaint tank basin in the Northern Province's Murunkan area was simple to gather during the dry season. These samples were taken at a depth of 0.9 meters below the surface in accordance with earlier research and review of literature. 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.

Calicut tile was utilized as filter material after being crushed into small pieces, about maximum 12mm in size. Before being placed in the treatment unit, the Calicut layer, which had a layer depth of 10.16 cm, was carefully cleaned with pipe-borne water. Additionally, the tank bottom was filled with 10mm aggregates from the environmental laboratory for a depth of 2". A vegetative mass produced by plant growth deflects flows and offers attachment sites for the growth of microorganisms. Furthermore, plants improve the permeability of substrates while stabilizing them, and they significantly raise the wetland's aesthetic value [10]. Cannas, Scirpus Grossus, and other wetland plants were planted in tanks 1, 2, 3, and 4, accordingly. The roots of cannabis plants typically pierce the supporting material 2 to 5 inches deep. The last tank is filled with murunkan clay slurry, which forms biofilm to help microorganisms survive the operating phase. Scirpus Grossus and Cannas were gathered from the marsh area of Kelaniya Peliyagoda. Before being placed in the treatment unit, these plants—and especially their roots—were carefully cleaned with pipe-borne water.

C. Sample Preparation

Preparation of clay biochar composite

Cinnamon 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. Characterization of Materials

CHN elemental analysis done to define the by-product of syngas production as biochar using Perkin-Elmer 2400 (series II) CHN elemental analyser. SEM analysis done with The Hitachi SU6600 Scanning Electron Microscope. SEM technologies provide rapid, high resolution imaging, and images with high depth of field gives morphology of the gasified biochar. SSA of gasified Cinnamon Biochar was 563m^2/g measured using Brunauer-Emmett-Teller (BET) theory.

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.

1). Hydraulic Design of Filter Media

Data such as Quantity of Waste water 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 [30],

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

2). Inflow and Effluent Concentration:
Influent BOD 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 Waste Water in to Inland Surface Waters".

RESULTS AND DISCUSSION

A. *Characterization of Influent Laboratory Wastewater*

In this bio-geo filter horizontal flow constructed wetland treatment system, wastewater is fed in at the inlet and flows slowly through the porous medium of each tanks in a more or less horizontal path [6] until reaching the 4th tank outlet zone, where it is discharge to the inland surfaces. During the passage, the wastewater enters in to contact with a network of aerobic, anoxic and anaerobic zones [6]. The aerobic zones are found around the roots and rhizomes that leak oxygen into the substrate [8]. This treatment system is mainly anaerobic, with complex physical, chemical and biological mechanisms. Composite sample of wastewater underwent physical chemical and biological characterization and the effluent characteristic were tabulated against the CEA's standard "tolerance limits for the discharge of industrial wastewater in to inland surface waters". All the parameters are high especially; COD concentration in the laboratory wastewater is extremely high (Table 1).

Table 1: Parameters of Influent (Diluted sample)

Testing Parameter	Unit	Influent
Turbidity	NTU	2.5
pH at ambient temperature		6.9
Biochemical oxygen demand (BOD5 in five days at 200C)	mg/1, max.	100
Chemical oxygen demand (COD)	mg/1, max.	1432
Dissolved phosphates (as P)	mg/1, max.	1.2
Total Kjeldahl nitrogen (as N)	mg/1, max.	1.7
Electrical Conductivity	µS/cm	640
Dissolved Oxygen	mg/1	
Total Dissolved Solids	mg/l	641
Salinity	g/l	0.7
Resistivity	MΩ	-1
Temperature	° C	29.1

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 [14].

Key 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. 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.

B. *Physiochemical Properties of Biochar*

The biochar samples produced at lower temperatures be blocked by volatile matter and ash hence, decreasing the porous volume. However, the porosity of the biochar increased with increasing temperature. SEM analysis of the cinnamon biochar illustrates the smooth surface morphology of porous structure with numerous hollow cells. Large channel-like structures proliferate the surface of the biochar, showing at least partial retention of the morphology of the initial input biomass [26].

Biochar produced at higher temperature facilitated the release of volatile materials and created more pores, resulting in larger surface area [22]. Greater surface area is desirable because it aids in improving soil properties and increases water retention capacity to a greater extent [18] . By compared with other materials cinnamon biochar shows higher BET specific surface area of 563m2/g via BET analysis. Hence, it shows more affinity for adsorption and enable to separate various heavy metals from laboratory wastewater [33].

Breaking of weaker bonds in biochar structure and removal of water, hydrocarbons, H_2, CO and CO_2 during pyrolysis at high temperature may cause lowering in O and H contents in tea waste biochar pyrolysis at 700°C and rice husk biochar pyrolysis at 700°C than those biochars produced at <700 ºC [23,22]. Biochar produced at high temperatures indicates an increase of aromaticity. The H:C ratio of a biochar indicates its stability and aromaticity [18]. The <0.1 molar H/C ratio in biochar indicated a more graphite-like structure [35]. Hence, cinnamon biochar is rich in aromatic graphite-like structure expected to remove heavy metals in the laboratory wastewater.
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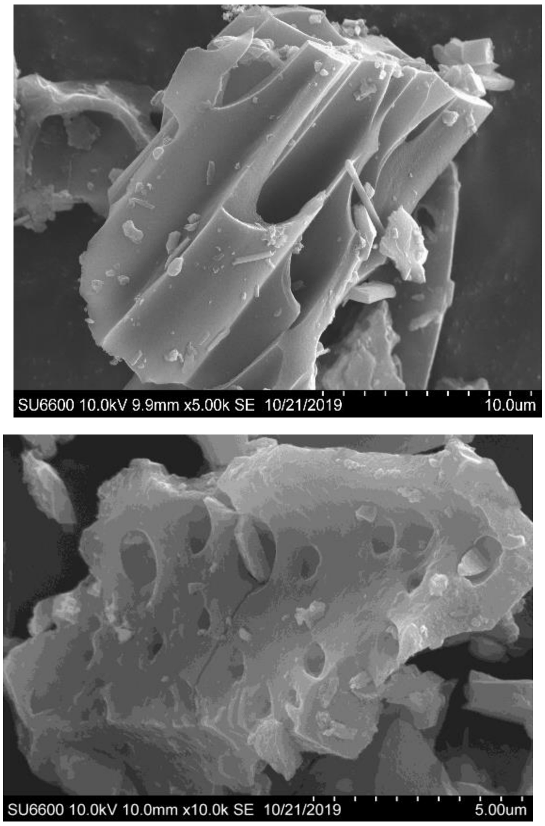

Figure 1 - SEM Images of Gasified Biochar

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Table 2 - CHN Results of Gasified Biochar

	C	H	N	O	H/C	O/C	C/N
Cinnamon	71.27	2.44	0.62	21.11	0.03	0.30	114.95

Furthermore, molar H/C ratio of ≤0.3 indicates highly condensed aromatic ring systems whereas molar H/C ratio of ≥ 0.7 suggests non-condensed structures [11]. Hence, gasified biochar (having molar H/C ratio of 0.03) may contain highly condensed aromatic rings in their structures. Nevertheless, low molar H/C ratio of indicating the higher degree of carbonization in biochar [18].

Moreover, molar ratio values of O/C cinnamon biochar was 0.30. As molar ratio of O/C is indicative of the polarity, biochar may express more hydrophilic [18]. Lower O/C ratios in biochar produced at higher temperature increases its stability in the environment [23]. The raw wood sawdust had relatively high H/C atomic ratio, reflecting low aromaticity compared to that of the biochar samples. The C/N ratio obtained for biochar produced at 450°C and 550°C were greater than 20. This indicated that the wood derived biochar had good potential for net N immobilization in the soil [21].

C. Properties of Clay

The advantages for applying MMT in the uptake of cationic contaminants are obvious, e.g., low-cost, high efficient, and environmentally benign [2,12,21] . Analysis was done using Hitachi SU6600 Analytical Variable Pressure FE-SEM (Field Emission Scanning Electron Microscope) and small amount from the sample was mounted onto the sample stub using carbon tapes and the images were taken at 10kV after gold sputter coating for 15 seconds. The presence of the MMT in clay sample was confirmed from figure 5 and through the plate-like appearance at the cross-section, indicating the flaky structure that are unique for the MMT clay mineral.

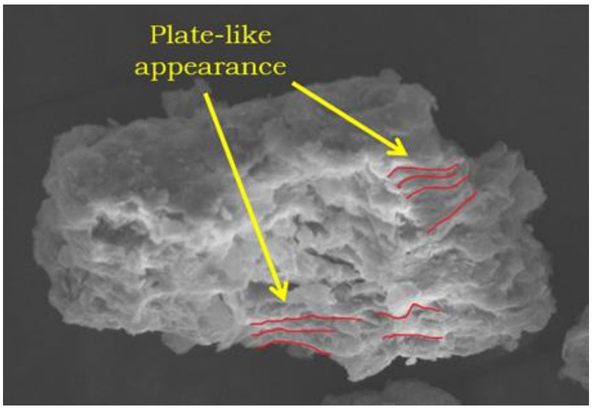

Figure 2 - Scanning Electron Microscopy (SEM) Images of the Clay Sample Obtained From Murunkan Area

FTIR analysis measures a sample's absorbance of infrared light at various wavelengths to determine the material's molecular composition and structure. As shown in the Table 3, Hydroxyl group stretching at 3650 cm-1 and Si-O stretching at 1050 cm-1 were the characteristics of MMT spectra [5]. The peak at ~3620 cm-1 is due to the stretching vibrations of the "inner OH groups" lying between the tetrahedral and octahedral sheets [22] Clay Sample collected from Murunkan gives peck at 3621.66cm^{-1}, Si-O stretching and Al-Al-OH stretching. This reveals that sample contain MMT clay which enhance the removal of contaminants especially heavy metals. These heavy metal cations create bonds (Vander walls, H bond, covalent, ionic bonds) and trapped between tetrahedral and octahedral sheets of MMT in the solute form enable the heavy metal removal. The peak at 1632 cm^{-1} corresponds to the deformation of OH groups of water [4]. The FTIR spectra of clay sample from Murunkan area presented in figure 3. It is composed of two tetrahedral silica sheets sandwiching one octahedral alumina.

sheet in between and contains exchangeable Na+ and Ca2+ cations for structural charge neutrality in its lattice [5]. Inner hydroxyl groups, lying between the tetrahedral and octahedral sheets, give the absorption near 3620 cm^{-1} [22]. The interlayer space between the sheets are occupied and held by those hydrated cations (e.g., Na), stabilizing the clay mineral structure and providing it with a high cation exchange capacity, high surface area, and swelling behavior that enables high absorptivity [22]. Hence, clay sample also having such properties and playing a major role in removing contaminants.

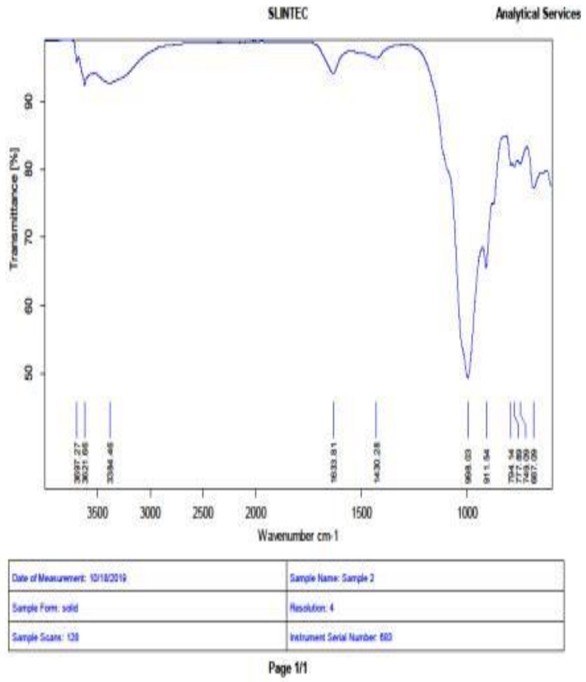

Figure 3 - FTIR Spectra of Clay Sample from Murunkan Area [15]

Table 3 - FTIR Bands and the Corresponding Stretching

Bands (cm^{-1})	Assignments	Remarks
3697.27	O-H stretching (Alcohol)	(Oikonomopoulos *et al.*, 2017)
3621.66	O-H stretching bonded with Al3+ cations	(Ahamed *et al.*, 2018)
3384.46	N-H	
1633.81	O-H bending (Hydration)	(Liang *et al.*, 2017)
1430.28		(Liang *et al.*, 2017)
998.03	Si-O stretching	(Liang *et al.*, 2017)
911.54	Al-Al-OH bending	(Liang *et al.*, 2017)
794.14	Si-O stretching	(Oikonomopoulos *et al.*, 2017)
777.89	C-H	(Oikonomopoulos *et al.*, 2017)
749.09	C-H	(Ahamed *et al.*, 2018)

D. Characterization of Effluent

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 The aerobic zones occur around roots and rhizomes that leak oxygen to the substrate.

Here, turbidity, EC, COD, TDS, BOD 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.

Laboratory wastewaters are small quantity and highly contaminated in nature. 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 (BOD5 in five days at 20°C), 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 BOD5 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 [7].

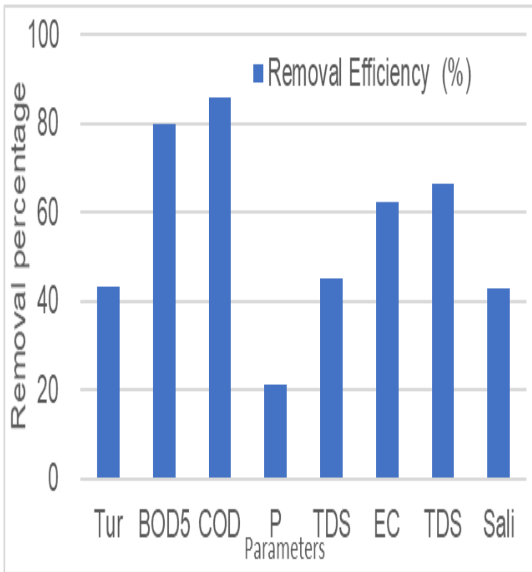

Figure 4: Removal Efficiency of Pollutants

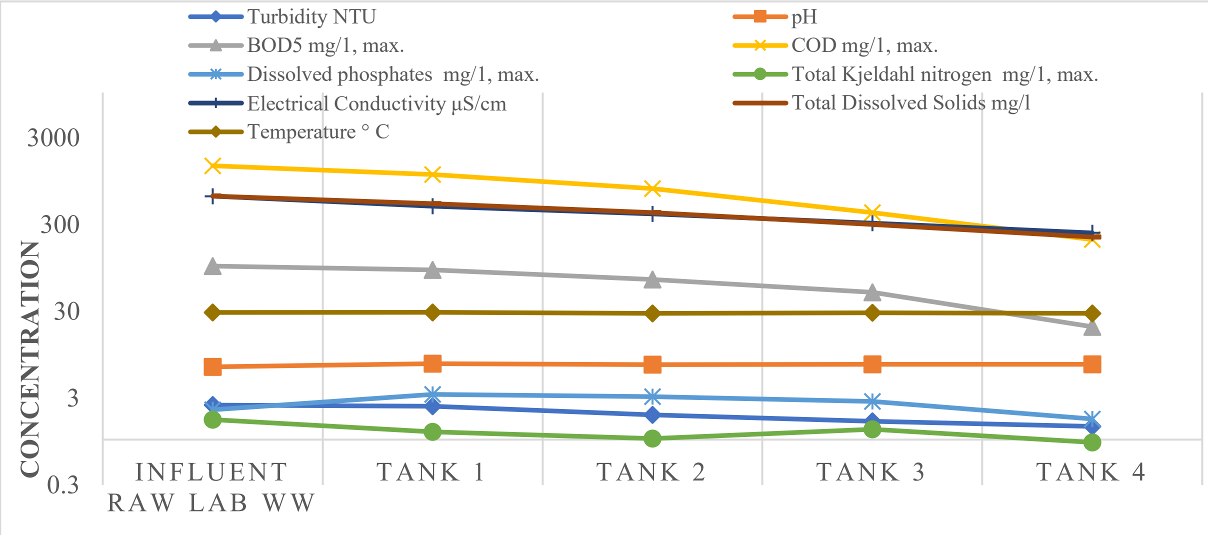

Figure 8: Variation of Physical and Chemical Parameters in the Treatment System

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 89% 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 cinnamon 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 figure 8 and pollutants removal efficiency with parameters illustrated in figure 7.

Higher removal efficiency of 80% BOD and 89% 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 cinnamon biochar.

CONCLUSION

Sending the 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 this gives overall good removal efficiency in laboratory wastewater contaminants. Over the other pollutants, BOD5 and COD results more than 80% of removal efficiency due to the clay: biochar 1:1 composite as a filter media. Plate-like appearance at the cross-section observed in SEM analysis of clay, indicating the flaky structure that are unique for the MMT clay mineral, which enhance the removal of heavy metals from wastewater. Significant drop observed absorbance of infrared light at 998.03cm-1 band due to Si-O stretching may played a key role in removing contaminants by forming bonds with contaminants. Higher reduction in 80% of BOD observed in treatment system may due to the clay bio-film filter media in tank no 4 and 89% of overall COD removal observed may from clay: biochar 1:1 composite.

SEM image of biochar illustrates the smooth surface morphology with porous structure. This helps to retain metals within their pores. H/C molar ration is 0.03, indicates aromatic graphite like structure, which expected to remove heavy metals in the laboratory wastewater. Moreover, it indicates higher degree of carbonization in biochar. Molar ratio of O/C is 0.3 indicative of the polarity; biochar may express more hydrophilic. Thus, clay: biochar 1:1 composite, biochar plays the major role in removing contaminants especially the heavy metals from the laboratory wastewater and reason for 89% of COD reduction in the treatment system.

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