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Preparation, Characterization of activated carbon fiber (ACF) from loofah and its application in composite vertical flow constructed wetlands for Tetracycline removal from water

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Abstract. ACF preparation from different materials and its application in constructed wetlands for wastewater treatment has been focused in environmental field. Different materials have been used to prepare ACF around the world. This study aims to prepare, characterize and use of ACF from loofah for removal of Tetracycline from water through composite vertical flow constructed wetlands. ACF was prepared and it was tested for characterization, later it was used for removal of Tetracycline from water through composite vertical flow constructed wetlands. In composite vertical flow constructed wetlands, three HRTs were set according to the experiment, 1D, 2D, and 3D is individually. Samples were transported immediately from collection point to laboratory for analyzing. Samples were measured for Tetracycline (TC), Total Phosphorus (TP), and Total nitrogen and COD. Tetracycline absorbance with respective 356nm was obtained good and HRT is important factor. Results show that composite vertical flow constructed wetlands with ACF from luffa is best option and it is recommended to study further deep analysis.

Keywords: loofah; characterization of ACF; antibiotic removal; tetracycline; CVFCW; wetland plants

1. Introduction

Personal care products (PPCPs) related to Pharmaceuticals are being used widely and bring environmental issues (Allen *et al.* 2009). In developing countries, wastewater containing several pollutants including pharmaceutical products are released into the environment without treatment. This extensive practice leads to contamination of bodies of water (UNWWDR 2017). Indeed, there are many methods to resolve this issue but have been observed to be unable to totally remove PPCPs. One of the most worrying groups of substances is that of antibiotics because hospital and municipal wastewater treatment plants cannot remove antibiotics and pharmaceuticals products from the water (Hughes *et al.* 2013, Joss *et al.* 2006, Akram, M *et al.* 2020). Antibiotic issue remained the most important challenge around the globe. In developing countries, impact of antibiotic is extremely serious due to misuse, overuse, and damping of antibiotics (Adam *et al.* 2016). Several studies have been conducted, specially ACF from different materials have given good results. ACF is added in constructed wetlands (CWs) which are best alternative system for wastewater treatment, with relatively low maintenance costs and consuming little energy. CWs are only feasible in small urban communities, this technology, with addition of ACF has been observed to be useful. Removal efficiency is more than other conventional WWTPs (Sanjrani 2019). Nevertheless, their efficiency to specifically remove antibiotics is still unknown (Conkle *et al.* 2010), but several studies have documented high removal efficiency of Antibiotics i.e sulfonamides, quinolones, macrolides, tetracyclines, and lactams range from -78:4% to 100.0%, 13.5% to 100.0%, -25:8% to 100.0%, 47.0% to 97.0%, and 6.0% to 45.0%, with mean values of 83.7%, 29.2%, 70.1%, 15.9%, and 51.1%, respectively (Yan *et al.* 2015, Chen *et al.* 2015, Hsieh *et al.* 2015). The aim of this study was to remove antibiotic products through ACF added in Composite Vertical Flow Constructed Wetlands. First water containing Tetracycline (TC) and other things such as Total Nitrogen (TN) and Total Phosphorous (TP) were stored in the tank, further it is processed in constructed wetlands. Constructed wetlands system consisted two plants Calamus and Chives. ACF (made from loofah) was also added in the system to improve the efficiency for the removal percentage. Results from previous researches show that the pollutants have been removed at considerable level (Sanjrani *et al.* 2020a). The hydraulic retention time (HRT) was manually controlled by valves for 3 days, 2 days and 1 day respectively. Therefore, ACF from loofah added in Composite Vertical Flow Constructed Wetlands is recommended for removal of antibiotics Tetracycline (TC), Total Nitrogen (TN) and Total Phosphorous (TP) from water.

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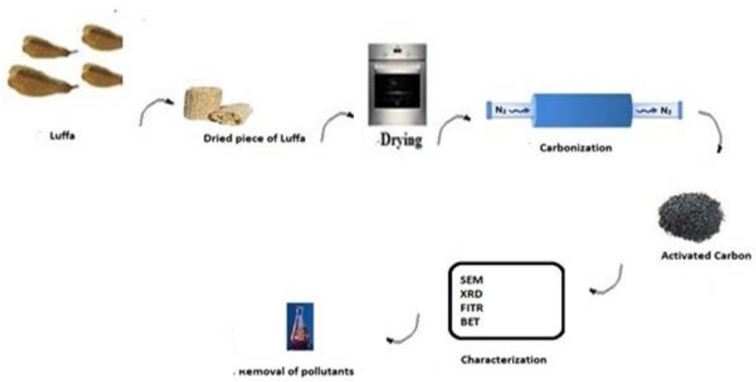

Fig. 1 ACF Preparation processes

2. Materials and methods

Activated carbon Fiber: The ACF used in this work was obtained from material loofah prepared and then characterized. Material was produced by Henan luohe hua hui co LTD. Preparation of ACF was conducted in lab with proper procedure (See Fig 1). Firstly, the loofah fiber was impregnated with a water-soluble phenolic resin for 24 hours, and dried. Later it is put into a high temperature resistance furnace after loofah fiber is baked in an oven (105°C) for 1-2 h for pre-oxidation, carbonization, activation treatment where activation temperature is 550~850°C. Finally, the obtained product was immersed in a 1 M hydrochloric acid solution for 2 h to remove impurities such as ash from the product. The product loofah activated carbon fiber is obtained.

Characterization: Scanning Electronic Microscopy (SEM), Transmission Electron Microscope (TEM), Fourier Transform Infrared Spectroscopy (FITR) and Brunauer Emmet Teller (BET) were conducted for the characterization. Results proved that ACF is better for treating pollutants. The experiments were performed after successful acclimatization and establishment of the plants and microbial community.

Constructed wetlands: Experimental set up was mainly composed of a composite vertical flow constructed wetlands unit with two types of plants Chalamus and Chives and ACF from luffa for treatment of TN, TP, COD and Tetracycline. Constructed Wetland was properly prepared, shown in Schematic diagram is shown in Fig.2

Plants: There are two types of plants, Acorus calamus and **Allium** tuberosum (also known as Garlic chives) planted in the system. Experimental work was started after plants grew well

3. Characteristics of activated carbon fiber

ACF can play a vital role for phosphorus removal. A scanning electron microscope (SEM) was used to measure surface morphology.Figure 2-4 shows the TEM and SEM morphologies. The ACF surface is expected to be positively charged in most natural aqueous conditions and electrostatically attract the negatively charged phosphate species. Moreover, several studies also have documented that ACF significantly affect to retain nitrogen (Ding *et al.* 2010). SEM and TEM results show that ACF is effective. After characterization, it was added in the system (composite vertical flow constructed wetlands) for antibiotic (tetracycline) removal from water.

BET summary report about surface area, pore volume and pore size. Results about surface area show that

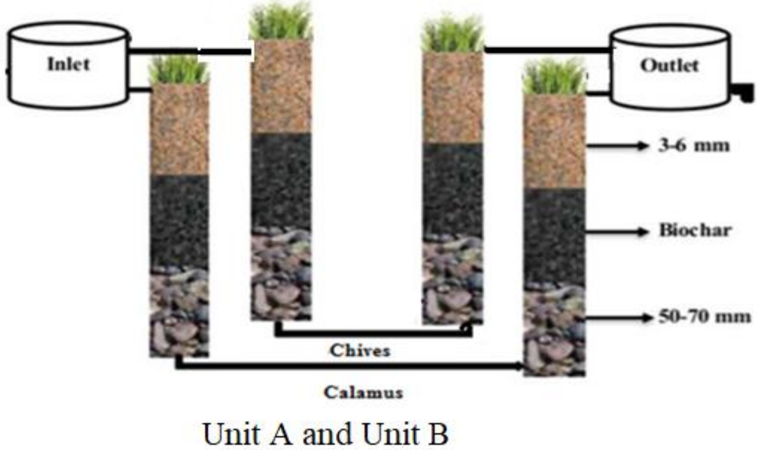

Fig. 2 Schematic diagram Composite Vertical Flow Constructed Wetlands

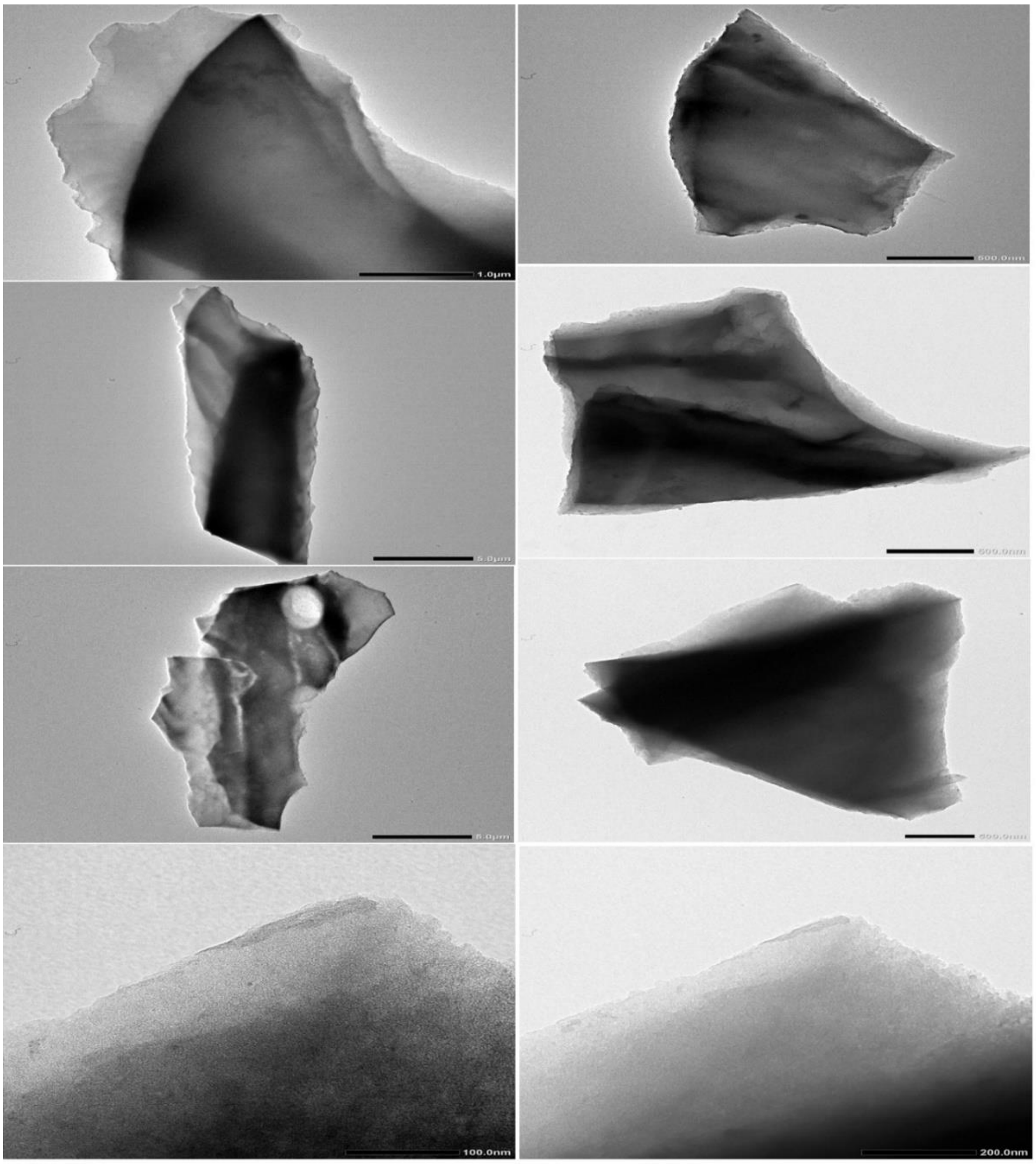

Fig. 3 TEM results of ACF

single point surface area at P/Po = 0.120789262: 1484.8788 m^2/g, BET Surface Area: 1440.3374 m^2/g, t-Plot micropore prea: 1255.6370 m^2/g, t-Plot external surface area: 184.7004 m^2/g, BJH Adsorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter: 33.127 m^2/g and BJH desorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter: 177.8197 m^2/g. BET results about pore volume is like this, single point adsorption total pore volume of pores less than 413.0665 nm diameter at P/Po = 0.995314738: 0.637063 cm^3/g, t-Plot micropore volume: 0.528401 cm^3/g, BJH adsorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter: 0.035674 cm^3/g, and BJH desorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter: 0.110151 cm^3/g. Pore Size results are given as, adsorption average pore width (4V/A by BET): 1.76920 nm, BJH adsorption average pore diameter (4V/A): 4.3076 nm and BJH desorption average pore diameter (4V/A): 2.4778 nm.

Fourier infrared spectroscopy (FITR) was also conducted to know more about ACF properties (see fig 5). After preparation and characterization of ACF, it was used in composite vertical flow constructed wetlands for antibiotics removal. Results show that antibiotic has been removed at considerable level, as ACF from luffa has shown promising results in several studies (Sanjrani *et al.* 2020b).

4. Operational procedure and sampling

Water containing Antibiotic Tetracycline (TC) was piped into the cells for various complex physical, chemical and biological processes. Wetland unit A and B are prepared for the experimental study. In each wetland unit, it has two cells connected to each other (with down-up flow) as a composite vertical flow constructed wetland system. Two different types of vegetation are planted in each wetland unit, for example two cells with calamus in Wetland unit A, and two cells with plant Chives in Wetland unit B. ACF was added to the system in the middle for efficient removal (See fig 1-2).

4.1 Physico-chemical analysis:

Intermittent water intake was used in the experiment. The hydraulic retention time was manually controlled by valves for 3 days, 2 days and 1 day. The antibiotic wastewater gradient ranged from low to high. Each kind of wastewater was operated for half a month. The samples were sampled under three different hydraulic retention times, and the weather light intensity on the day of sampling was recorded. Three HRTs were set according to the experiment, 1D, 2D, and 3D is individually. Samples are transported immediately from collection point to laboratory for analyzing. Samples were measured for Tetracycline (TC), Total Phosphorus (TP), and Total nitrogen, following the procedure of national standard monitoring methods. The total nitrogen is determined by potassium persulfate digestion spectrophotometry, the total phosphorus is determined by ammonium molybdate spectrophotometry, and TC were determined by UV spectrophotometer method, after wavelength scanning, the optimal absorption wavelength of tetracycline is 356 nm.

4.2 Influent water quality

In order to prevent the influence of the chroma of tetracycline on the experimental results, a comparative experiment was carried out under the condition of ultraviolet spectrophotometry. Two beakers A and B with quantity 1L were taken and were added with the same amount of nitrogen and phosphorus sources. The latter B was added with 1 mg tetracycline hydrochloride to prepare a 1L solution. TP and TN were measured in two kinds of solutions, and three groups of parallel samples were taken for each solution. For TP and TN, different amount of tetracycline was added, see table 1-2. Tetracycline wastewater was prepared in 50L, and the concentration of tetracycline was 100ug/L. Hydraulic retention time HRT=3d, flow rate v=5.8ml/min.

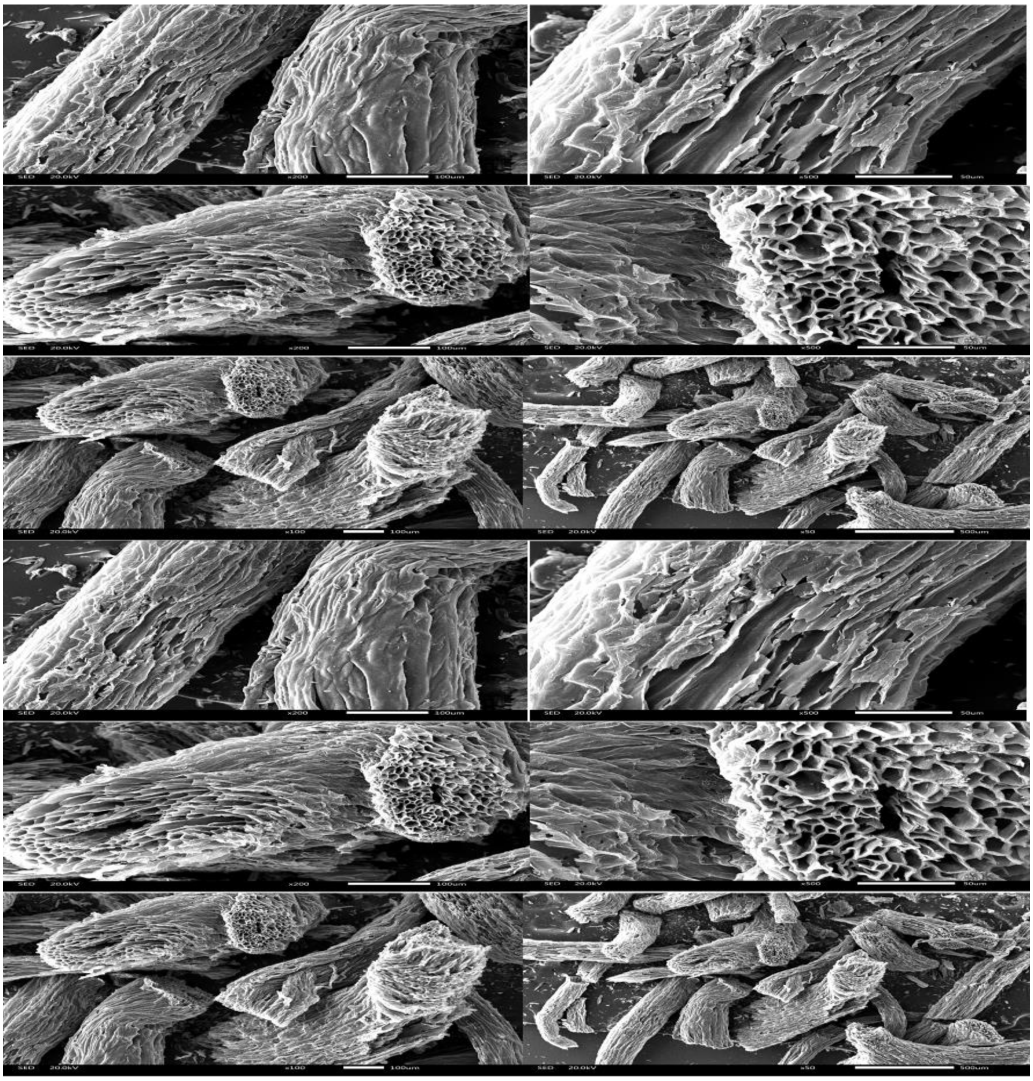

Fig. 4 SEM results of AC

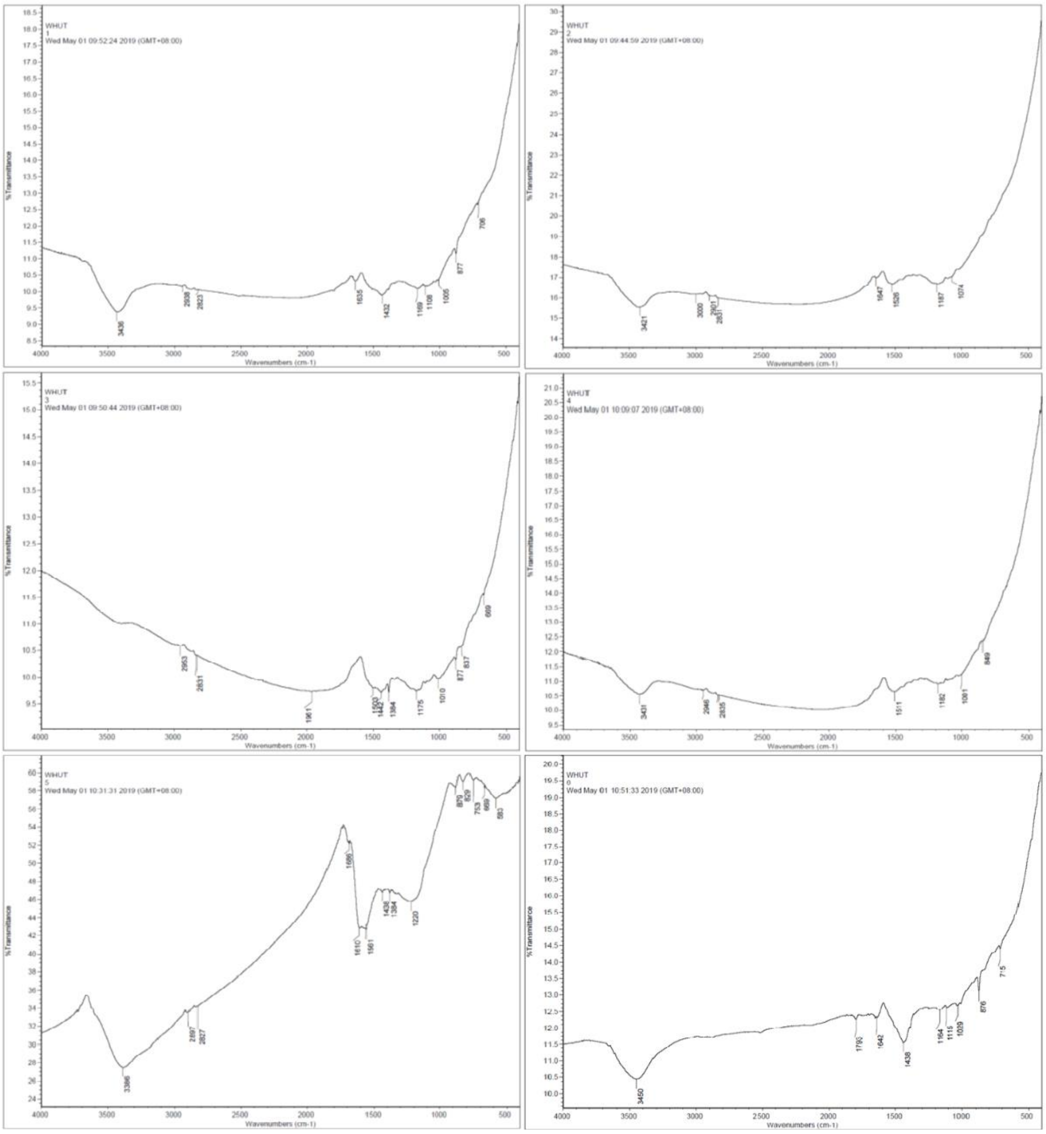

Fig. 5 Fourier infrared spectroscopy (FITR)

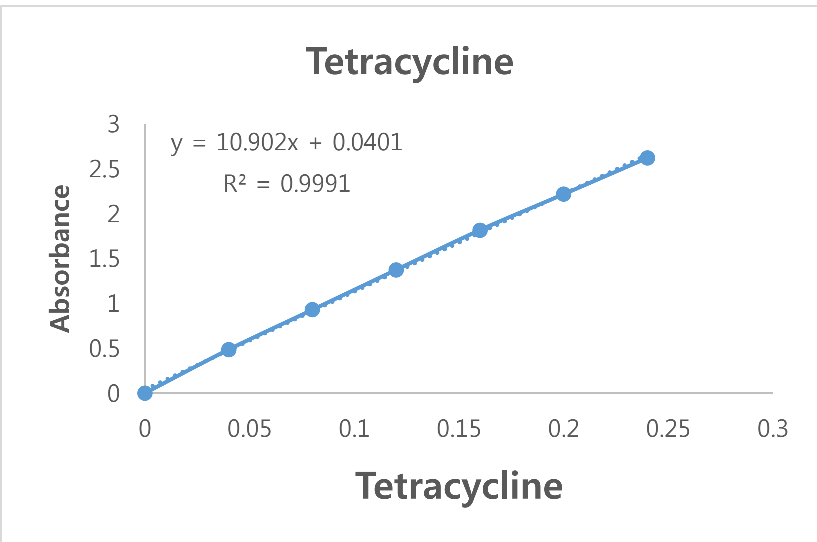

Fig. 6 Tetracycline (TC) results

Table 1 TP (Influent water quality)

Number	1	2	3	4	5	6
	without tetracycline			added with tetracycline		
Absorbance	0.426	0.439	0．443	0.434	0.407	0.408
mean value	0.436			0.416		
Concentration (ug/ml)	46.48			44.35		

Table 2 TN (Influent water quality)

Number	1	2	3	4
	Conventional (regular)		Added with tetracycline	
Absorbance	1.837	1.868	1.803	1.873
mean value	1.852		1.838	
Concentration (ug)	178.5		177.5	

5. Results and discussion

5.1 Plant development and analysis

The plant growth and number of shoots were monitored during operation. In relation to the height of the plants, small differences were registered along the systems operation. In general, plants developed and proliferated without showing signs of toxicity. The macrophytes growing in the system are essential components because they have several properties in relation to the treatment processes (Calheiros *et al.* 2007). In the system (A and B) showed positive growth, height increased in 3 months without any obvious symptoms of toxicity or nutrient deficiency. Root and rhizome growth accounted to be grown. The biomass growth was varied among the wetland units and no significant effect was observed with respect to ACF addition on plant growth. Indeed, ACF was found more efficient to be added in this system.

5.2 Constructed wetlands performance

In Tables 3–5, the results of the physico-chemical analysis corresponding to the inlet and outlet of TN, TP and TC. sampling 150ml at a time. Samples are taken about once a week. A month's sample. (All are the average of parallel samples). All samples were collected in the morning between 10:00 and 11:00 and were taken directly to the laboratory. Three HRTs were set according to the experiment, 1D, 2D, and 3D is individually. Analysis of water samples for quality in the CVFCW showed that the physicochemical quality of the treated water increased; a decrease in TC, TN and TP concentrations was obtained from inlet to outlet of the CVFCW in both planted constructed wetlands unit. According to lab results, the removal effect of Tetracycline (TC), Total Phosphorus (TP), and Total nitrogen under different HRT and in different wetlands (A and B) has shown different results. Tetracycline absorbance with respective 356nm was obtained good, as y= 10.902x + 0.0401 while R^2 = 0.9991. The results is given below in the table 3 and Fig 6.

5.3 Total nitrogen *removal*

The mechanisms (volatilization, nitrification/denitrification, ammonification, plant uptake, and matrix adsorption) play vital role in the nitrogen removal in CWs (Shi-Kuan Jiang *et al.* 2017, Vyamzal 2007). Total nitrogen absorbance with respective 220nm and 275nm in different wetlands unit has shown good results as and y= 0.0098x + 0.0125 and R^2 = 0.9992 related results are given in fig 7. Moreover, several studies also have documented that ACF significantly affect to retain nitrogen (Ding *et al.* 2010). In this study, effect of vegetation was observed on nutrient removal among all the three wetlands (A and B)

Wetlands (A and B) containing Calamus and Chives plants have removed higher percentage of TN. Moreover, microbial degradation process is also responsible for removal mechanisms, which also requires microbial activity and longer retention time. Furthermore, there were significant differences in total nitrogen removal rates under different HRT.

Table 3 Tetracycline (TC) with respective 356nm

Contents (mg)	356nm
0	0
0.04	0.485
0.08	0.928
0.12	1.373
0.16	1.816
0.2	2.216
0.24	2.62

Table 4 Results of TN

TN	220 nm				275 nm			
	Waste water	Effluent	wetlands unit A with plants Acorus calamus	wetlands unit B with plants Chives (leek)	Waste water	Effluent	wetlands unit A with plants Acorus calamus	wetland unit B with plants Chives (leek)
1	0.544	0.322	0.352	0.322	0.011	0.031	0.016	0.017
2	0.504	0.312	0.342	0.332	0.013	0.021	0.017	0.016
3	0.553	0.322	0.332	0.312	0.016	0.017	0.016	0.013
4	0.484	0.302	0.332	0.338	0.013	0.021	0.014	0.014
5	0.486	0.312	0.342	0.338	0.012	0.019	0.014	0.011

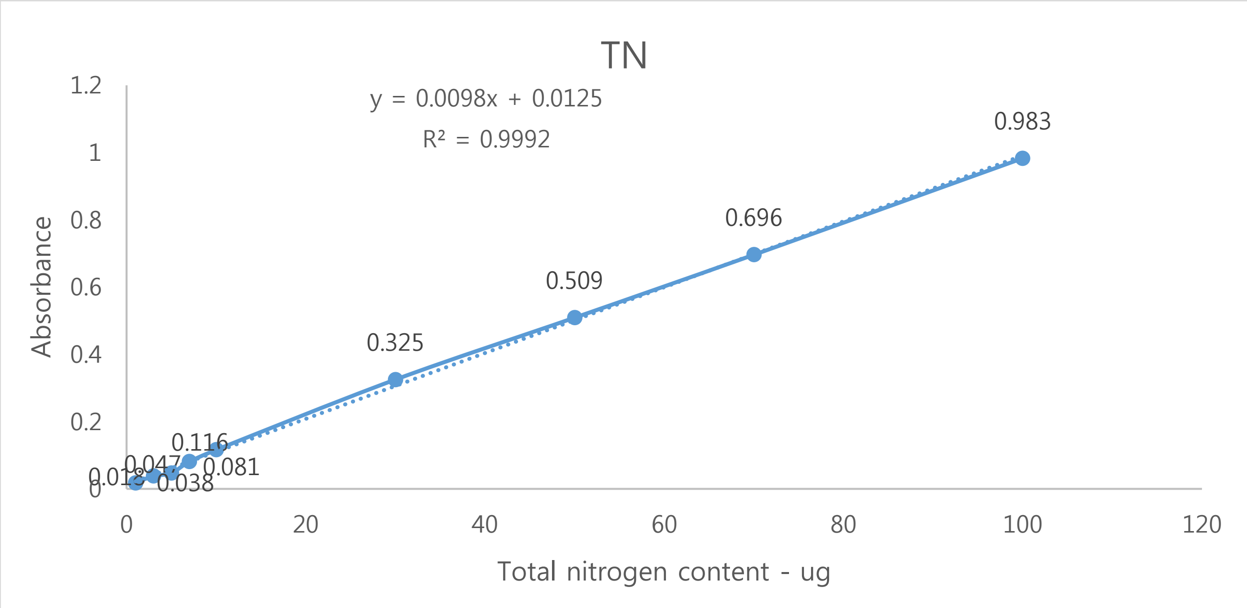

Fig. 7 TN Absorbance Results

Table 5 Absorbance Results for TN

S.No	ml	ug	220nm	275nm	Absorbance Result	
1	0	0	0.102	0.012	0.078	
2	0.1	1	0.104	0.004	0.096	0.018
3	0.3	3	0.124	0.004	0.116	0.038
4	0.5	5	0.145	0.01	0.125	0.047
5	0.7	7	0.169	0.005	0.159	0.081
6	1	10	0.202	0.004	0.194	0.116
7	3	30	0.407	0.002	0.403	0.325
8	5	50	0.603	0.008	0.587	0.509
9	7	70	0.796	0.011	0.774	0.696
10	10	100	1.063	0.001	1.061	0.983

5.4 Total phosphorus removal

The physiochemical and hydrological properties of the filter material are associated with removal of phosphorus, whereas phosphate is mainly precipitated or adsorbed in filter media. In broad meaning, the removal of phosphorus depends on the physical adsorption of the matrix, the chemical deposition, microbial degradation and plant adsorption. (Shi-Kuan Jiang *et al.* 2017, SUN *et al.* 2014, Braskerud 2002).

In addition, ACF can play a vital role for phosphorus removal, the ACF surface is expected to be positively charged in most natural aqueous conditions and electrostatically attract the negatively charged phosphate species. The assimilation by microorganisms and plants mainly supports the phosphorus removal in CWs (Huett *et al.* 2005). Moreover, in this study, the vegetation played an important role as they provide temporary storage of phosphorus. The Plant Calamus and Chives demonstrated good results for Phosphorus removal in wetland system. Contact area between plants and influent water is increased

Table 6 Absorbance result for TP

TP	700 nm (Absorption wavelength)			
	Waste water	Effluent	wetlands unit A with plants Acorus calamus	wetlands unit B with plants Chives (leek)
1	0.095	0.034	0.056	0.048
2	0.090	0.033	0.050	0.045
3	0.087	0.030	0.052	0.048
4	0.089	0.035	0.049	0.047
5	0.088	0.032	0.048	0.047

Table 7 Result for TP

Concentration	Group number	Absorbance 1	Absorbance 2	Average
0	1	0.023	0.031	0.027
1	2	0.026	0.033	0.0295
2	3	0.032	0.039	0.0355
6	4	0.077	0.069	0.073
10	5	0.108	0.115	0.1115
20	6	0.206	0.21	0.208
30	7	0.298	0.31	0.304
G Blank	1st group	2nd group	Average	
1	0.098	0.179	0.1385	
2	0.009	0.279	0.144	
6	0.054	-0.031	0.0115	
10	0.085	-0.031	0.027	
20	0.183	-0.031	0.076	
30	0.275	-0.031	0.122	

by the developed root system of calamus and also it brings aerobic, anoxic and anaerobic conditions in the wetland bed, plant roots and surrounding environment. It provides attachment sites for microorganisms. Under aerobic conditions, phosphate is taken up from water because microorganisms use oxygen as an electron acceptor to generate energy by PHB decomposition.

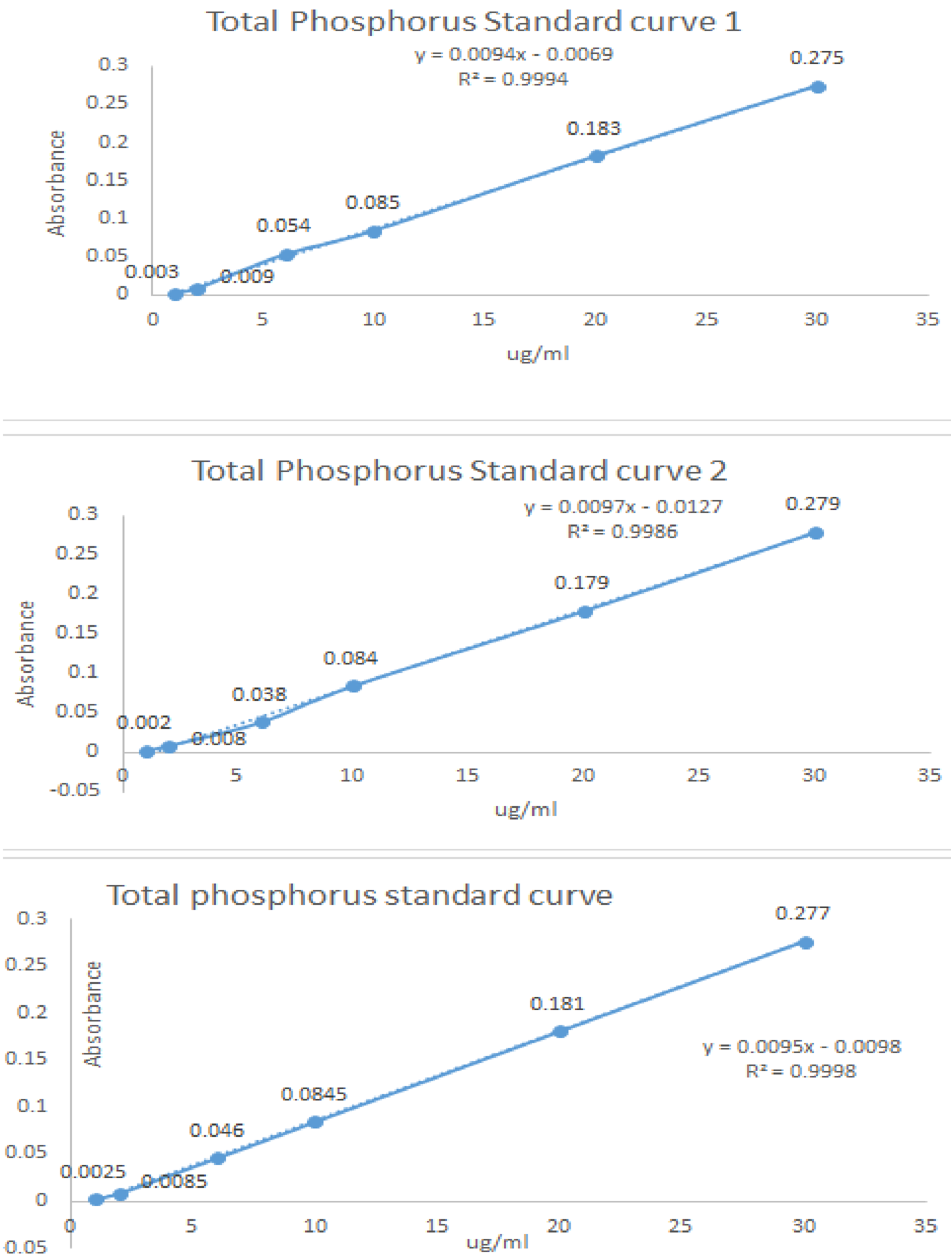

Fig. 8 TP Absorbance curve

Table 8 COD Results

COD value	Absorbance
0	0.0355
200	0.1178
500	0.2553
800	0.3784
1000	0.4662

5.5 COD removal

COD removal with constructed wetland has been documented by many researchers. Here is the result of COD absorbance in Table 8 and fig. 9.

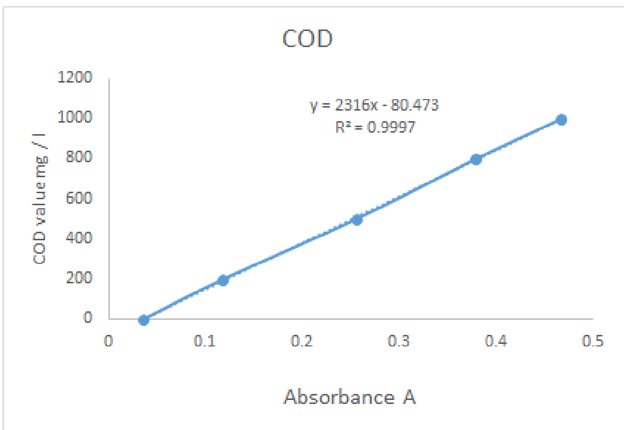

Fig. 9 COD Absorbance Results

6. Conclusions

It is concluded that based on characterization and the results from Composite Vertical Flow Constructed Wetlands for antibiotic treatment has been found efficient. This study observed that wetlands with ACF and selected plants were more efficient as compared to the wetland with gravels alone in the removal of various organic and inorganic pollutants, it is also proved in our previous studies (Sanjrani Manzoor Ahmed *et al.* 2020 a, Sanjrani Manzoor Ahmed *et al.* 2020 b). So, it is recommended that a Composite Vertical Flow Constructed Wetlands for rainwater treatment is a good option. However, this study is preliminary, there is still gap; it is acknowledged that further studies with rigorous methodologies are needed before conclusions can be drawn. such as modification of adsorbent to enhancement adsorption capacity, uilization of composite adsorbents, multi-component adsorption, treatment of real effluents, fixed-bed studies, and enhancement of regeneration. Furthermore, studies should also be extended to pilot and full scale to evaluate the potential use of ACF at the industrial level. In general, plant calamus is efficient and the use of ACF in wetlands is best choice.

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