Optimisation and performance evaluation of a semi-pilot scale iHydroMET for decentralised domestic wastewater treatment

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A dissertation submitted for the partial fulfilment of BS-MS dual degree in Science

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Certificate of Examination

This is to certify that the dissertation titled "Optimisation and performance evaluation of a semi-pilot scale iHydroMET for decentralised domestic wastewater treatment" submitted by Mr. Siddhant Sahoo (Reg. No. MS16138) for the partial fulfilment of BS-MS dual degree programme of the Institute, has been examined by the thesis committee duly appointed by the Institute. The committee finds the work done by the candidate satisfactory and recommends that the report be accepted.

Dr. Santosh B. Satbhai   Dr. Baerbel Sinha   Dr. Sunil A. Patil

(Supervisor)

Dated: May 21, 2021

Declaration

The work presented in this dissertation has been carried out by me under the guidance of Dr. Sunil A. Patil at the Indian Institute of Science Education and Research Mohali.

This work has not been submitted in part or in full for a degree, a diploma, or a fellowship to any other university or institute. Whenever contributions of others are involved, every effort is made to indicate this clearly, with due acknowledgement of collaborative research and discussions. This thesis is a bonafide record of original work done by me and all sources listed within have been detailed in the bibliography.

Siddhant Sahoo
(Candidate)

Dated: May 21, 2021

In my capacity as the supervisor of the candidate's project work, I certify that the above statements by the candidate are true to the best of my knowledge.

Dr. Sunil A. Patil
(Supervisor)

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List of Figures

List of Photographs

List of Tables

Abbreviations

BM	Bed Matrix
BOD	Biological Oxygen Demand
CCV	Closed Circuit Voltage
COD	Chemical Oxygen Demand
CP	Cocopeat
CWs	Constructed Wetlands
DS	Domestic Sewage
GAC	Granular Activated Charcoal
HRT	Hydraulic Retention Time
MFC	Microbial Fuel Cell
MLD	Million Liters Per Day
MPN	Most Probable Number
N	Nitrogen
P	Phosphorus
PAOs	Phosphate Accumulating Organisms
PPCPs	Pharmaceuticals and Personal Care Products
QS	Quartz Sand
SDGs	Sustainable Development Goals
STP	Sewage Treatment Plants
TN	Total Nitrogen
TP	Total Phosphorus
UV	Ultraviolet

Contents

Abstract

In recent decades, high socio-economic development has led to an increase in water consumption, which, in turn, generates large volumes of wastewater. The centralized wastewater treatment facilities are not foreseen as a sustainable solution to manage the water and wastewater resources due to associated issues like high capital investments, high maintenance costs, upgradation challenges, and operational difficulties in scarcely populated or unsuitable terrain regions. Nowadays, wastewater is increasingly regarded as the non-conventional water source to meet increasing water demands for different purposes. The processes used in the treatment plants determine the quality of treated effluent. In recent times, decentralised treatment processes capable of resource recovery are highly encouraged over the globe. In particular, integrated technologies that can help manage the wastewater at the point sources are emerging as promising alternatives to the existing approaches. In this context, our lab tested the idea of iHydroMET with the aim of developing sustainable decentralised technology. iHydroMET stands for Integrated Hydroponics Microbial Electrochemical Technology. It is based on the integration of physicochemical, biological, and bioelectrochemical processes in a single reactor system to remove different pollutants present in domestic wastewater. In a proof-of-concept design study, it worked in principle and proved its potential for wastewater treatment along with simultaneous resource recovery. As a logical continuation of the work, my thesis focussed mainly on optimising the reactor components of iHydroMET to improve the system performance.

We tested three major components; namely, i) support bed matrix, ii) cathode placement and iii) plant selection for better nutrient removal. Different granular activated charcoal (GAC) and cocopeat (CP) ratios were tested as the bed matrix component. GAC:CP in 20:80 combination showed removal of around 70% of COD, 69% ammonia, and 80% orthophosphate. The configuration of ¾ immersed cathode in effluent showed higher closed circuit voltage (by a margin of 34±4 mV) compared to the cathode placement at the top of the bed matrix. Among different locally available plants, namely Vinca, Pothos, Peppermint, Lemongrass, and Brahmi, the Vinca plant outcomepted others in nutrient removal capabilities (total nitrogen; TN: 44 mg/L and total phosphorous; TP: 2.1 mg/L

within 48h). It offers additional benefits like perennial flowering and phytoremediation of heavy metals.

The semi-pilot scale iHydroMET with the selected reactor unit components was then evaluated for domestic wastewater treatment and resource recovery. The upgraded system achieved 79±6.7 %, 20±8.4 %, 10±2.7 % removal efficiencies for COD, TP, and TN after 3h of operation in the fed-batch tests. The removal efficiencies enhanced to 91±3.3 %, 32±1.1 %, 34±5.7 % after 24h. The maximum power densities were recorded at low levels of 1.2 mW/m^2 in series and 11.7 mW/m^2 in parallel connections. The UV treatment of the secondary effluent of iHydroMET resulted in complete disinfection. As per the standards of ‘The Food and Agriculture Organization, United Nations’, the treated effluent quality is fit for reuse in gardening purposes.

To summarize, the selected reactor components contributed to the improved COD removal performance by iHydroMET. However, the system could not substantially remove TN and TP and produced low electric power. It was most likely due to the lack of anoxic conditions in the lower part of the reactors. Hence, further modifications in system operation like creating a saturation zone in the lower part of the reactor units are needed to address the inefficient removal of nutrients from domestic wastewater and improve the electric output of the iHydroMET technology.

Chapter 1

Introduction

1.1 Wastewater: An environmental problem but promising solution to address water stress

Water is an essential commodity of life. All types of life formsdepend on water for survival as almost all cell functions require water directly or indirectly. Besides biological importance, water is used for a wide variety of daily activities like washing, cooking, industrial application, agriculture, etc., due to its unique chemical and physical properties. Due to rapid socio-economical development over the recent decades, water is used extensively and unethically by humans, resulting in an imbalance in the water use cycle. It has led this crucial resource, i.e., freshwater, on the verge of scarcity. One-quarter of the world's population in developing and poorly developed countries is affected by moderate to high water stress (Sands, 2020). The global population boom and urbanization expansion over the last decades also created a challenge of managing huge quantities of generated wastewater. The domestic wastewater streams, agricultural runoffs, and industrial discharges contribute massively to freshwater pollution, making freshwater unfit for direct human and livestock usage. In water-stressed countries like India, freshwater supply is projected to become an alarming issue with time. According to a 2018 Niti Aayog report, water availability is set to decline from 1,544 cubic meters per capita in 2011 (and 1,816 m^3 in 2001) to 1,465 cubic meters by 2025 (NITI Aayog 2018). According to the Central Pollution Control Board, India will need 1.5 trillion cubic meters of water by 2030 (Researchers Devise New Wastewater Recycling System, India Science Wire, 2018). Hence, there is a need to manage wastewater efficiently and exploring unconventional water sources to meet the increasing freshwater demand.

For a very long time, wastewater has been considered an environmental hazard and health concern. However, in recent years, the change in this attitude can be noticed. Now, the non-conventional sources of water, like properly treated wastewaters, are being seen as a potential source to meet future water demands. Transitioning of wastewater treatment plants into resource recovery facilities is considered a promising way to achieve resource sustainability (Wang et al., 2015). The trend is changing speedily towards maximizing resource recovery from wastewaters, mainly in the developed countries. However, in developing countries like India, the current focus is to balance the equation of wastewater produced and treated by increasing the treatment capacity. Also, strict norms and guidelines are being imposed to make existing wastewater treatment systems more efficient to produce treated effluents of desirable discharge quality and prevent pollution in aquatic freshwater bodies (Schellenberg et al., 2020). The treated wastewater can be reused to provide ecological benefits such as irrigation, flushing, and washing which ultimately reduces the demand for freshwater and augment the existing water supplies (Mo & Zhang, 2013).

1.2 Existing wastewater treatment infrastructure

Globally, more than 80% of wastewater gets discharged to various natural water bodies in the environment without much treatment or no treatment at all. In rich countries, around 70% of the wastewater gets treated, whereas, in low-income countries, the treatment is just 8% of the total wastewater generated (Wastewater, The Untapped Resource, United Nations Educational, Scientific and Cultural Organization, 2017). In India, considering the available infrastructure, existing wastewater treatment technology, and technical support, only around 34.74% of 75,020 MLD (million liters per day; data for 2018) wastewater generated could be treated in class-II towns (www.indianinfrastructure.com). The municipal wastewater generation load is so high that a high percentage (65%) of the wastewater gets discharged to surface water bodies without proper treatment causing significant pollution. Most of the existing wastewater treatment infrastructure is centralized across the globe. However, the centralized large conventional sewage treatment plants are challenged by changing climate conditions and cannot sustain the current and growing demand (Mercer, 2014). In addition, the aging wastewater collection networks suffer due to several reasons such as costs intensive maintenance and repair and the associated harmful environmental impacts due to wastewater leakage from pipes (Electricity Use and Management in the Municipal Water Supply and Wastewater Industries, 2014). Moreover, most plants are difficult to upgrade with the new and resource recovery capable technologies. Thus, there should be more emphasis on finding alternative technologies that do not burden the environment and economy.

Initial progress in this sector has been mostly made through various government and private investments to meet Sustainable Development Goals (SDGs). It has created an opportunity for alternative approaches in cities and towns where centralized wastewater treatment systems are either absent or not efficient to accomplish the level of performance (Water, Sanitation & Hygiene, Bill & Melinda Gates Foundation, 2020). However, as the water-related sector is operated by conservative institutions that require decades to respond to the development of new technologies, there are fewer chances for an instant shift (Kiparsky et al., 2013). Whenever engineers propose novel ideas for making centralized treatment systems more efficient, the authorities tend to focus mostly on incremental improvements in the existing system rather than implementing new effectual technologies because of the possibility of incurring misfortune or failure (Rabaey et al., 2020).

1.3 The era of decentralization

As the existing centralized conventional approach has a limited network, inadequate functioning, and has cost restrictions to build and operate, we should not rely on them as the only way to manage wastewaters. Hence, alternative approaches that do not rely upon centralized sewer systems are gaining attention (Rabaey et al., 2020). Decentralised wastewater management approaches are considered a sustainable and cost-effective alternative as these systems treat and discharge or offer reuse of the effluent at the point sources. They have been promoted because of their low operational and maintenance requirements and small-scale investments. Their implementation also makes it feasible to treat wastewater from those areas that are not connected to centralized wastewater management networks. Such areas are sometimes scarcely populated and located at complex terrains; it is difficult to sustain a centralized sewage network. The major advantage a decentralised wastewater treatment system offers is the ability of the consumer to control the quality of water delivered according to its usage (Rabaey et al., 2020). For example, if one needs treated water for gardening only, it does not require additional nutrients removal steps. The commercialization of these technologies has been started. For instance, Hydraloop turnkey systems are already on the market that enable greywater treatment for reuse as flushing water or washing purposes (Innovative Water Recycling System, Hydraloop, International, 2020). Additionally, many decentralised technologies are under investigation in different countries for managing wastewater at point sources.

1.4 Emerging decentralised technologies for wastewater treatment

Considerable efforts are going on developing technologies that are cost-effective, easy to implement, and capable of treating a sufficient amount of wastewater to meet the load at household or community levels. Developing a standalone new technology does not seem feasible in the near future, so more focus is drawn on technologies that are based on the integration of different types of existing processes or technologies. It allows using several advantages like a high treatment rate system capable of resource recovery and overcoming the limitations of the individual treatment processes. In recent times, most of the developments revolve around technologies that are ecologically engineered to mimic nature’s ways of managing wastewaters. Generally, an engineered system consists of several different tanks interconnected in series through which wastewater flows. The wastewater is passed through diverse biota like aquatic macrophytes, aquatic plants, and filter feeders for wastewater treatment through ecological interactions and purification (Chiranjeevi et al., 2013). Constructed wetlands (CWs) is one such technology that offers promising solutions for wastewater treatment and water management. They are based on phytoremediation principles. In the last decade, integration of CWs with microbial fuel cells (MFC) has become a promising approach (Yadav et al., 2012). MFCs are an emerging bio-electrochemical technology for simultaneous wastewater treatment and electricity production. In recent times, further advancement in CWs has been done by replacing the non-conductive bed matrix with conductive materials. It is termed electro-wetlands (Ramírez-Vargas et al., 2018). These technologies seem to be complex but are easy to implement and manage in the real fields and consume low energy. In all cases, the main challenge is to make technology proficient and sustainable in recovering and reusing resources like water, nutrients, and energy from the wastewater.

1.5 The idea of iHydroMET

With an aim to develop a sustainable and economically feasible solution for wastewater management at the households, the integrated drip hydroponics-MFC system design was tested for domestic wastewater treatment in our lab (Yadav et al., 2020). We refer to the technology as iHyroMET. It consisted of a drip manifold system for influent feeding and effluent collecting duct along with ten reactor units. Each unit housed three key reactor components: the cocopeat bed matrix, a graphite electrode assembly, and a lemongrass sapling. After 3h operation in a batch recirculation mode, the system achieved 72 ± 2.4% COD, 83 ± 1.1% phosphate, and 35 ± 4.2% ammonia removal efficiencies. It also yielded low levels of power output and plant biomass. The arrangement of different components reactor units facilitated various physicochemical, biological, and bio-electrochemical processes in different microenvironments, resulting in removing different pollutants in the integrated system. Overall, this low-cost and simple integration worked in principle and proved its potential for wastewater treatment along with simultaneous resource recovery. However, clogging of bed matrix and low COD and N removal were the issues faced during the operation. It should be noted that the performance achieved by the system was with unoptimised reactor components and operation parameters. Hence, the scope existed for further optimisation studies focusing on different process parameters to improve both wastewater treatment and resource recovery performance. These include operational parameters such as drip rate, feed volume and HRT, and reactor parameters such as design components, support bed materials, and plants with better growth rates and nutrient uptake capabilities. My thesis work focused on optimising the reactor design and components like bed matrix, cathode placement, and plant selection, followed by the performance evaluation of iHydroMET with the optimised components.

Chapter 2

Objectives

- To optimise the reactor components of the iHrdroMET system.
- To test the wastewater treatment performance of the upgraded iHydroMET system with optimised reactor components.

Chapter 3

Materials and Methods

3.1 Selection and optimisation of support bed matrix components

Different support bed matrices were tested to optimise a favorable support bed medium for plant growth and the proliferation of different microbial communities. The purpose was also to target the removal of both emerging pollutants and nitrogen efficiently without compromising the removal of other wastewater contaminants. These include granular activated charcoal (GAC), quartz/silica sand (QS), cocopeat (CP), and their combinations in different proportions (Photograph 1).

3.1.1 Cocopeat

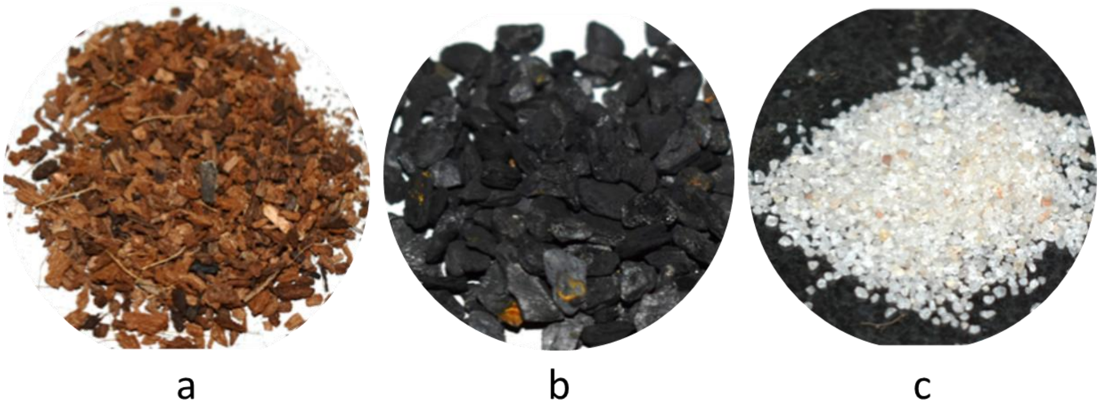
CP is known for its potential as a good support material not only to facilitate the growth of diverse microorganisms but also for anchoring plants (Awang et al., 2010). It is a natural fibre, which is extracted as a by-product from the outer coconut husk. It is widely used in horticulture and mixed with soil, which helps trap moisture for a longer time. It is very stable in terms of its physical and chemical properties. CP used in this study was manufactured in Karnataka, India, and bought from TrustBasket online store. The product was washed two times and dried at room temperature to remove its natural reddish colour and to wash off unwanted soil particles and dust before use. After drying, it was sieved to get particles of size 3-5 mm to get rid of fine-sized particles, which may cause severe clogging in the system.

3.1.2 Granular activated charcoal

It is a form of carbon derived by processing charcoal to achieve low-volume pores that increase its surface area for adsorption. It is widely used in wastewater treatment systems due to its adsorption capability of various contaminants (Hung et al., 2005). The GAC (Andel Marketing & Services) of size around 5 mm was used. It was washed twice and dried at room temperature to remove dirt and fine black charcoal particles before use.

3.1.3 Quartz sand

Quartz sand (QS) is white-yellowish sand composed of silicon dioxide. It is one of the low-cost bed materials used in conventional bio-filtration technologies like trickling filters, constructed wetlands, etc. (Dacewicz & Chmielowski, 2018). The QS of size around 2-3 mm was obtained from the Institute's STP. Before use, it was also washed and dried to remove dirt.



Photograph 1. Different materials tested as the bed matrix components: a) cocopeat, b) granular activated charcoal, and c) quartz sand

3.1.4 Bed matrix optimisation

The custom-made reactor units of dimensions 20 cm length and 7.5 cm diameter were filled up to 15 cm height with different support bed materials mentioned above, and their combinations (proportion by the length of the reactor and represented from bottom to top).

These include GAC:CP (50:50), GAC:QS (50:50), GAC:CP:QS (50:25:25), and GAC:QS:CP (50:25:25) (Fig. 1). GAC was used in all combinations. The removal of the major wastewater pollutants, namely, organic matter, phosphates, and ammonia, was then investigated by passing the wastewater through different bed matrices. All experiments were conducted in duplicate. During the acclimatization period, all the reactor units placed in a system were fed parallelly by continuously recirculating fresh 10L domestic wastewater twice a day. After attaining the stabilized performance, the domestic wastewater was fed in a down-flow manner to the individual reactor units with a flow rate of 40 ml/min with the help of a peristaltic pump for 20 min. The effluent samples were collected from the bottom of each reactor unit. The performance was assessed by analyzing different conventional wastewater parameters such as chemical oxygen demand (COD), orthophosphates, and ammoniacal-N at least three times for each reactor unit. Based on the results of different combinations of materials, we then tested different proportions of GAC and CP, such as GAC:CP (20:80), GAC:CP (40:60), and GAC:CP (60:40) in batch experiments under similar conditions (Fig. 1).

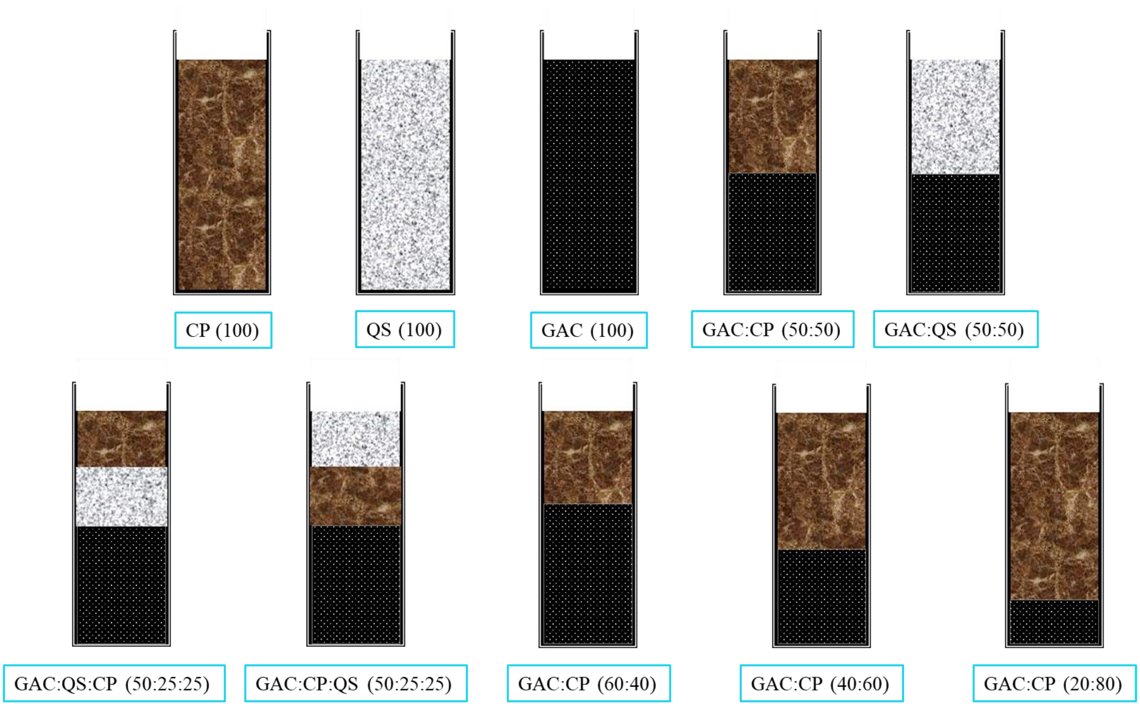


Fig. 1. Schematic of different tested bed matrices in the reactor unit

3.2 Cathode position optimisation

Graphite flat plates were used as the electrodes (IPGI Instruments, Chennai). Graphite possesses good electrical conductivity and biocompatibility and is non-corrosive. Also, it is cheaper than other types of electrode materials such as stainless steel, platinum, etc., and is readily available. Non-catalysed, rectangular-shaped graphite cathode with 4 cm x 4 cm and anodes with 2 cm x 3 cm dimension were used. Electrodes were pre-treated by acid-alkali method. Aluminum connecting wires were attached to the electrodes by drilling and sealing them using conductive cement. The anode was placed vertically in the activated charcoal bed.

Three identical bed matrix reactors, housing similar graphite anodes (surface area - 6 cm^2) at the bottom and cathodes (surface area - 16 cm^2), were used for the experiments. The reactors were placed in effluent chambers, each filled with 500ml of fresh domestic wastewaters. The wastewater was recirculated in each reactor using a peristaltic pump at a flow rate of 50 ml/min. The bottom of the reactor units was submerged by 1 cm in the effluent chamber containing wastewater to achieve a continuous medium for closing the circuit (Fig. 2). A 1000 Ω external resistor was used for the experiment to measure the closed-circuit voltage (CCV) using a digital multimeter (Fluke 179) in both configurations. It was found to be the optimum resistance during polarisation recorded at stable voltage by varying resistance method from 50K-250 Ω using resistance box. The cathode was switched between the two tested configurations, i.e., (i) top of the reactor unit and (ii) ¾ immersed in the effluent chamber while recording the CCV data. The ¾ immersion of cathode was selected based on the report suggesting it to be the best configuration for electricity harvest (Walter et al., 2019). The readings were taken for 6h at a regular interval of 1h and were repeated three times with fresh domestic wastewater each time.

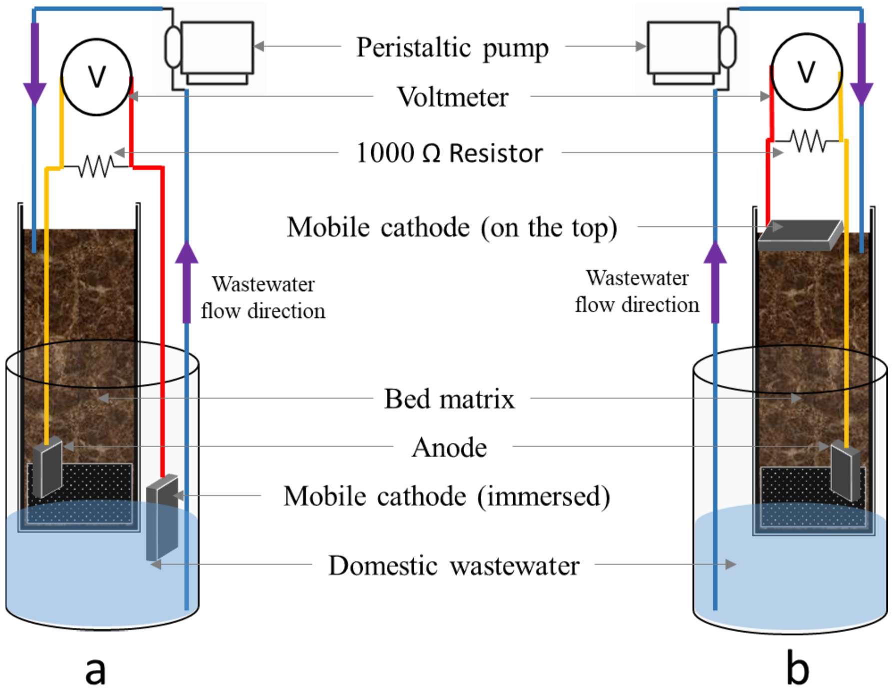
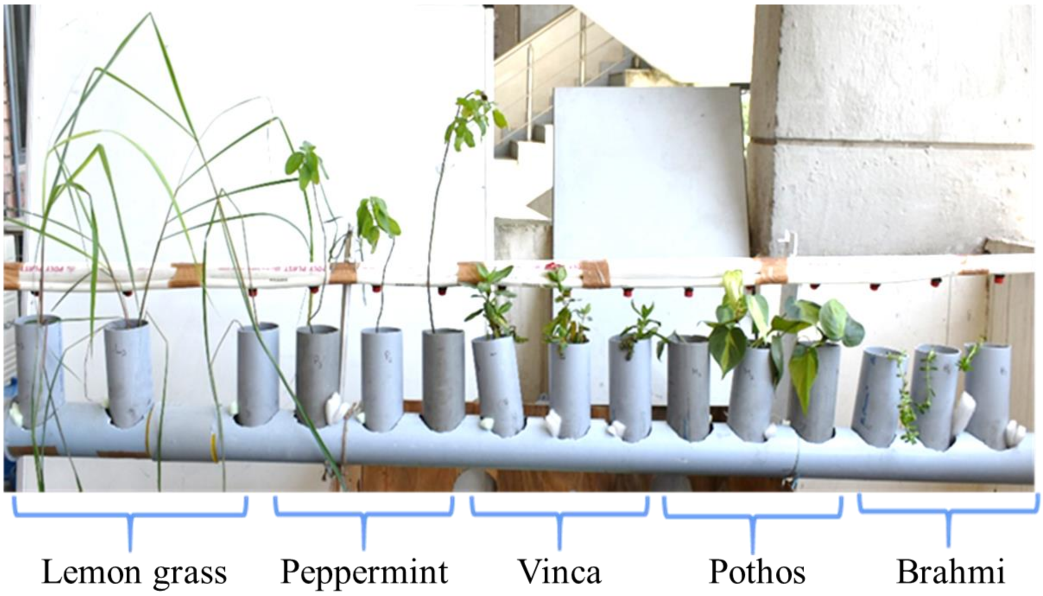


Fig. 2. Schematic of the experimental setup for harvesting electricity in two cathode positions: a) ¾ immersed in the effluent chamber and b) top of the reactor unit

3.3 Plant selection

Plants are an integral component of the integrated system. They important role in optimal wastewater treatment performance, particularly in removing nutrients N and P. Several types of plants with fibrous roots were tested for their capabilities to sustain growth in the reactor conditions. These include floricultural or aesthetic value plants (e.g., Vinca and Pothos) and commercially important plants (e.g., Peppermint, Lemongrass, and Brahmi). These were obtained from the IISER Mohali campus horticulture facility and Ajith Nursery Mohali. The experiments with different plants were conducted in triplicates. Different plants were fed with fresh domestic wastewater daily in a drip-hydroponics recirculation operation mode for a month-long period (Photograph 2). They were analyzed for easy adaptation to the domestic sewage condition, wastewater feeding operation, and visual growth. In the second set of experiments, nutrient removal by individual reactor units containing well-adapted and survived plants (i.e., Vinca and Pothos) was assessed. The plants were fed with 500 ml of fresh domestic wastewater in a recirculation mode (Fig. 3). The samples were taken after 12h, 24h, and 48h during recirculation and were analysed for TN and TP removal. The analysis was done three times with fresh feed each time.



Photograph 2. Different test plants planted in domestic wastewater-fed reactor units

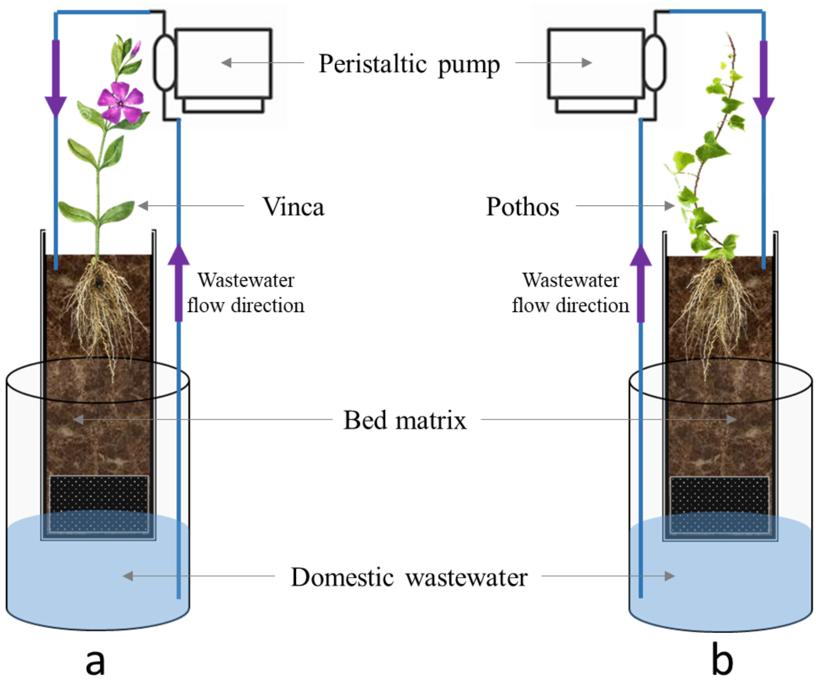


Fig. 3. Schematic of the experimental setup used to evaluate nutrient removal performance of the test plants: a) Vinca and b) Pothos

3.4 Performance evaluation of the upgraded iHydroMET system with the optimised set of reactor components

The upgraded iHydroMET system was fabricated using raw materials purchased from the local market. The system consisted of 10 reactor units, each having length of 20 cm (Figs. 4 and 5). The integrated condition comprises bed matrix (BM), electrodes-assembly, and plant sapling. All the reactor units were filled from bottom to top with, as per the selected combination (Section 3.1), i.e., GAC 20:80 CP proportion by the length of the reactor unit. The requisite number of similar stem cuttings of Vinca were propagated by placing them in tap water to develop roots and finally used for the main experiments. The graphite anode (surface area: 6 cm^2) was placed within the bed matrix embedded in the GAC. The graphite cathode (surface area: 16 cm^2) was vertically placed in ¾ immerged condition outside the reactor in the effluent collection duct, adjacent to each reactor unit (Fig. 4). Daily 10 L of fresh domestic sewage (DS) was collected at around 10 am from the sedimentation tank (primary treatment unit) of the sewage treatment plant facility located at IISER Mohali residential campus. In each experimental run, 10 L clarified domestic wastewater (i.e., primary effluent) was fed to the system via the drip manifold pipe. Each reactor unit received the feed at a flow rate of 50 ±10 ml/min. The system was operated indoors under ambient temperature (25 ± 4°C) and light illuminance (~5000 lux with 16h to 8h day and night period cycles) conditions and in a batch-recirculation drip-hydroponics mode for the wastewater feeding. Before starting the sampling period, the system was acclimatized by feeding the fresh wastewater for 15 days. The influent and the effluent samples of different hydraulic retention times (HRTs) such as 3h, 6h, 12h, and 24h were analyzed for five cycles with fresh domestic wastewater each time. The secondary effluent collected after 24h was passed through the UV system (GL6, Goodlife UV disinfection system) at a flow rate of 400ml/min. The wastewater treatment performance of the integrated system was assessed by monitoring pH (Oakton PC2700 pH meter), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD_5), total solids, nitrogen (i.e., Total Nitrogen, ammoniacal-N, nitrate-N, and nitrite-N), total phosphates, and MPN test for *E.coli.*

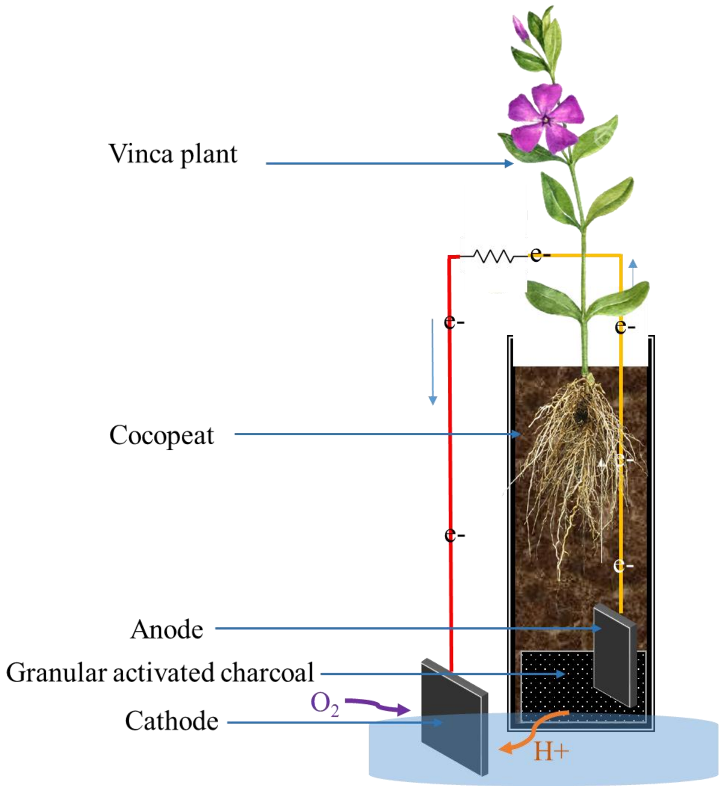


Fig. 4. Schematic of a single reactor unit configuration with the major components.

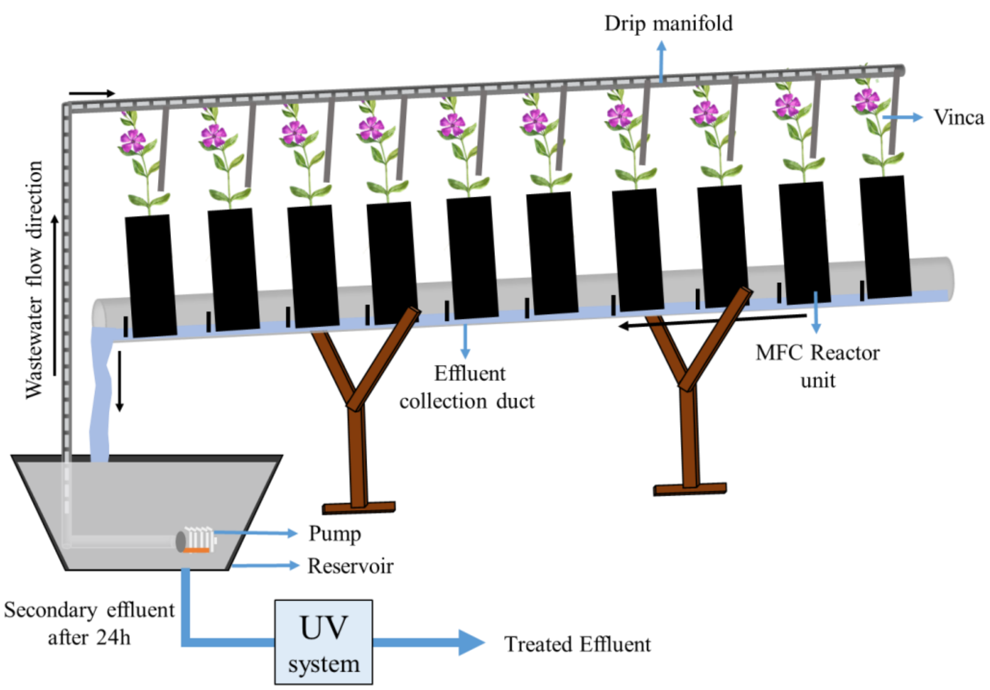


Fig. 5. Schematic of the upgraded iHydroMET system.

3.5 Analyses and calculations

3.5.1 Wastewater parameters

Different wastewater parameters were analysed according to the goal of each experiment. The pH was estimated using Oakton PC2700 pH meter, and BOD was estimated with the help of Oxitop BOD method, incubation at 20°C. The COD, ammonium-N, orthophosphate-P and *E.coli* were estimated by closed reflux, phenate, stannous chloride colorimetric and MPN methods, respectively using standard protocols adapted from "*Standard Methods for the Examination of Water and Wastewater"* (APHA, 2012). Nitrate-N and nitrite-N were estimated by reagent-free method (OptRF measurement) of UV-Vis spectrophotometer (Photolab 7600, Xylem Analytics, Germany). TN and TP concentrations were analyzed by the alkaline persulfate digestion method (De Borba et al., 2014). For TN, 2 ml sample was taken in digestion vials and 0.3 ml of alkaline persulfate digestion solution (0.3 gm of potassium persulfate in 5 ml of 1.5M NaOH solution) was added to it. The samples were digested at 120°C for 30 min and then analysed and quantified using UV-Vis spectrophotometer by taking the spectrum for the range of 220-230 nm and taking the double derivative of it. For TP, 2.5 ml of sample was taken in digestion vials, and 50 µL of H_2SO_4 solution was added to it. Then 20 mg of ammonium persulfate was added to the samples. The vials were then put for digestion at 120°C for 60 min. These samples were analysed for orthophosphate using a colorimetric test.

Standard calibration curves for COD, TN, TP, and ammonia at different concentrations were plotted using spectrophotometry (Photolab 7600 UV-VIS) and used to estimate the concentrations of these constituents in the test samples. The treatment performance was evaluated by estimating removal efficiencies (ξ) calculated using Eq. (1), where C_O represents the initial concentration of the specific pollutant in the influent, and C_S denotes the concentration of the same pollutant (mg/l) in the effluent.

$$\xi = \frac{C_o - C_s}{C_o} \times 100 \qquad \text{Eq. (1)}$$

The data are presented as averages along with uncertainties based on at least three analyses for each parameter in all experiments. All the visual observations were recorded in pictorial form using a Nikon D3400 DSLR camera.

3.5.2 Electrochemical parameters

Polarization behavior was recorded during the stable voltage phase of operation. The reactors of iHydroMET were connected in series and parallel configuration to assess energy harvest in a closed circuit mode. The polarization test was performed to determine the cell design point of the system using a various range of resistances in both series (50k-100 Ω) and parallel (1000-5 Ω) configurations. The closed-circuit voltage was recorded using the auto-range digital multimeter and datalogger (Keithley 2400). The CCV at the determined optimum resistance was also recorded for 6h in two cycles, each starting with a fresh load, for both series and parallel connections.

The slope of the polarization curve represents the internal resistance of the system, which was calculated using Eq. (2)

$$R_{int} = -\frac{dE}{dI} \quad \text{Eq. (2)}$$

Where, R_{int}: internal resistance (Ω),

E: potential (V)

I: current (A)

During the closed-circuit mode operation, the current (I) was calculated using Ohm's law, i.e., V = IR, and Power (mW) was derived from P=IV equation. Current density (mA/m^2) and power density (mW/m^2) were calculated by normalizing the respective absolute current and power values with the cathode surface area (0.0016 m^2).

Chapter 4

Results and Discussion

4.1 Selection and optimisation of support bed matrix components

All the tested bed material components and their combinations in the reactor units achieved stabilized performance within 10-15 days of wastewater feeding. The CP was the best performing support bed material among all for COD, ammonia, and phosphate removal (Table 1). These results validated the use of CP in our earlier experiments and suggested its use in future experiments as well. It has a high water-holding capacity which helps to stop the oxygen diffusion in the medium and helps to achieve anaerobic conditions. It supports plant growth and microbial growth by giving support and a nutrient-rich environment due to its high absorption capabilities (Awang et al., 2009). The data suggest that CP facilitated microbial degradation of organic matter and ammoniacal nitrogen conversion and phosphate adsorption more efficiently than the QS and GAC materials. QS was not effective in the removal of pollutants. It is primarily used as a filter bed without much associated physicochemical, specifically, adsorption or biological activities. GAC also did not show significant removal of COD, ammonia, and phosphate as compared to CP. However, it has been reported as good material for facilitating nitrate removal in the anoxic environment *via* microbial denitrification and as a high surface area adsorbent for emerging micropollutants like pharmaceuticals and personal care products (PPCPs) from the wastewater (Elumalai et al., 2014; Ohe et al., 2003). One gram of activated charcoal can have a surface area of up to 3,000 m^2 due to this high microporosity (Saleem et al., 2019). Moreover, the inclusion of conductive material like GAC at the bottom of the bed matrix can act as the additional electron acceptor for electroactive microbes, contributing to more electricity harvest (Caizán-Juanarena et al., 2020). Hence, we tested different proportions of GAC and CP, such as GAC:CP (20:80), GAC:CP (40:60), and GAC:CP (60:40). Among these three combinations, GAC:CP (20:80) combination showed comparable removal efficiencies to the only CP bed material (Table 1). Hence, it was selected for further experiments. The GAC offers additional benefits of nitrate and emerging pollutants (like PPCPs) removal (Ohe et al., 2003).

Table 1. COD, ammoniacal-N, and orthophosphate removal efficiencies (%) achieved by different support bed material matrices in reactor units.

S. No.	Bed matrix components (proportion %)	Removal efficiencies (%)		
		COD	**NH_3-N**	**Orthophosphate**
1	CP (100)	71.80 ± 2.64	79.80 ± 4.28	80.61 ± 1.18
2	QS (100)	65.11 ± 3.47	66.67 ± 6.69	73.23 ± 5.53
3	GAC (100)	35.42 ± 2.71	37.35 ± 3.49	66.24 ± 4.12
4	GAC:CP (50:50)	57.79 ± 2.67	58.16 ± 2.03	74.17 ± 1.51
5	GAC:QS (50:50)	50.60 ± 2.96	54.78 ± 1.90	70.52 ± 2.4
6	GAC:CP:QS (50:25:25)	48.44 ± 3.66	59.05 ± 1.55	72.03 ± 4.71
7	GAC:QS:CP (50:25:25)	45.31 ± 1.45	54.75 ± 2.03	69.82 ± 1.65
8	GAC:CP (20:80)	69.69 ± 1.94	68.75 ± 2.53	79.67 ± 1.33
9	GAC:CP (40:60)	65.22 ± 0.65	61.40 ± 2.31	77.32 ± 2.12
10	GAC:CP (60:40)	52.12 ± 2.45	56.23 ± 1.93	71.38 ± 3.46

4.2 Cathode position optimistion

The position of the electrodes in the reactors plays a crucial role in electricity harvest. Cathodes are usually reported as a limiting component for the oxygen reduction reaction, hence tested for proper placement in the reactor units (Khotseng, 2018). The CCV recorded at an optimised external load of 1000 Ω, in the two tested cathode placement conditions i.e., a) on the top of bed matrix and b) ¾ immersed in the effluent (Walter et al., 2019), were in the range of 47.17 ± 21.21 mV to 54.67 ± 24.32 mV and 77.75 ± 31.25 mV to 92.41 ± 32 mV respectively during 6h of operation. The maximum CCV was recorded after 1h of operation, and thereafter it gradually decreased over the operation time irrespective of the tested condition (Fig. 6). It might be because the organic load was initially higher, and it decreased slowly with time. The CCV obtained in the case of cathode immersed in the effluent was higher (by a margin of 34±4mV) compared to cathode placed on the top of the bed matrix. Three possible reasons for higher CCV in the immersed cathode condition as compared to top placed cathode include (i) short effective distance (1-2 cm) between the electrodes in the former condition as compared to the later (13-14 cm) (Sajana & Mitra, 2013), (ii) the liquid medium condition between electrodes in former as compared to the less conductive solid bed matrix in the later condition, and (iii) direction of the hydrogen ion movement according to the wastewater flow making available more H^+ ions for the oxygen reduction reaction at the cathode in the former case.

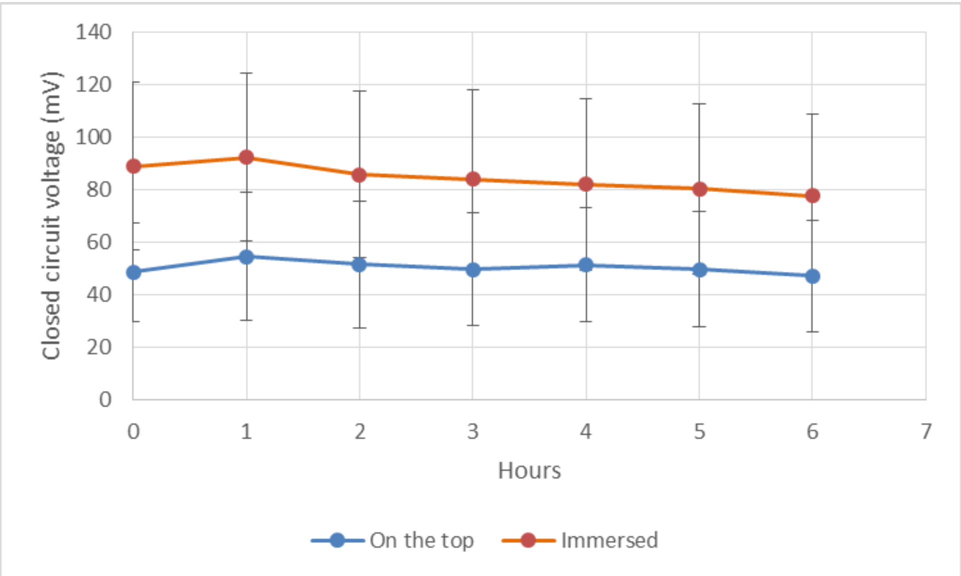


Fig. 6. Closed-circuit voltage in two cathode placement conditions for 6h.

4.3 Plant Selection

Domestic wastewater has a high level of nutrient concentration in terms of nitrogen and phosphorus. All plants require these essential nutrients for their growth and development, and they uptake them from the surroundings (Roy et al., 2006). Among the tested plants, Brahmi and lemongrass died due to unfavorable nutrient conditions or system feeding pattern and external factors like pest attack on lemongrass. The rest of the three plants, i.e., Vinca, Pothos, and Peppermint, survived throughout the experimental cycle, but Peppermint did not show substantial growth (Fig. 7). Vinca and Pothos showed fast growth. The initial concentration of TN was 52±11 mg/L and TP was 4.2±0.5 mg/L in the fresh domestic wastewater. The final TN concentration after 48h was around 18 mg/L for Pothos and 8 mg/L for Vinca (Fig. 8). The final TP concentration after 48 hours was 2.7 mg/L for Pothos and 2.1 mg/L for Vinca (Fig. 9). The removal of TN (Vinca: 44 mg/L; Pothos: 34 mg/L) and TP (Vinca: 2.1 mg/L; Pothos: 1.5 mg/L) was slightly higher for Vinca compared to Pothos in 48h tests. These observations are in agreement with reported studies on Vinca's capability for nutrient removal from wastewater and phytoremediation of heavy metals like cadmium, lead, zinc, copper, and nickel (Frantz, 2013; Barbosa et al., 2020; Pandey et al., 2007). Based on better nutrient removal results and considering Vinca's additional benefit of producing attractive flowers compared to Pothos, it was selected as the potential plant candidate for the iHydroMET technology.

Fig. 7. Pictorial view showing the increment in shoot biomass ofrepresentative tested plants grown for 1 month: a) Vinca; b) Pothos; c) Peppermint; d) Brahmi, and e) Lemongrass

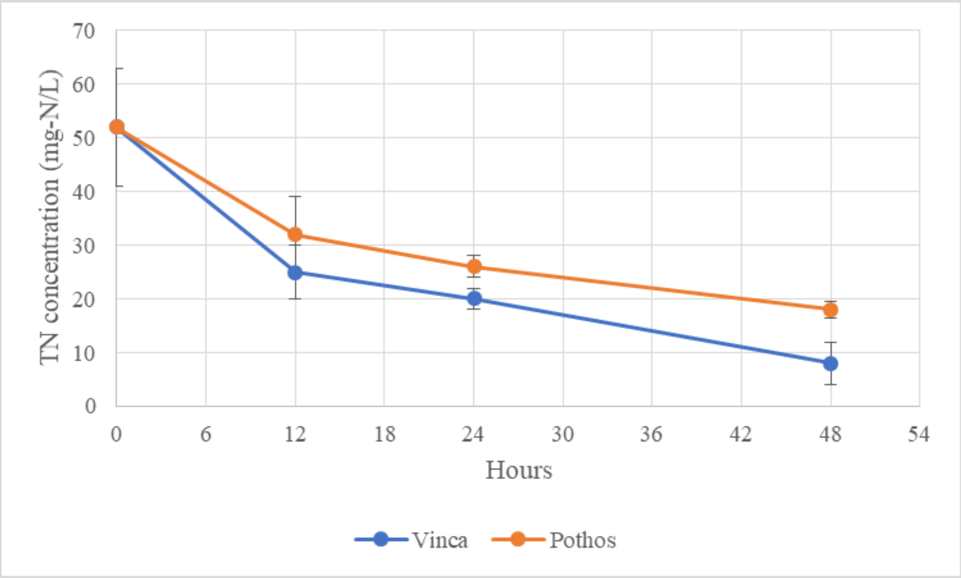


Fig. 8. Removal of total nitrogen (TN) by the tested plants for 48h period

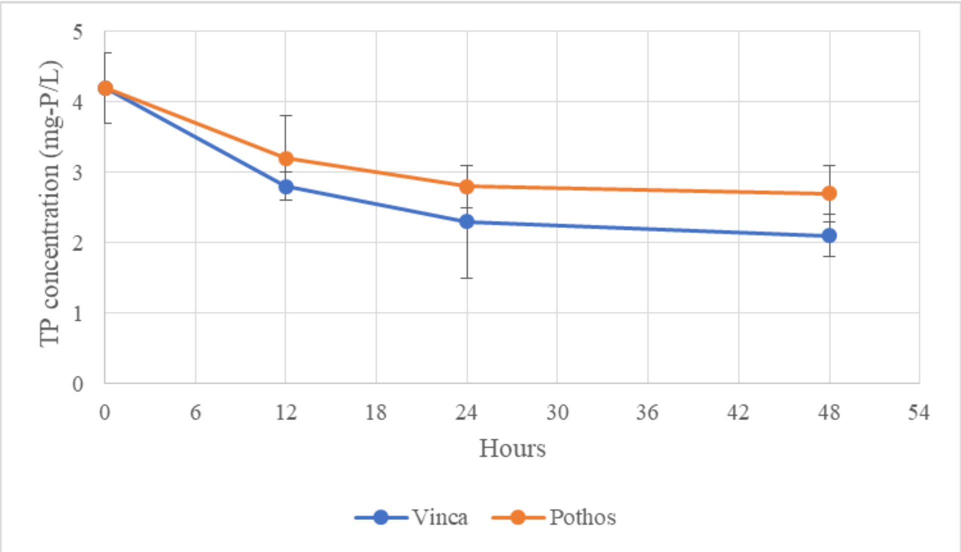


Fig. 9. Removal of total phosphorus (TP) by the tested plants for 48h period

4.4 Performance evaluation of the upgraded iHydroMET with the optimised reactor components

The characteristics of the influent (primary clarified) domestic wastewater and effluent are shown in Table 2. The wastewater treatment performance of the upgraded iHydroMET in terms of removal efficiencies of different components achieved at different HRTs is discussed in the following subsections.

Table 2. Consolidated data of wastewater treatment performance parameters of iHydroMET at different HRTs

Parameters	Influent	Effluent at different HRTs			
		3h	6h	12h	24h
pH	7.62±0.14	7.68±0.03	7.79±0.01	7.90±0.02	7.95±0.01
Conductivity (µS/cm)	883±84.85	837±4.82	798.5±1	767.5±1.76	749±3.6
COD (mg/L); (%)	237±66	48±16; (79±6.7)	35±16; (84±6.3)	23±13; (90±3.1)	22±8; (91±3.3)
BOD (mg/L); (%)	193±46	40±7 (79)	30±1 (84)	8±4 (96)	7±3 (97)
BOD:COD	0.81±0.09	0.71±0.15	0.68±0.1	0.31±0.07	0.27±0.12
Total Phosphorus (mg/L); (%)	6.59±0.36	5.26±0.27; (20±8.4)	4.65±0.22; (28±0.6)	4.59±0.19; (29±0.9)	4.76±0.19; (32±1.1)
Ammonia (mg/L); (%)	30.78±5.2	8.99±2.23; (70±2.2)	1.37±3.41; (95±3.4)	0.09±0.65; (99±0.6)	0±0.07; (100)
Nitrate (mg/L)	1.49±0.26	17.78±0.44	25.44±0.42	32.9±1.92	36.27±2.42
Nitrite (mg/L)	0.56±0.49	3.81±0.2	5.81±0.82	0.48±0.83	1.12±1.12
Total Nitrogen (mg/L); (%)	38.14±7.45	33.96±3.93; (10±2.7)	31.89±4.46; (16±9.2)	28.63±7.19; (25±7.4)	27.89±8.48; (34±5.7)

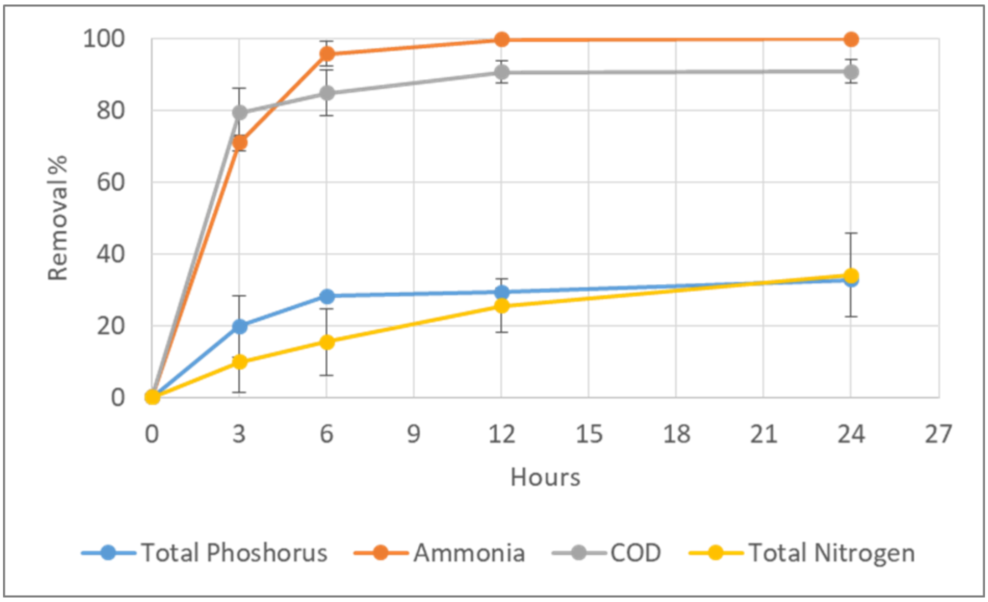


Fig. 10. Removal efficiencies of various pollutants by iHydroMET at different HRTs

4.4.1 Organics

Domestic wastewater is mainly composed of a biodegradable fraction of organics. The average BOD:COD ratio of the influent was 0.81 ± 0.1 during the experimental period. It suggests that a majority of the organics content is biodegradable (Choi et al., 2017). Hence, the BOD:COD ratio decreased with time as the fraction of biodegradable organics was consumed/degraded by the microbes (Table 2). About 80% of the organic matter was removed in the first 3h of operation due to entrapment of the organics in the bed matrix, which can be further degraded by microorganisms. Around 90% COD removal efficiency was achieved after 24h of operation (Fig. 10). It is mainly due to the following reasons: (i) the increase in the bed matrix component, i.e., cocopeat and GAC. Due to an increase in height, the wastewater had to travel longer in the reactor, which leads to maximum adsorption of the organics in the bed matrix and eventual degradation by microbes. (ii) The wastewater is recirculated in the reactor, which provides sufficient aeration for aerobic microbial interactions to oxidise organic matter (Tantak et al., 2014). (iii) In addition to aerobic and anoxic microbial activities, the diverse microbiota in the rhizospheric zone can also contribute to COD removal by facilitating conversion of complex organic forms into simpler ones that can be used up by other microbes. (iv) The electrogenic microorganisms present in the anodic zone oxidise organic matter using the anode as an electron acceptor and further enhance organics removal (Jiang et al., 2021). Overall, the upgraded system showed a significant improvement, from 72% removal (in the earlier system) to 80%, in terms of COD removal after 3h of operation (Yadav et al., 2020).

4.4.2 Total Phosphate

The maximum phosphate removal efficiency of around 32% was achieved in the upgraded system after 24h operation. Plant uptake and adsorption of orthophosphate on bed matrix are the two key activities contributing to TP removal. From Fig. 10, it is clear that most of the removal was observed in the first 6h, which might be due to the trapping and adsorption (Shi et al., 2011), along with plant uptake of different types of phosphate forms (Frantz, 2013). However, no removal occurred after 6h suggesting a low requirement of phosphorus by plants. The specific microbes, i.e., phosphate-accumulating microorganisms (PAOs) are also reported for phosphate removal under controlled alternate aerobic and anaerobic conditions (Ubukata, 2006). However, it was most likely not the case in the tested reactor units due to the inadequate anaerobic zone.

4.4.3 Total Nitrogen

The characterization of influent shows higher concentration of ammonia (31 mg/L) and lower nitrite (0.6 mg/L) and nitrate (1.5 mg/L) concentrations. After 6h of operation, almost all ammonia got converted to other nitrogen forms, i.e., nitrite (5.8 mg/L) and nitrate (25.4 mg/L) due to microbial nitrification processes facilitated by continuous aeration and mixing of wastewater due to recirculation. We earlier hypothesized that the presence of cocopeat might facilitate a significant anaerobic microbial environment in the lower reactor portion, enhancing the denitrification process and conversion of nitrite to nitrate and further nitrate reduction to free nitrogen. However, from results, it is clear that the effect of the denitrification process was not so prominent, resulting in the accumulation of nitrate and nitrite forms in the effluent. It suggests that the wastewater feeding mode and process were not appropriate to achieve a prominent anoxic zone for better removal of nitrate and nitrite by the denitrification process. In addition, the continuous recirculation of aerated wastewater does not allow its retention for a longer duration, thereby creating a low or negligible anoxic environment. Overall, around 34% removal in the TN concentration was observed after 24h operation. From Fig. 10, for the first 12h there is a steady increase in the removal efficiency, which might be mostly due to the trapping and adsorption along with plant uptake of different types of nitrogen forms (Ohe et al., 2003). However, the slow removal trend after 12h can be justified by only the plant uptake process. Apart from the conversion of ammonia into nitrite and nitrite, the organic nitrogen forms get trapped or adsorbed in the bed matrix, which are then converted to simpler inorganic nitrogen forms by microbial interactions. Plants can ultimately take these up for its growth. It can be confirmed by the visual growth observed in Vinca. An emerging anammox process, which is reported for ammonia oxidation process at the anodic zone, might not be happening due to inadequate anaerobic zone in our reactor units (Qu et al., 2014).

4.4.4 pH

pH is very crucial for biological interactions, chemical and physical treatment of the wastewater. Biological processes would not function properly at abnormal pH, leading to a decrease in the pollutant removal rates. pH was monitored throughout the operation cycle, which co-relates with the stable removal efficiencies. Influent pH was 7.6. As the time progressed, the pH increased (3h - 7.7; 6h - 7.8; 12h - 7.9; 24h - 8.0). It might be happening due to the stripping of CO_2 due to operational conditions and organic matter degradation (Cohen & Kirchmann, 2004).

4.4.5 *E. coli*

Fecal coliforms (predominantly *E. coli*), which originate from fecal wastes of humans and animals, are found in wastewater streams. The influent MPN was $(3.6\pm1.5)\times10^{11}$ MPN/100ml. There was no removal of coliforms after 24h operation in the secondary effluent. It suggests that the iHydroMET system is not efficient for the removal of microbial load. Hence, the UV treatment system was used. It achieved the removal of almost all coliforms since the final treated effluent showed a negligible count (<2 MPN index/100 ml) for *E. coli*. The disinfected iHydroMET effluent can be used for different reuse purposes.

4.4.6 Polarization curve and power output

Polarization behavior is used for analyzing the electrical output performance of the fuel cells. With an increase in resistance, there was a decrease in current. At low resistance, current density was high, but the potential was low because more electrons flow in the circuit at low resistance. At maximum load, 50 kΩ in series and 1000 Ω in parallel, negligible current output was recorded. At the resistances of 9000 Ω in series and 150 Ω in parallel, maximum power densities were observed, i.e., 1.2 mW/m^2 in series and 11.7 mW/m^2 in parallel connection (Fig. 11). In the proof of principle study, power densities were 30.9 mW/m^2 and 31.6 mW/m^2 in series and parallel connections, respectively (Yadav et al., 2020). The lower power density output might have happened in the upgraded system because the organic load in the influent was low as COD in the influent was around 200 mg/L less compared to the proof of principle study (COD: 411±32 mg/L). It is reported that the power output is directly dependent on the organic load of the wastewater (Velvizhi & Venkata Mohan, 2012). In addition, most of the complex organics might have been trapped in the upper bed matrix of the reactor unit, which can be likely degraded by the aerobic microbes without contributing to energy harvest. Some loss of organics might also be happening while reaching the bottom zone of the reactor unit, covering a distance of around 10 cm. Hence, at the active anodic bottom zone, a low amount of organics would be available for electroactive microbes to produce free electrons and hydrogen ions. Also, due to poor anoxic conditions in the anodic region, fewer microbes can use anode as the terminal electron acceptor as aerobic or facultative microbes dominate such environments.

In serially connected MFCs, the output voltage will be higher, but the current will be less, whereas in parallelly connected MFCs, the output current will be higher, but the voltage will be less. As per the appliance requirement, the type of connection, i.e., serial or parallel, can be chosen. The combination of series and parallel connection of MFCs can be done to optimise the energy harvest of the whole system.

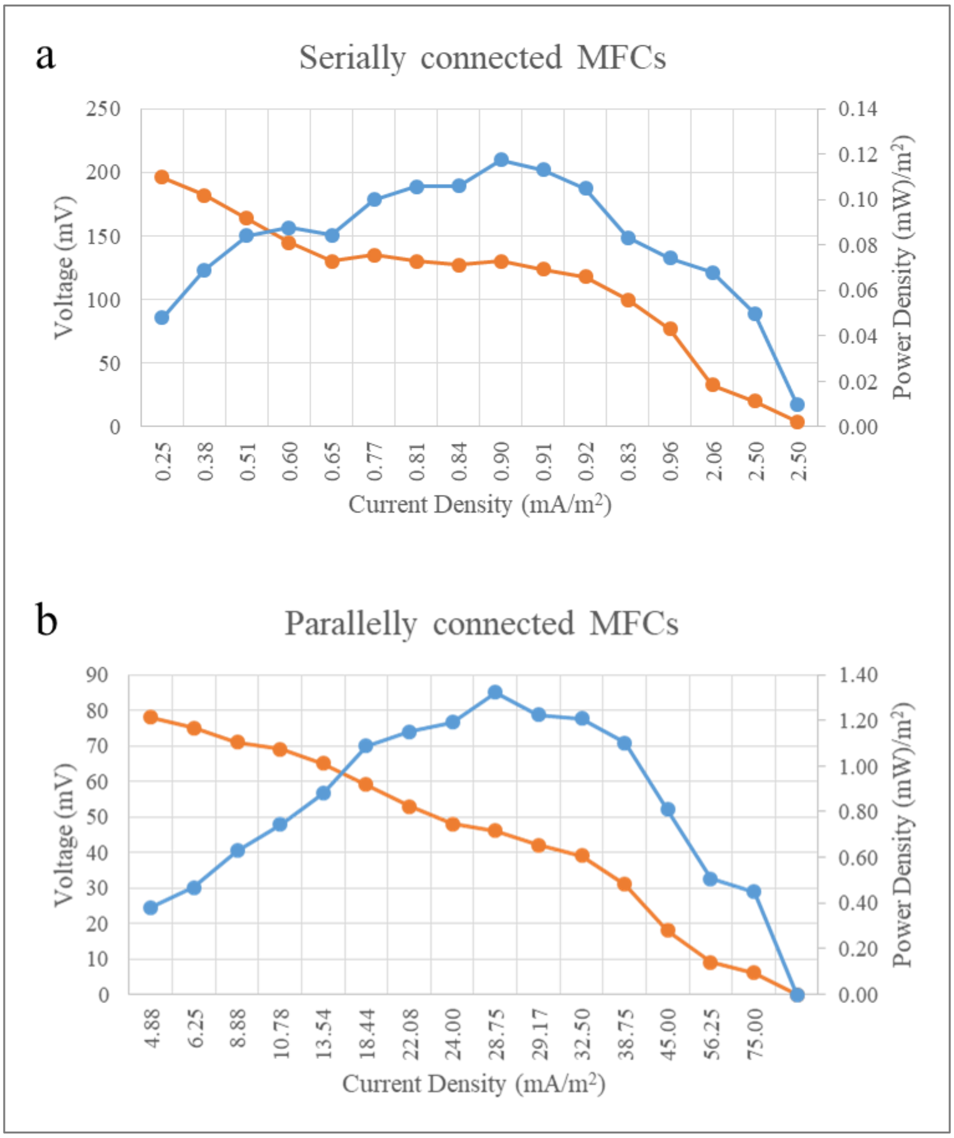


Fig. 11. Polarization (orange) and Power density (blue) curves of a) serially connected MFCs, and b) parallelly connected MFCs of the iHydroMET system.

At the determined resistances (9k Ω and 150 Ω in series and parallel, respectively), the long-term closed-circuit power generation performance was recorded for 6h (Fig. 12). The CCV dropped with time and stabilised at ~50 mV and ~10 mV in series and parallel connections, respectively. It is the actual energy harvest during the system operation.

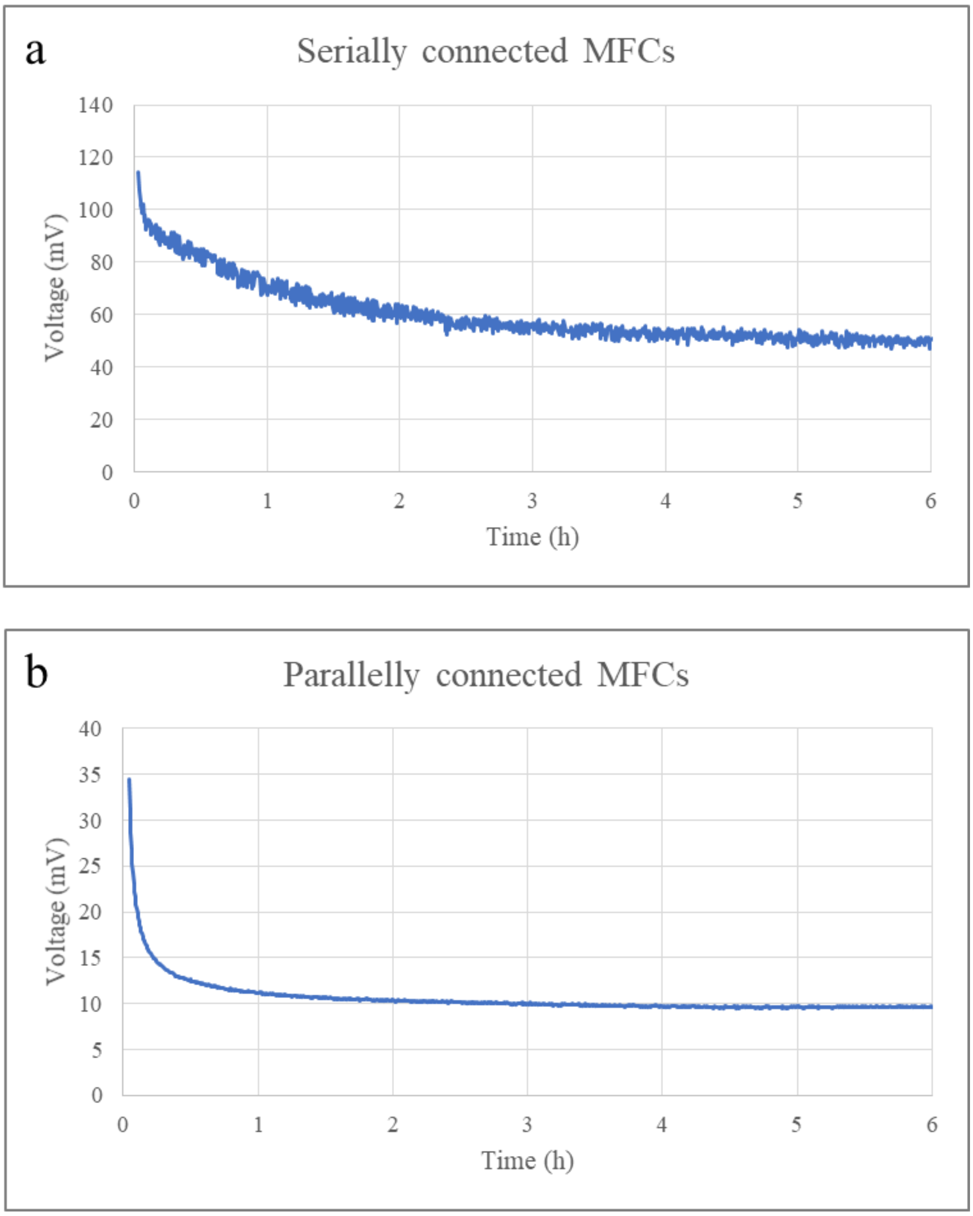


Fig. 12. Closed-circuit potential curves for 6h at optimum resistance with the a) serially connected MFCs, and b) parallelly connected MFCs of the iHdroMET system.

Chapter 5

Conclusions and future perspectives

In this work, the optimisation of different reactor components for the iHydroMET system was successfully achieved. The combination of GAC:CP (20:80) was found to be the best performing bed matrix to facilitate the removal of nitrogen, organics, and phosphates. The cathode placement in the effluent was the best for maximum energy harvest in the tested reactor units. Among different plants, Vinca was chosen for further studies because of its efficient capabilities for removing nutrients such as N & P and improving the aesthetics of the system.

The upgraded iHydroMET with the optimised reactor components showed efficient organic removal (up to 90% within 24h) but could not achieve the TN and TP removal efficiencies to the desired levels. UV disinfection further enhanced the effluent quality of iHydroMET. The effluent can be used for different purposes like gardening and flushing, as the effluent contains a good percentage of N and P, which is free of harmful microbes (Pescod, 1992). TP removal was less due to the low phosphorus requirement by plants and inefficient PAOs' activity because of the inadequate anoxic zone. TN removal was low because the denitrification process was not prominent due to the absence of anoxic conditions and continuous recirculation of aerated wastewater. The system may be modified slightly by submerging the reactor units half in the wastewater to make a significant anoxic zone and enable the denitrification process in the lower portion of the reactor, which was the limiting process in this study.

The TN removal efficiencies might be improved considerably by creating a saturation zone at the bottom region of the reactor unit. This modification may also increase the system's power output due to favorable growth conditions for electroactive microorganisms. In addition, the combination of series and parallel electrical connections of reactor units can be optimised to get maximum energy harvest from the whole system. The TP removal can be enhanced by maintaining controlled alternate aerobic and anaerobic conditions to enrich PAOs. Also, the microbial community assessment can be investigated to improve performance further. Recirculation can also be avoided to stop continuous mixing of treated effluent to the influent, which is not an effective way of feeding. A combination of serially and parallelly hydraulically connected reactor unit combinations can be tried. The optimisation studies focusing on different parameters such as mode of operation, hydraulic retention time (HRT) or recirculation time, drip rate to each reactor unit, hydraulic and organic loading rate need to be conducted to enhance the performance of iHydroMET. In conclusion, my thesis work suggests that a better performing iHydroMET can be configured with further improvements, which can be implemented as a low-cost treatment technology for managing wastewater at the points sources.

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