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Domestic wastewater treatment in a coupled SBR-ECR process

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Domestic wastewater treatment in a coupled SBR-ECR process

ABSTRACT

The effectiveness of a sequenced biological-physicochemical reactor system for treating sewage was studied. The biological degradation was conducted in a Sequential Batch Reactor, which had innovative features for simplifying the operation and maintenance of the reactor. The reactor was operated at 4, 6, 8, and 12 hr cycle. Up to 82% removal of Chemical Oxygen Demand (COD), 50% removal of Dissolved Organic Carbon (DOC), 45% removal of Total Nitrogen (TN), and 45% removal of Total Phosphorus (TP) was achieved. The treated effluent was further polished in a Continuous-flow Bipolar-mode Electrochemical Reactor to remove additional recalcitrant organic matter from the wastewater. The process parameters were optimized using Response Surface Methodology. At the optimum condition (pH = 8.7; Current = 1.0; reaction time =9.0), up to 90% removal of COD, 67% removal of DOC, 61% removal of TN, and 99.9% removal of TP was achieved in the coupled system. Micropollutants belonging to Pharmaceutically Active Compounds, pesticides, etc. were significantly removed. The coupled system completely removed *Salmonella*, *Pseudomonas*, and *Staphylococcus*. However, coliforms were detected at the outlet samples. A UV or ozone disinfection treatment is recommended for the safe reuse of the treated water for non-potable purposes.

Keywords: Domestic wastewater treatment; Sewage; Sequential Batch Reactor; Electrocoagulation

1. Introduction

About 75000 million liters per day (MLD) of wastewater is produced from class I cities (with over 1,00,000 populace) and Class II towns (between 50,000 and 99,999 populace) of India. Only about 26000 MLD receive some level of treatment (ENVIS Centre on Hygiene, Sanitation, 2019). The rest is released into receiving water bodies with no treatment, thereby causing water pollution. It is essential to treat wastewater to an extent such that the treated water can be, at least, reused for non-potable purposes, thereby preserving freshwater reserves. Wastewater treatment has become very challenging due to the presence of micropollutants, which are introduced into wastewater streams from several sources. The occurrence of micropollutants in the aquatic condition has become a global issue of increasing environmental concern. Micropollutants also called 'Emerging Contaminants', are present in the aquatic systems at levels of ng/L - μg/L and comprise a large and growing array of organic and inorganic compounds that have anthropogenic as well as natural sources (Luo et al., 2014). Micropollutants have toxic, carcinogenic, and mutagenic effects on life forms exposed to them. The anthropogenic micropollutants include pharmaceutical & personal care products, bulk drugs, steroids & hormones, pesticides, industrial chemicals, petroleum additives, heavy metals and metalloids, surfactants, endocrine disruptors, and disinfection by-products (Luo et al., 2014; Martínez et al., 2014). The existing wastewater treatment plants are not specifically designed to remove micropollutants; therefore, they fail to address this issue. Regulations and monitoring of micropollutants have not yet been well established. Many micropollutants can pass through wastewater treatment plants due to their persistence or/and constant addition and, therefore, end up in the aquatic environment (Luo et al., 2014).

Several combinations of biological and physicochemical processes have been used in treating domestic wastewater. Sequential Batch Reactor (SBR) is one such biological treatment system that has been used quite extensively in domestic wastewater treatment. An SBR is a one-tank system which is operated in batch (fill-and-draw) mode. This reactor is process-wise different from the Activated Sludge Processes (ASP), which operate on continuous-flow mode. The operation of SBR proceeds through five phases, namely, fill, react, settle, draw, and idle (Metcalf & Eddy, 2003). In the beginning, the reactor is seeded with active biomass. In the 'fill' phase, the reactor is filled with influent. During the 'react' phase, the content of the reactor is aerated and mixed vigorously. It is during this phase that biodegradation of organics takes place. During the 'settle' phase, the mixing is stopped and the content is allowed to settle. The supernatant is drawn out of the reactor during the 'draw' or 'decant' phase. This is followed by the

'idle' phase during which the reactor is at rest. Excess sludge is also wasted during this phase. The whole cycle is then repeated.

SBR systems require less space and have lesser operating costs in comparison with ASP systems (Lim, Seng, Lim, Ng, & Sujari, 2011). This system, in its different configurations, is efficient in removing nitrogen, phosphorus, and chemical oxygen demand (COD) effectively (Showkat & Ahmed, 2019). SBR is widely accepted for treating domestic wastewater (DWW). However, it is still challenging to meet water reuse guidelines due to the presence of micropollutants, which are persistent and are difficult to degrade biologically (Wolska, Cieszynska-semenowicz, & Ratajczyk, 2019).

The above problem may be mitigated by further polishing the effluent of a biological treatment system in a downstream electrochemical reactor (ECR) system. Electrochemical (EC) reactions are complex processes involving quick redox responses that happen at the electrodes. Literature indicates that hydrogen gas that evolves at the cathode results from the reduction process, while oxidation at the anode causes the generation of metal ions (adsorbent). Hydrogen gas causes the aggregated particles to float on the water (Kumar, Ponselvan, Malviya, Srivastava, & Mall, 2009a). The electrochemical disintegration of the iron anode releases Fe^{+2} ions, which ultimately oxidize to form an iron hydroxide coagulant due to dissolved oxygen in water (Damaraju, Gupta, Bhattacharyya, Panda, & Kurilla, 2020). Electrochemical strategies can be suitable alternatives for removing recalcitrant compounds and supplements present in wastewater because of their high expulsion proficiency, simple process, and low treatment costs (Damaraju, Gupta, et al., 2020). EC process has been tried for the treatment of poultry-wastewater, pharmaceutical-wastewater, textile-wastewaters, and restaurant-wastewater. It has been used to remove organics, boron, kaolin, lead, nitrate, and heavy metals from wastewaters. The organic pollutants are removed by adsorption on the coagulant, generated during the electrochemical reactions, or passive oxidation at the electrode (Damaraju, Gupta, et al., 2020). The removal of the inorganic pollutants takes place through adsorption on to coagulant or due to chemical precipitation; both the mechanisms are pH-dependent (Damaraju, Bhattacharyya, Panda, & Kurilla, 2020b). EC process, when coupled with other treatments like anaerobic fixed film bed reactor (AFFBR) and photocatalysis, enhanced the biodegradability of emerging contaminants (Zaied et al., 2020).

In this study, a modified Sequential Batch Reactor (SBR) coupled with a continuous-flow bipolar-mode Electrochemical Reactor (ECR), was used to treat domestic wastewater to address

the emerging problem related to micropollutants. Apart from micropollutant analysis, other conventional water quality parameters like Chemical Oxygen Demand (COD), Dissolved Organic Carbon (DOC), Total Nitrogen (TN), Total Phosphorus (TP), pH, Dissolved Oxygen (DO), Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), and pathogens were also analyzed. While the purpose of the SBR was to remove the biodegradable portion of the organics present in wastewater, ECR was meant to remove more recalcitrant fractions, including micropollutants, which are usually bio-resistant. The SBR used in this study had a novel spring-based floating decanter, a compartmentalized sloping base, retractable diffusers, and other features. This reactor has several advantages over conventional SBRs in terms of operation (for example, sludge wasting and retention of adequate biomass in the reactor) and maintenance. The ECR consisted of a parallel array of iron plates, also called sacrificial electrodes, inserted between the two end electrodes, which are connected to the power supply. The continuous-flow bipolar mode electrochemical reactors require a smaller footprint, reaction time, and energy compared to conventional monopolar mode electrochemical reactors for the same degree of wastewater treatment (Damaraju, Bhattacharyya, et al., 2020b).

2. Materials & methodology

2.1. Domestic wastewater

The experiment was set-up on the premises of a Common Effluent Treatment Plant (CETP) in the city of Hyderabad, India. A nearby canal, which collects domestic wastewater (sewage) from different locations in the city, was selected as the source of the feed water for the system. The inlet and the outlet samples were collected daily during the period of operation. The collected samples were properly stored at a temperature below $0^{o}C$ before analyses.

2.2. Reactor set-up and operation

Aerobic SBR - A 7-m^3 circular steel tank (Diameter = 2.25m; Height = 2.25 m; Working volume = 4250 L) was fabricated. The reactor had a compartmentalized sloping base, retractable diffusers, and other improved features aimed at simplifying the reactor operation (for example, sludge wasting and retention of adequate biomass in the reactor) and regular maintenance. A valve-based floating decanter was used. The spring-valves at the inlet to the decanter prevented the

accumulation of suspended solids inside the decanter during the aeration phase and minimized the escape of suspended solids with the treated effluent. Usually, the decant phase starts after the complete settling of sludge. In the present study, a floating device enabled the decanter to float near the surface of the wastewater and moves up and down along with the water level in the SBR. The improved feature of the spring-based decanter allowed water to be decanted from the top as soon as it gets clarified instead of waiting for complete settling of sludge. The design of the decanter allowed a reduction in the settling time. It prevented the escape of suspended solids with the supernatant (through the decanter) without extending the time of withdrawal. A schematic of the reactor is shown in Fig-1(a).

The SBR was seeded with activated sludge, which was collected from the aeration tank of the CETP. The seeding sludge had a mixed liquor suspended solids (MLSS) concentration of 7 g/L and a sludge volume index (SVI) of 85.5 mL/g. The seed sludge volume was adjusted to have an initial MLSS of 4 g/L in the SBR. The SBR was operated at four cycles – 12, 8, 6, and 4 hours. The aeration phase constituted 50% of the total recycle time. A Programmable Logic Controller – Motor Control Centre (PLC-MCC) (Make: Siemens) was used to control the sequence of the whole operation of the SBR. Pumping, aeration, settling, and discharge functions were automatically controlled by the program incorporated in the system.

ECR - The ECR, a Continuous Bipolar-mode Electrochemical Reactor, was connected in series with the SBR when the latter was operated at 4-h cycle time. During operation, after being treated biologically, a fixed fraction of the effluent was pumped into the ECR in continuous mode. The ECR was a 1-L vessel made up of polyacrylic sheets. It had a working volume of 765 mL. The reactor contained an array of 15 parallel iron electrodes, each with dimensions 15 cm x 3.4 cm x 0.3 cm. The electrodes functioned as central sacrificial electrodes and end electrodes were attached to a DC (Direct Current) supply with a variable output of 0-220 V. The raw DWW entered the reactor from the bottom, and the treated wastewater left the reactor from the top. The ECR set up is shown in Fig-1(b).

2.3. Process optimization of ECR

The optimization of the process was carried out using response surface methodology (RSM) for maximizing the treatment efficiency. Central composite design (CCD) was used. The independent process variables and their range considered in the study were pH (X_1): 2-10, retention time (X_2,

min): 0.5-10, and current (X_3, amp): 1-3. The removal efficiencies of COD, DOC, TN, and TP were selected as the dependent variables.

A second-order polynomial model, as shown in equation-1, was used to express the percentage removal as the function of the independent variables.

$$Y = \beta_0 + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_i^2 + \sum_{i=1}^{3}\sum_{j=i+1}^{3}\beta_{ij} X_i X_j \quad (1)$$

Where Y is the predicted response for independent variables X_i, X_j, the constants in the equation, that is, β0, β_i, β_{ii}, β_{ij} are, constant term, i^{th} linear, quadratic, and interaction coefficients, respectively. Response optimization and data processing were performed using Design Expert 11.1.2.0. The responses which were considered in this present study were COD removal (Y_1, %), DOC removal (Y_2, %), TN removal (Y_3, %), and TP removal (Y_4, %).

2.4 *Analytical methods*

Physicochemical analysis

The pH, COD, DOC, TN, TP, TSS, and VSS were determined following the protocols given in the *Standard Methods for the Examination of Water and Wastewater* (APHA, 2012). Total COD (CODt) was measured directly. The samples were passed through filters with a pore size of 0.45 µm before determining the filtered or soluble COD (CODs). DOC and TN were analyzed using the TOC-L analyzer (Brand: Shimadzu TOC-L series) and TN analyzer (Brand: Shimadzu TNM-L series).

Micropollutant analysis

A high-resolution accurate mass (HRAM) LC-MS/MS system (Agilent 6545 QTOF) was used for the micropollutant analysis. The full-scan method was used for identifying the known-unknowns and the unknown-unknowns. The Zorbax Aq column (10 X 4.6 mm, 3.5 u) was used for the separation. For analysis, 20 µL Aliquots of each sample were injected into the HRMS system. The temperature of the oven was maintained at 30°C and the flowrate was 400µL/min. Mobile phase A consisted of 1 mM ammonium acetate and 0.1 % Acetic acid in water (Mobile Phase A). Mobile Phase B consisted of Acetonitrile with 0.1% Acetic acid (Mobile Phase B). Methanol /Water (50/50, V/V) was used as the flushing solvent. After elution, the detection was performed in ESI-positive and ESI-negative modes. The nitrogen (sheath gas) was used at 350 deg ^{o}C, 30 psi nebulizer pressure, 4500 V capillary voltage in positive mode, and 3500 V capillary voltage in

negative mode. The nozzle voltage for positive mode was 500 V and for negative mode, it was 1000 V. The data processing was performed with the advanced data mining algorithm named Molecular Feature Extraction (MFE). Targeted compounds were identified using Agilent Mass Hunter Water Screening Personal Compound Database and Library (PCDL). The compounds identified with a score above 95% were reported.

Microbial analysis

Indicator microorganisms such as *Escherichia coli, Staphylococcus aureus, Pseudomonas spp. and Salmonella spp.* were identified in the influent and the effluent samples using Multiple-tube Techniques described in the *Standard Methods for the Examination of Water and Wastewater* (APHA, 2012). The membrane filtration technique was used for measuring fecal coliforms and E. coli.

3. Results and Discussion

3.1. Characteristics of the domestic wastewater

The initial characteristics of the domestic wastewater are given in table-1. The pH of inlet wastewater was 7.7 ± 0.35 and COD was 135.9 ± 27.2 mg/L. The TN was 38.6 ± 8.09 mg/L and ammonical nitrogen (NH_4^+-N) was 23.8 ± 2.2 mg/L. Nitrogen was present mostly in the organic or reduced form. During the reactor operation, foaming was observed, which indicates the presence of detergents and surfactants in the wastewater. However, the TP value was low, 1.36 ± 0.45 mg/L, which is because detergents are, nowadays, produced with less phosphorus content. TSS was high and highly variable (280.3 ± 91.7 mg/L). Typically, domestic wastewater can be classified into high-, medium-, and low-strength wastewater. Table-1 shows typical values of different water quality parameters. It can be inferred that the domestic wastewater used in this study was low-strength wastewater. Similar values were also reported elsewhere (Alagha, Allazem, Bukhari, Anil, & Mu′azu, 2020; A Singh et al., 2015).

3.2. Phase-1

3.2.1 Performance of the SBR

pH is known to affect microbial growth (Grady, Daigger, Love, & Filipe, 2011). pH between 7.5 and 9.2 is essential for the active growth of microorganisms (Chan, Chong, & Law, 2010). In the current study, the inlet pH was 7.6 ± 0.3 and the outlet pH was pH 8.1 ± 0.21. The slight increase

in pH was possibly due to the removal of any residual carbon dioxide from the wastewater due to the sparging effect of aeration. Following the principles of carbonate equilibrium in water, it can be said that as the carbon dioxide concentration decreases, the pH increases. At pH around 8.34, no carbon dioxide (or carbonic acid) exists in the system (Sawyer et al. 2003). Previous researchers have also noticed a rise in pH during the aerobic biological treatment of wastewater (Lijklema, 1972).

Dissolved oxygen (DO) concentration below 0.5 mg/L in the reactor results in the bulking of sludge and limits the oxygen penetration into the sludge (Chan et al., 2010). In general, DO concentration should be in the range of 1.5 and 2 mg/L to remove the carbonaceous BOD. Higher DO concentration (>2 mg/L) improves the nitrification process (Metcalf & Eddy, 2003). According to the literature, SBR sludge has good thickening properties and a reasonable settling rate at higher concentrations (2-5 mg/L) of DO (Balme & Wile, 1999). In the present study, the raw water DO was around 0.5 ppm and never exceeded 1 mg/L. Outlet DO was mostly between 2.5 and 4.5 mg/L.

The performance of the SBR was evaluated based on COD, DOC, TN, TP, and TSS removals at 12, 8, 6, and 4 h cycle, as shown in Fig-2. Fig-2(a) indicates the total COD concentration and its removal and Fig-2(b) indicates soluble COD concentration and its removal. The total COD and soluble COD removals were above 70% at all substrate concentrations except during the 4h cycle. Fig-2(c) represents the DOC concentration and its removal at various cycles. DOC removal efficiency was found to be above 45% at 12, 8, and 6h cycles. However, in the 4h cycle, the removal efficiency dropped to 41%. The COD and DOC removal efficiencies were reduced at the 4h cycle because of the lesser availability of aeration time. Similar observations were reported in the literature as well (Kayranli & Ugurlu, 2011). The total COD in the effluent was less than 50 mg/L, which satisfied the revised discharged guideline laid down by the Central Pollution Control Board of India (CPCB, 2015). Fig-2(d) represents the TN removals at various cycles. The TN of the influent wastewater ranged between 25 and 45 mg/L and decreased to 15-25 mg/L after treatment, indicating a nitrogen removal of about 40% in the process. Since the outlet DO was well above 2.5 mg/L, it can be inferred that the removal of nitrogen was primarily due to its assimilation by the microorganisms for new biomass growth (Gupta, Choi, Pawar, Jung, & Lee, 2016). Li and Irvin, (2007) have drawn similar conclusions. The outlet nitrogen was in the form of nitrate-N, which indicates that the wastewater was sufficiently nitrified before discharge. However, the outlet TN value could not satisfy the revised discharge guideline (=10 mg/l) issued

by the Central Pollution Control Board of India (CPCB, 2015). These results show that the process should be modified to include a biological denitrification step. A polishing step should be introduced after SBR to meet the discharge guideline for nitrogen. Fig-2(e) depicts the TP removals. The total phosphorus in the effluent was less than 1 mg/L at all the cycles. The Fig-2(f) represents TSS concentration and its percentage removal at different cycles. The inlet wastewater TSS was 280.3 ± 91 mg/L, which indicates significant fluctuation. The effluent average TSS was around 60 mg/L at all the cycles. This corresponds to a removal efficiency of about 75%. The reduction happens due to the settling of the sludge after the aeration phase. The TSS concentration and the fluctuations were significantly reduced after the treatment. The spring-based valves at the entry to the decanter prevented the accumulation of suspended solids inside the decanter during the aeration phase. Fernandes et al., (2013) operated a full-scale sequential batch reactor for domestic wastewater with an 8h cycle and observed 70% TSS removal efficiency.

The SVI parameter is useful for assessing sludge stability in any suspended growth (activated sludge) reactor. SVI was monitored after the end of the REACT (or aeration) phase of each cycle. An SVI of 150 ml/g is often regarded as the boundary between sludge with good settling (SVI <150) and sludge with poor settling (SVI> 150) (Grady et al., 2011). In the present study, the sludge had an average SVI of 70 ml/g and had good settling properties.

MLSS and Mixed Liquor Volatile Suspended Solids (MLVSS) were monitored periodically throughout the study. MLSS is comprised of volatile solids and inert solids. Volatile suspended solids give an estimate of the microorganism concentration. The MLSS and MLVSS of the reactor varied from 6000-8000 mg/L and 3000-4500 mg/L, respectively. Maintaining adequate biomass in the reactor helps achieve high efficiency during wastewater treatment (Chan et al., 2010). The optimum biomass concentration (MLSS) for the proper functioning of aerobic suspended growth processes is 3000-6000 mg/L (Metcalf & Eddy, 2003).

Table-2 summarizes the performance of the present system and compares it with the performance of some of the similar systems reported in the literature. Jakubaszek and Stadnik, (2019) studied the performance of a household wastewater treatment plant at Koscian, which operated an SBR. At 6-h cycle time, 87.6% of COD, 94.5% of TSS, 77% of TN, and 61% of TP removal were achieved. The authors observed that the treated water met the discharge requirements. They concluded that the proper functioning of the system depends largely on proper utilization and upkeep of the system. Debik and Manav, (2010) tried to determine the optimum cycle time with short aeration time in an SBR treating domestic wastewater. The highest removal

efficiency was achieved when FILL, ANAEROBIC, AEROBIC1, ANOXIC, AEROBIC 2, SETTLE, and DECANT phases were operated at 0.5, 2, 2, 1, 0.75, 1, and 0.5 h, respectively. The COD, TKN, and TP removals were observed to be 91%, 78%, and 87%, respectively. The researchers claimed that higher removal efficiency at shorter aeration time would ensure a low-cost solution for nutrient removal from domestic wastewater. Pelaz et al., (2018) operated a 5-L SBR at 12, 8, and 6h cycle time with domestic wastewater having a low COD/N ratio. At the 6-h cycle time, the anoxic-aerobic-anoxic sequence of treatment yielded the best result; about 84% of TN and 77% of soluble COD were removed. The researchers concluded that the process might be successfully applied as a post-treatment for the removal of nitrogen from anaerobically treated domestic wastewater. Alagha et al., (2020) operated a pilot-scale reactor under different anoxic conditions and studied the suitability of SBR for wastewater treatment. COD, TN, TP, and TSS removals were about 91%, 83%, 90%, and 99%, respectively**.** The authors concluded that the SBR system could treat municipal wastewater and could be used in remote and arid areas**.** Lin and Cheng, (2001) tried a treatment method consisting of chemical coagulation and SBR. A novel SBR was designed that allowed continuous wastewater inflow which was in contrast to intermittent wastewater feeding of the conventional SBRs. The SBR was operated at 12-h cycle. The chemical coagulation was able to remove COD and color by up to 75 and 80%, respectively**.** SBR was able to bring down the COD and turbidity to below 20 mg/L and 2 NTU, respectively. The treated effluent was deemed fit for agricultural reuse.

It can be seen from Table-2 that although the SBR in the present study was able to significantly remove pollutants from domestic wastewater, its performance was somewhat less compared to the studies reported in the literature. Also, the treated effluent could not satisfy the latest discharge guideline for TN (=10 mg/L), and TSS (=20 mg/L) laid down by the Central Pollution Control Board of India (given later in Table-4). The micropollutants in the wastewater (discussed in section 3.4) might have had an inhibitory effect on the microorganisms present in the SBR, thereby, reducing the performance of the system. Therefore, it was decided to integrate a physicochemical treatment unit, an ECR in this case, to the SBR to polish the effluent further so that the final water quality can meet the discharge criteria.

3.2.2 Material Balance for Carbon

The material balance was performed by assuming the chemical formula of the biomass to be $C_5H_7NO_2$ and the equation for balancing the carbon in the system is shown below (Eq. 2)

V * (C in inlet DOC + C in inlet VSS) = V* (C in outlet DOC + C in outlet VSS) + dC (2)

Where,

V - Volume of water treated per cycle (4500L)

C - Carbon

DOC - Dissolved organic carbon

VSS - Volatile suspended solids

dC - Carbon released in the form of CO_2

Table-3 shows the mass balance for carbon. DOC and VSS have been expressed as carbon. The change in the total inlet carbon and the total outlet carbon can be assumed as the carbon leaving the system during the aeration period as carbon dioxide. As seen from the table, 55-65% of the total inlet carbon was released in the form of carbon dioxide during the aeration phase. Washington and Symons, (1962) conducted a carbon balance experiment and reported that around 63-90% of the inlet carbon was eliminated as CO_2. Lijklema, (1972) reported that most of the carbon compounds in wastewater are oxidized at high sludge concentrations.

3.3 Phase-II

3.3.1 ECR

The ECR was integrated with the SBR to bring effluent COD, DOC, TN, TP values within the permissible limit. During the 4-hour cycle treatment in the SBR, a fraction of the effluent water was pumped into the ECR in continuous mode. SBR performance was found to be excellent at 12-hour, 8-hour, and 6-hour cycles. However, during the 4-hour cycle, the performance decreased. It was decided to select this cycle to couple the ECR with the SBR and to operate the combined system at higher loading without compromising the quality of the treated effluent.

3.3.2 Model formulation & Validation

Central composite design (CCD) was used. Altogether, 20 experimental runs were performed in duplicates, which included six central points, 2k axial points, and 2^k factorial points (k-number of the independent variables). Design Expert (version 11.1.2.0) was utilized for designing the experiment.

Analysis of variance (ANOVA) was performed to determine the statistically significant independent variables affecting the response (Almeida, Erthal, Padua, Silveira, & Am, 2008). The

aptness of the model is determined by the Coefficient of Determination (R^2). The R^2 value must exceed 0.75 for a good model (Naik & Setty, 2013). In the present work, the R^2 values of COD, TOC, TN, and TP were observed to be 95.40, 97.71, 86.21, and 95.43, respectively. The coefficient of determination for all the responses was greater than 0.75, indicating satisfactory model performance. The adjusted R^2 value was also checked. In all the cases, R^2 and R^2_{adjusted} values were in close agreement with one another, thus, demonstrating the significance of the model (Jadhav & Mahajan, 2013).

It has been stated in the literature that for linear, interaction, and quadratic responses, a quadratic model, would be statistically significant if the P-value is below 0.05. (Kumar, Ponselvan, Malviya, Srivastava, & Mall, 2009b). In the current study, it was observed that linear, quadratic, and interaction terms were below 0.05, thereby, indicating that the terms were significant for COD, DOC, TN and TP removals (except for 2-way interaction for TN and TP removals). A lack-of-fit test is used to evaluate the statistical model (Almeida et al., 2008). Lack-of-fit values for all variables were more than 0.05, indicating the non-significance, which proves the model was statistically significant (Damaraju, Bhattacharyya, Panda, & Kurilla, 2019).

3.3.3 Significance of Process variables

The responses - COD, DOC, TN, and TP removals were determined after the completion of the experimental runs. The second-order quadratic model was fitted between the responses and the independent variables by using multiple regression analysis. Regression models produced quadratic equations and surface response plots for all the responses. The quadratic equations and surface plots have been explained. In the equations, for the main terms, the positive sign indicates a synergistic effect and the negative sign demonstrates an antagonistic effect (Jadhav & Mahajan, 2013).

Effect of the independent variables (operating parameters) on COD removal:

The quadratic equation for COD removal is represented by equation-3.

$$Y_{COD} = 171.3 - 15.88\ pH + 15.58\ RT - 133.2\ C + 0.476\ pH*pH - 0.954\ RT*RT + 31.23\ C*C + 0.796\ pH*RT + 5.75\ pH*C - 6.17\ RT*C \quad (3)$$

The current was seen to be the most important parameter among the first-order and the second-order terms. The interaction between the retention time and current was the most significant among the interaction terms. The 3D surface plots for the removal of COD have been presented in Fig-3(a-c). Fig-3(a) represents the interaction of pH and retention time for COD

removal. The increase in retention time improves COD removal efficiency. The removal efficiency was observed to be almost constant with pH. Fig-3(b) express the interaction of pH and current. The COD removal efficiency decreased with a decrease in pH and significantly increased with the current. The pH of the solution is critical for the performance of the EC process. It influences the removal rates of TOC and COD. Many studies reported that a pH of around 7.0 is optimal for treating different types of effluents in an EC process (Garcia-Segura, Eiband, de Melo, & Martínez-Huitle, 2017). The pH range between 6.0-8.3 has shown good removal efficiency in the present study. The higher removal efficiency might be due to adsorption (at pH >7) and charge diffusion (at pH <7) mechanisms (Zaied et al., 2020).

Fig-3(c) indicates the association between current and retention time. The pattern of the graph suggests higher removal of COD at the center of the selected range of retention time and current. In this study, the current more than 1.8 amp has shown high COD removal. COD removal is known to increase with increasing the dose of iron in chemical coagulation (Kumar et al., 2009a). Thus, in the EC process, the amount of hydrous oxide formed in the solution governs the removal of COD. According to Faraday's principle, the theoretical amount of material liberated per unit surface area is directly related to the current density. Thus, COD removal by EC is governed by the generation of metal hydrated complexes of ferric oxide (Kumar et al., 2009b).

Effect of the independent variables (operating parameters) on DOC removal

The quadratic equation for DOC removal is represented by Equation-4 and explains all the important terms in the model for DOC removal.

$$Y_{DOC} = 66.5 + 8.63\ pH - 4.01\ RT - 44.55\ C - 0.905\ pH*pH + 0.1695\ RT*RT + 6.79\ C*C + 0.310\ pH*RT + 1.616\ pH*C - 0.891\ RT*C \quad (4)$$

The current was found to be the most critical parameter. The interaction between pH and current was observed to be critical than the other interaction terms. The surface plots of DOC removal are shown in Fig-3(d-f). Fig-3(d) indicates the interaction of retention time and pH on DOC removal. The highest DOC removal was found near the center of the selected pH range and when the retention time was in the range of 2.5-6 min. Fig-3(e) indicates the association of current and pH on DOC removal. As the pH increase, the DOC removal also increases to some extent and then it decreases; however, it increases with an increase in current. Fig-3(f) indicates the association of current and retention time, which depicts an increase in efficiency of removal with increment in the current. The removal efficiency was observed to be almost constant within the selected range

of RT.

In the EC operation, pH is an influencing factor in removing suspended particles by producing Fe^{3}+ (from Fe^{2}+) (Zaied et al., 2020). The surface charge of coagulant can change with a change in pH. Charge neutralization and adsorption or indirect chemical oxidation are the possible mechanisms for TOC removal. When the pH is between 4 and 5.5, the charge neutralization dominates. Flocs of Fe $(OH)_3$ are observed when the range of pH is more than 7. These flocs accelerate the process of adsorption by providing more surface area (Damaraju, Bhattacharyya, Panda, & Kurilla, 2020a). When pH is less than 6, indirect chemical oxidation may happen. In indirect chemical oxidation, intermediate compounds were formed in the reactor when the organic contaminants undergo oxidation. The escape of carbon in the form of carbon dioxide also happens. In this study, high current densities at the pH range 4.5-8.5 favored good removal efficiency.

Effect of the independent variables (operating parameters) on TN removal:

The quadratic equation for TN removal is represented as equation-5 and explains all the essential terms in the model for TN removal.

$$Y_{TN} = -93.3 + 5.27\ pH + 16.11\ RT + 57.7\ C - 0.384\ pH*pH - 1.039\ RT*RT - 13.48\ C*C + 0.579\ pH*RT + 0.63\ pH*C - 3.41\ RT*C \quad (5)$$

It was seen that all the main effects, including pH, RT, and C were positively associated, and the interaction terms including pH*pH, RT*RT, C*C, and RT*C were negatively associated with TN removal. The literature shows similar observations in the past (Damaraju, Bhattacharyya, et al., 2020a). The current was found to have a significant effect on main and quadratic terms. The interaction between retention time and the current was seen as the most noteworthy among the interaction terms. The surface plots of TN removal are shown in Fig-3(g-i). Fig-3(g) indicates the interaction of retention time and initial pH on TN removal. The TN removal was found to increase with an increase in pH and retention time. Bui, (2018) reported a similar observation. When the retention time has increased the generation of hydrogen gas at cathode and metal ions at anode observed, this phenomena improves the removal of pollutants in the EC process. The current density and time of reaction are proportional to the removal of typical pollutants (Zaied et al., 2020). Fig-3(h) indicates the interaction of pH and current on TN removal. With an increment in pH marginal improvement in removal, efficiency was observed. The highest removal efficiency was observed at the central value of the current (1.75-2.5 amp). However, when the current is

beyond 2.5 amp, there is a reduction in TN removal.

Fig-3(i) shows the interaction of current and retention time. The pattern of the graph indicates plateau, which depicts the higher removal efficiencies at near central values of retention time and the selected current range. It was found that due to the mechanism of adsorption and desorption. The treatment efficiency increased with the retention time up to a certain point, but after that, it gradually decreased.

Effect of independent variables (operating parameters) on TP removal:

The quadratic equation for TP removal is represented as Equation-6 and explains all the important terms in the model for TP removal.

$$Y_{TP} = 70.9 + 8.48\ pH + 2.24\ RT - 10.42\ C - 0.7110\ pH*pH - 0.0915\ RT*RT - 0.13\ C*C - 0.049\ pH*RT + 1.530\ pH*C - 0.245\ RT*C \quad (6)$$

Among the first-order terms, the current was seen as a critical parameter and the pH was seen as more critical among the second-order terms. The interaction between current and pH was observed to be the most significant among the interaction terms. The 3D surface plots for TP removal are represented in Fig-3(j-l). Fig-3(j) represents the interaction of pH and retention time on TP removal. The removal efficiency of TP increased with an increase in pH and retention time. Fig-3(k) shows the association of pH and current on TP removal. The removal efficiency of TP was found high near the central values of pH (Bui, 2018; Nez, 2003). However, the removal efficiency was seen as nearly constant with the current. It was found that the removal of phosphate pH acts as a sensitive parameter. Chemical precipitation and adsorption are the two methods widely used for the removal of phosphate; the reactions are given in Eqs 7 and 8.

$$Fe^{3+} + {PO_4}^{3-} \leftrightarrow FePO_{4(s)}\downarrow \quad (7)$$

$$FePO_4\downarrow \leftrightarrow Fe^{3+} + {PO_4}^{3-}\ K_{spFePO_4} = 10^{-21.9} \quad (8)$$

At pH below 6.5, phosphate precipitates as Ferric phosphate ($FePO_4$). At higher pH, iron gets converted to Fe $(OH)_3$ and adsorption becomes the dominant mechanism. The oxides and hydroxides act as binding sites for adsorption.

Fig-3(l) represents the interaction of current and retention time on the removal of TP. The TP removal was found to be constant and similar at all the current and retention time ranges. Since the inlet TP was very less, the effects of the independent variables on the removal of TP were not very prominent in this study.

3.3.4 Multi-objective optimization

Multi-objective optimization was performed for each response by building up a desirability function. The response process variable was converted into a dimensionless desirability scale between 0 and 1. The d = 0 shows an undesirable response; d=1 indicates the most desired response. The composite desirability function D is the weighted geometric average of each response (Bezrra, Erthal, Padua, Silveira, & Am, 2008). The responses considered for optimization were COD, DOC, TN, and TP removal efficiencies. The optimum condition was achieved when the pH was 8.78, retention time was 9.04 min, and the current was 1 amp. Individual desirability function of COD, DOC, TN, and TP removals were observed to be 1.0, 0.94, 0.90, and 1.0, respectively. Composite desirability was 0.96; all values were close to 1. For model validation, an experimental run was performed at the optimized condition in triplicate. The sample from the optimized experimental run was used to find the responses and was compared with the model's predicted responses. The predicted COD, DOC, TN and TP removals were 85.17%, 44.21%, 44.09% and 100%, respectively, whereas the corresponding experimentally obtained values were 76.56%, 42.86%, 39.2% and 100%. At the optimized point, a decent correlation was observed among the predicted and observed responses.

The TN concentration in domestic wastewater was 38.6 ± 8.09 mg/L. The outlet TN value dropped down to around 10 mg/L after EC treatment. The overall TN removal in the combined SBR-ECR system was more than 70% and was able to achieve the discharge criteria set by the Central Pollution Control Board of India (CPCB, 2015). Table. 4 shows the inlet and the outlet parameters when the SBR was operated at a 4-h cycle and the ECR was operated at the optimized condition. The treated effluent satisfied the latest discharge guideline for COD and TSS laid down by the Central Pollution Control Board of India (CPCB, 2015). The effluent TN was also very close to the new limit set by the regulatory body.

3.4 Micropollutant Removal

The LC-HRMS analysis was performed to identify the organic contaminants in the raw influent and the treated effluent. Based on the abundance values (area under the peaks of the corresponding compounds in the mass spectrum), the removal efficiency was calculated. Table-5 shows the removals of the micropollutants in the SBR and the ECR. The overall removal has been calculated and compared with the data available in the literature. The organic compounds belonging to different categories are discharged as a parent compound or their metabolites (Brown & Wong,

2018). Micropollutants are likely removed through adsorption on the coagulants which are produced from the anode during the electrochemical process. Marie et al., (2019) stated that electrostatic attraction between micropollutants and the positive metal complexes aggravates the removal of micropollutants like Diclofenac (DCF) during electrocoagulation. Removal through passive oxidation at the electrode is also a possibility. However, this aspect was not studied in this research. Ten out of nineteen micropollutants observed in this study were pharmaceutically active ingredients. The remaining nine compounds belonged to the pesticide family. Most of the micropollutants were completely removed in the SBR-ECR coupled system.

3.5 Pathogen Removal

Total coliforms and fecal coliforms are commonly used as indicator bacteria to detect the presence of pathogens in water or wastewater (Kermani, Bina, Movahedian, Amin, & Nikaein, 2008). The removal of coliforms and pathogens in a wastewater treatment plant depends on several factors such as pH, retention time, organic load, hydraulic loading rate, and the influent characteristics (Anju Singh et al., 2017). In this research, *Escherichia coli, Staphylococcus .aureus, Pseudomonas spp. and Salmonella spp* and Fecal Coliform have been monitored. The SBR alone was not effective in removing the pathogens, whereas the coupled system completely removed *Salmonella, Pseudomonas*, and *Staphylococcus aureus*. The bacterial cell wall contains a negative charge due to the presence of teichoic acids in the underlying plasma membrane or linked to the peptidoglycan layer (Weidenmaier & Peschel, 2008). During electrochemical reactions, the charge on the cell surface is neutralized in the presence of Fe^{2+}/Fe^{3+}. This helps in bacterial floc formation and eventual precipitation along with the metal hydroxides (Drogui, Brar, & Benmoussa, 2008). In this study, coliforms were detected at the outlet samples even after the ECR treatment. A tertiary UV or ozone disinfection treatment is, therefore, recommended for safe reuse of the treated water for non-potable purposes.

4 Conclusions

The treatment of domestic wastewater by a coupled SBR-ECR reactor system was investigated in this study. The primary objective of this research was to enhance the quality of the treated domestic wastewater to meet water reuse guidelines. The SBR was operated at 4, 6, 8, and 12 hr cycle. Up to 82% removal of Chemical Oxygen Demand (COD), 50% removal of Dissolved Organic Carbon (DOC), 45% removal of Total Nitrogen (TN), and 45% removal of Total

Phosphorus (TP) was achieved. The treated effluent was further polished in a continuous-flow bipolar-mode Electrochemical Reactor to remove additional recalcitrant organic matter from the wastewater. The process parameters - pH, current, and reaction time were optimized using Response Surface Methodology. At the optimum condition (pH = 8.7; Current = 1.0; reaction time = 9.0), up to 90% removal of COD, 67% removal of DOC, 61% removal of TN, and 99.9% removal of TP was achieved in the coupled system. Several micropollutants belonging to pharmaceutically active ingredients and pesticide families were detected in the wastewater. Most of the micropollutants were completely removed in the coupled system. The coupled system completely removed *Salmonella, Pseudomonas*, and *Staphylococcus aureus*. However, coliforms were detected at the outlet samples. A tertiary UV or ozone disinfection treatment is, therefore, recommended for safe reuse of the treated water for non-potable purposes.

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Captions for Tables

Table 1: Initial characterization of the domestic wastewater

Table 2: Comparison of the present study with existing literature

Table 3: Material balance for estimating the conversion of carbon to carbon dioxide

Table 4: Wastewater characteristics after SBR-ECR treatment

Table 5: Details of Micro-pollutants and their removal efficiencies at different stages of treatment

Parameter	**Present Study**	**Literature** (Metcalf & Eddy, 1991)			**Literature** (Alagha, Allazem, Bukhari, Anil, & Mu′azu, 2020)	**Literature** (Singh et al., 2015)	**Unit of measurement**
		Strong	Medium	Weak			
pH	7.7 ± 0.35	--	--	--	7.4 ± 0.23	6.82	
COD	135.9 ± 27.2	--	--	--	180 ± 70.9	242	mg/l
DOC	104 ± 49.72	--	--	--	--	--	mg/l
BOD	70 ± 17.10	300	200	100	79.9 ± 25.8	89	mg/l
TN	38.6 ± 8.09	85	40	20	17.2 ± 3.91	--	mg/l
($NH4^+$-N)	38.6 ± 8.09	--	--	--	--	18	mg/l
NO_3-N	0.67 ± 0.42	--	--	--	0.165 ± 0.135	--	mg/l
TP	1.36 ± 0.45	20	10	6	2.23 ± 1.2	1.3	mg/l
TSS	280.3 ± 91.7	350	200	100	887 ± 375	148	mg/l

Note: (--) Data not available, (COD) Chemical oxygen demand, (DOC) Dissolved oxygen carbon, (BOD) Biological oxygen demand, (TN) Total nitrogen, ($NH4^+$-N) Ammonical nitrogen, (NO_3–N) Nitrate-nitrogen, (TP) Total Phosphorus, (TSS) Total Suspended Solids.

Table 1

Reference	Cycle time (h)	Effective Reactor Volume (L)	COD			TN			TP			TSS		
			INLET (mg/L)	OUTLET (mg/L)	Removal (%)	INLET (mg/L)	OUTLET (mg/L)	Removal (%)	INLET (mg/L)	OUTLET (mg/L)	Removal (%)	INLET (mg/L)	OUTLET (mg/L)	Removal (%)
This study	12	4250	141.3 ± 31.2	24. 2 ± 9.4	82.2 ± 8.5	39.4 ± 4.3	21.5 ± 3.1	45.0 ± 8.6	1.6 ± 0.2	0.9 ± 0.1	45.2 ± 11.4	317.2 ± 113.3	53.6 ± 18.4	82.3 ± 5.1
	8		125.7 ± 29.7	27.0 ± 5.5	77.9 ± 5.1	43.8 ± 7.3	24.23 ± 6.0	43.7 ± 15.1	1.7 ± 0.3	0.9 ± 0.1	42.8 ± 10.7	262.2 ± 71.2	58.6 ± 22.7	77.8 ± 5.1
	6		136.1± 23.4	31.6 ± 5.0	76.0 ± 7.5	42.9 ± 6.7	26.8 ± 5.5	37.3 ± 9.6	1.4 ± 0.2	0.8 ± 0.1	40.3 ± 10.9	281.5 ± 95.6	63.7 ± 16.4	76.1 ± 6.6
	4		138.8 ± 20.1	49.3 ± 10.4	63.6 ± 9.6	28.3 ± 3.9	18.7 ± 2.6	33.7 ± 7.5	0.7 ± 0.3	0.4 ± 0.1	43.9 ± 7.4	282.8 ± 92.2	64.8 ± 24.1	74.2 ± 13.6
(Jakubaszek & Stadnik, 2019)	6 h	3500	910	116.3	87.6	56	13	77	11.8	4.5	61.1	454	27.5	94.5
(Lin & Cheng, 2001)	12h	50	375	24	93.6	--	--	--	4.6	2.9	37	67.1	6.2	90.8
(Debik & Manav, 2010))	8h	4	545	50	91	57 (TKN)	12 (TKN)	78 (TKN)	12	1.6	87	--	--	--
(Pelaz, Gómez, Letona, Garralón, & Fdz-Polanco, 2018)	6h	6	273	63	77	90.6	14.6	84	--	--	--	--	--	--

(Alagha et al., 2020)	14h	240	359	28	91	21	3.13	83	4.57	0.48	90	1737	14	99

Note: (--) Data not available, (COD) Chemical oxygen demand, (TN) Total nitrogen, (TP) Total Phosphorus, (TSS) Total Suspended Solids.

Table 2

Cycle (hr)	INLET DOC (mg/L)	INLET VSS-C (mg/L)	OUTLET DOC (mg/L)	OUTLET VSS (mg/L)	ΔC (mg/L)	% C converted to CO_2
12	125.53	110.17	62.77	19.15	153.78	65.25
8	136.53	93.95	67.14	20.07	143.27	62.16
6	133.55	102.14	74.23	23.33	138.1	58.61
4	102.00	91.01	59.01	24.80	109.2	56.58

Note: (DOC) Dissolved oxygen carbon, (VSS) Volatile suspended solids

Table 3

Parameter	SBR Inlet	SBR Outlet	ECR Outlet	% Removal after SBR treatment	% Overall removal	Standards for effluent discharge from STP's (Central Pollution Control Board (CPCB), 2015)
COD (mg/L)	162.00	74.00	17.00	54.32	89.51	50.00
DOC (mg/L)	17.41	9.91	5.69	43.08	67.32	--
TN (mg/L)	27.79	18.13	10.78	34.76	61.21	10.00
TP (mg/L)	1.76	0.94	0.00	46.59	99.93	1.00
TSS (mg/L)	282.80	64.80	0.00	77.09	100.00	20.00
VSS (mg/L)	171.40	46.70	0.00	72.75	100.00	--
pH	7.66	8.11	8.87	--	--	6.50-9.00

Note: a- CPCB, (2015); STP- Sewage treatment plant; (--) Not available.

Table 4

Name of the Compound	Type of Compound	Present Study						Previous Study (From literature)			
		SBR Inlet abundance	SBR Outlet abundance	ECR Outlet abundance	% Removal SBR	% Removal ECR	Overall % Removal	Type of Wastewater	Type of Treatment	Overall % Removal	Reference
10-Hydroxycarbazepine	Anticonvulsant Drug, anthropogenic marker	5324	788	BDL	85	100	100	Synthetic wastewater	MBR	68	(Hai, Li, Price, & Nghiem, 2011)
2,6-Xylidine (Lidocaine-M) (Dimethylaniline)	Anasthetic treatment	37160	BDL	BDL	100	100	100	Raw sewage	WWTP	50	(Rúa-Gómez & Püttmann, 2012)
Cyclophosphamide	chemotherapy Drug	492221	BDL	BDL	100	100	100	Hospital wastewater	Attached biomass growth reactor	59 ± 15	(Česen et al., 2015);
Diazepam	Anxiety disorder treatment	11496	5743	2654	50	54	77	Raw Sewage	Full-scale MBR	24-47	(Trinh et al., 2012)
Duloxetine	Anxiety disorder treatment	5053	1933	BDL	62	100	100	Spiked Distilled water	Photo degradation (UV irradiation) and chlorination	100	(Osawa, Carvalho, Monteiro, Oliveira, & Florêncio, 2019)
Pentoxifylline	Muscle aching reliever	2344	BDL	BDL	100	100	100	Pentoxifylline in tap water	Non-thermal plasma treatment	92	(Magureanu et al., 2010)
Phendimetrazine	Anti-obesity drugs	59307	22519	BDL	62	100	100	Raw wastewater	WWTP	--	(Kim & Oh, 2020)
Tolfenamic acid	Anti-inflammatory drug	61790	25674	BDL	58	100	100	Spiked with De-ionized water	Direct photolysis and the UV-ABC/H2O2 process	100	(de Melo da Silva et al., 2016)
Irbesartan	Hypertension treatment	1780	856	BDL	52	100	100	Wastewater	Lab-scale sewage plants	36	(Bayer et al., 2014)
Guaifenesin	Expectorant	5006	BDL	BDL	100	100	100	Spiked with Distilled water	UV and visible radiation	48	(da Silva, Lansarin, Livotto, & dos Santos, 2015)
Atraton	Pesticide	2859	BDL	BDL	100	100	100	Lake water	RO	92	(Comerton, Andrews, Bagley, & Hao, 2008)
o-Toluidine	Herbicide and Dye manufacturing	6101	1555	BDL	75	100	100	petrochemical wastewater	Ozonation and O3/TiO2 processes	89.5 and 96	(Shokri & Mahanpoor, 2017)

Ethofumesate	Herbicide	58754	BDL	BDL	100	100	100	agriculturally dominated landscapes water	WWTP	--	(Münze et al., 2017)
Diethofencarb	Fungicide	17317	6998	2794	60	60	84	water and fruit juice samples	--	--	(Cheng, Xia, Zhou, Guo, & Chen, 2011)
Oxadixyl	Fungicide	2780	BDL	BDL	100	100	100	Aqueous solution	Biomass Treatment	--	(Saiano & Ciofalo, 2007)
Pirimicarb	Insecticide	87252	36686	BDL	58	100	100	Environmental water samples	Photocatalysis	97.6	(Wu et al., 2016)
Propetamphos	Insecticide	10208	3049	1267	70	58	88	--	--	--	--
Disulfoton-sulfone	Pesticide	45057	BDL	BDL	100	100	100	--	--	--	--
Propetamphos	Pesticide	10207	3048	BDL	70	100	100	--	--	--	--

Note: BDL- Below detectable level; MBR- Membrane Bio-Reactor, WWTP- Wastewater treatment plant; RO- Reverse Osmosis, (--) Not available.

Table 5

Captions for Figures

Fig 1: Schematic of the reactor (a) SBR system (b) ECR system

Fig 2: Inlet and outlet wastewater characteristics and pollutant removals during different cycles of SBR operations (a) Total COD (b) Soluble COD (c) DOC (d) TN (e) TP and (f) TSS

Fig 3: Response Surface plots for COD, DOC, TN and TP removal at different pH, current and retention times. (a)-(c) COD (d)-(f) DOC (g)-(i) TN (j)-(l) TP

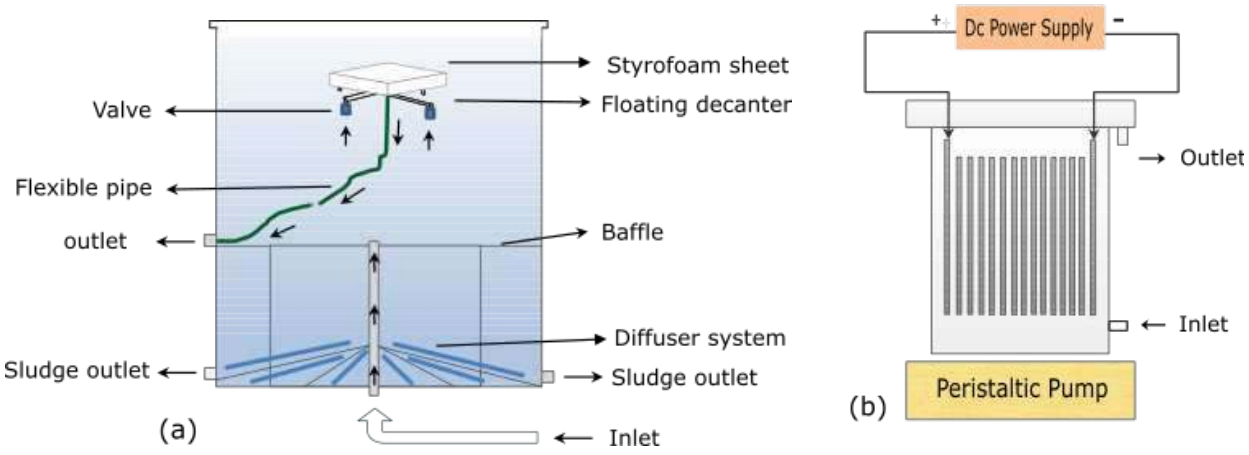

Fig 1

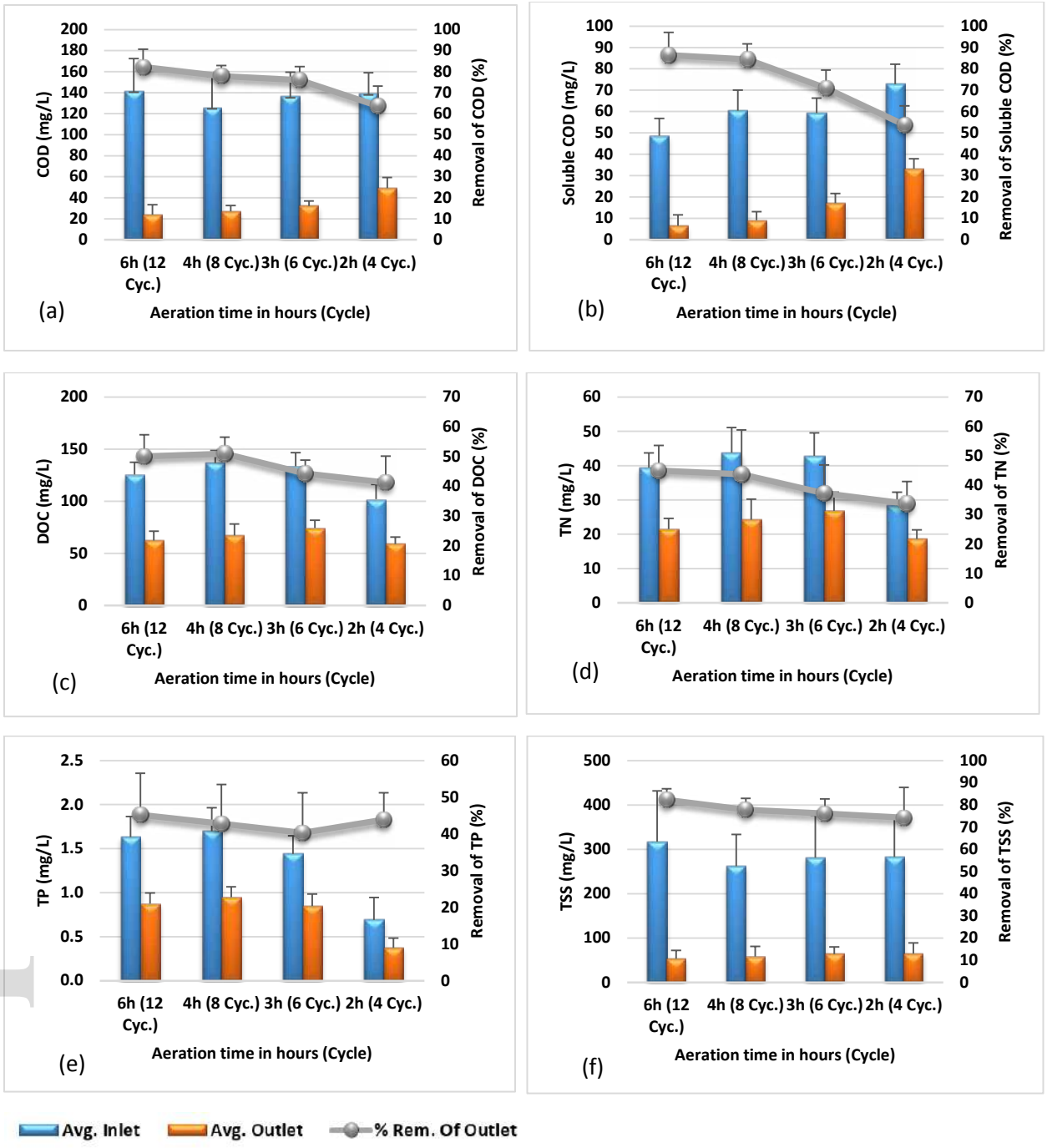

Fig 2

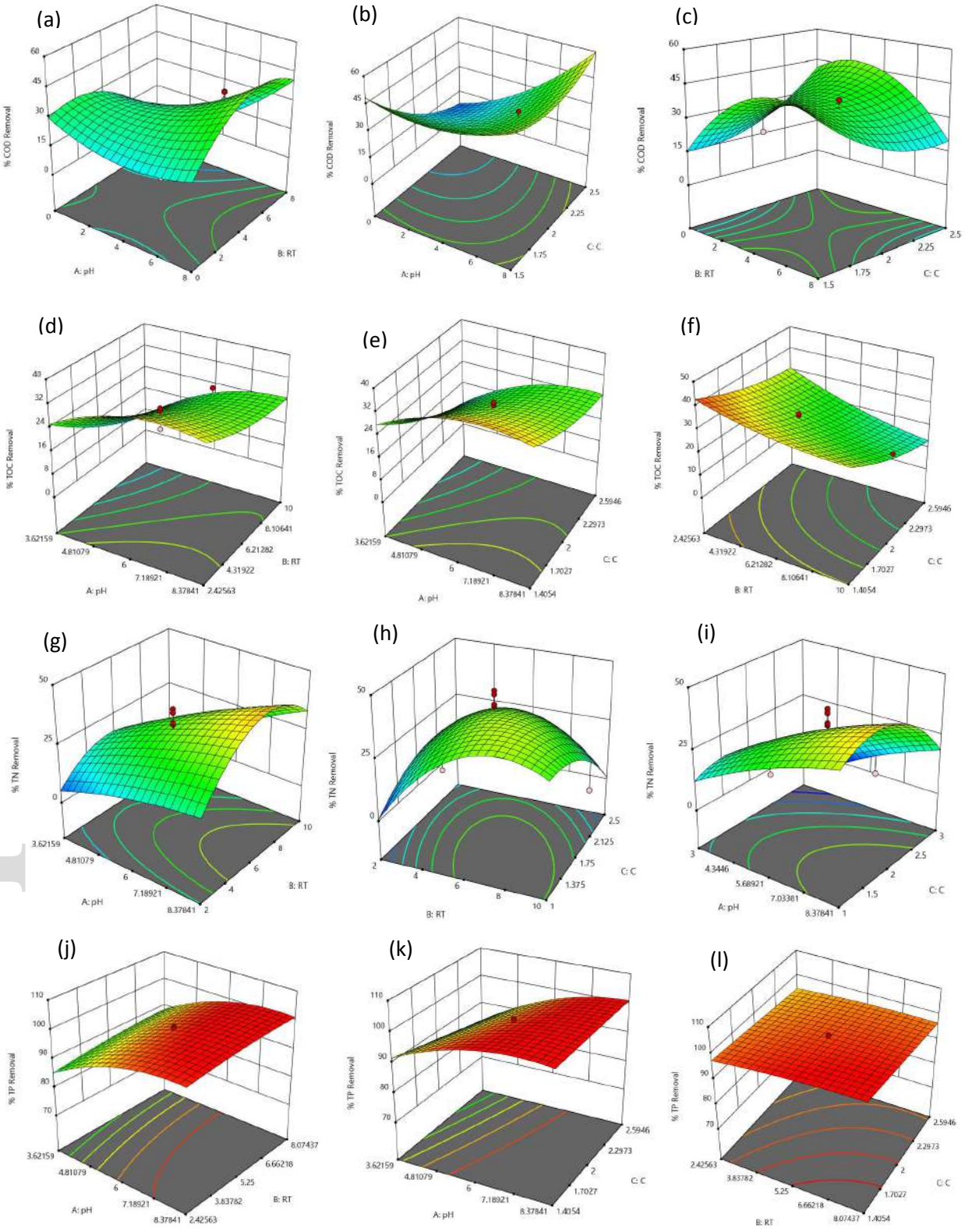

Fig 3