

Wastewater Treatment in a Sequential SBR-ECR Process

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1. Introduction

On an average, put together, about 72,368 million liters per day (MLD) of wastewater is produced in Class I cities (population of over 1,00,000) and Class II towns (population between 50,000 and 99,999) of India. Of those, only about 26,869 MLD receive some level of treatment [1]. The rest, be it industrial or domestic wastewater, is released into the receiving water bodies without any treatment, thereby causing water pollution. There is thus a significant gap between wastewater generation and its treatment in India. It is essential to treat wastewater to an extent such that the treated water can be reused at least for non-potable purposes, thereby, preserving freshwater reserves.

The degree of industrialization is usually considered the most accurate indication of a country's advancement. Agro-based industries that directly or indirectly connect to agriculture have long been seen as a guarantee of the initial stage of development [2]. Of the total large and medium scale industries in our country, about 45% of them use agricultural products as their raw material. According to one study, in many parts of the world, especially in agrarian countries such as India, rapid development in agro-based industries poses a danger to water quality and creates stress on water supplies [3]. The *State of Indian Agriculture Report 2015-16* indicates that agriculture and related sectors will continue to play a vital role in the growth and development of the Indian economy [4]. Agro-based industries usually consume a large amount of water and simultaneously generates enormous quantity of effluent. This has made the agro-based industry the second-largest source of water pollution, ranking next only to domestic wastewater. Effluents from agro-based industries contain high amounts of chemical and biological oxygen demand (BOD), solids, phosphorus, and nitrogen. Their disposal without treatment causes environmental problems [5]. However, agro-based industrial effluents, being less toxic (unlike those originating from pharmaceutical, textile, dye, and pesticide industries), can be reused if adequately treated, and renewable energy can be generated as a byproduct [6].

Several treatment methods, including biological, chemical, and physical processes, are being used to treat wastewater on both small and large scales. Sequential Batch Reactor (SBR) is one such biological treatment system that is used extensively in treating domestic, agricultural and industrial wastewaters. An SBR reactor is a one-tank system that is operated in batch (fill-and-draw) mode. The reactor is quite effective in treating organic wastewater. However, it is still a challenge to meet water reuse guidelines due to the continued presence of recalcitrant compounds and emerging micropollutants, which are persistent and difficult to degrade biologically [7]. 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. EC strategies are suitable alternatives for removing recalcitrant compounds and supplements present in wastewater because of their high expulsion proficiency, simple process, and low treatment costs [8].

Both SBR and ECR are effective techniques for the treatment of different wastewaters. In this study, Domestic Wastewater (DWW), Marigold flower processing wastewater (MWW), and Palm oil mill effluent (POME) have been identified as highly polluted effluents that are loaded with organic carbon, nutrients, and recalcitrant organics. In order that the treated effluents meet the regulations for effluent reuse, we proposed wastewater treatment in a modified sequencing batch reactor (SBR) and polishing treatment in a continuous bipolar-mode electrochemical reactor (ECR) as two sequential processes for Zero-liquid Discharge (ZLD) treatment systems.

The modified SBR has several improved features that make sample collection and operation & maintenance of the reactor easy (**Figure 1(a)**). Two types of floating decanter configurations were used – (a) a spring-based decanter (OT-S) and (b) a membrane-based decanter (OT-M). The ECR (**Figure 1 (b)**), which has a series of sacrificial electrodes inserted between the two end electrodes (which are connected to the power supply), requires less footprint, reaction time, and energy compared to monopolar mode electrochemical reactors for the same degree of wastewater treatment. The modified SBR was earlier used to successfully polish an anaerobically-treated dye effluent at a laboratory scale [9]. The ECR was used to treat anaerobically-digested distillery effluent and palm oil mill effluent in the past [10].

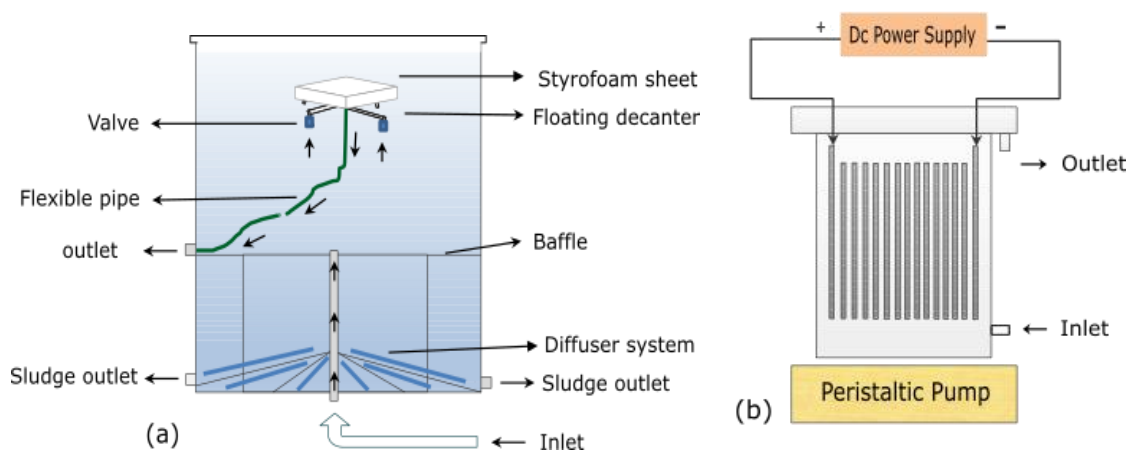


Figure 1. Schematic of the reactor (a) SBR system (b) ECR system

2. Objectives and scope of the work

In this research, a sequential SBR-ECR system has been developed to address the above-mentioned issues, namely, zero-liquid discharge, water reuse, removal of emerging contaminants, and the recovery of materials in useful forms. A stand-alone SBR-ECR system cannot, by itself, achieve the above objectives. This SBR-ECR combination is expected to form an important component of a ZLD system in which a series of unit operations and processes are put to use for stage-wise removal pollutants from water. The specific objectives and scope of the work are

1. Design and fabrication of a novel Sequential Batch Reactor.

2. Performance evaluation of the reactor using domestic wastewater and effluents from marigold flower processing and palm oil processing industries.
3. Integration of Continuous Bipolar-mode Electrocoagulation System and process optimization of the integrated system.
4. Micro-pollutant analysis of wastewaters before and after SBR and ECR treatment
5. Studying kinetics of biodegradation.
6. Sludge characterization, preparation of sludge-based adsorbent, and studying its performance characteristics.

3. Organization of the thesis

The thesis has been organized into six chapters.

Chapter 1. Introduction and Review of Literature

This chapter provides a brief introduction to the need for the development of coupled SBR-ECR system. It contains an overview of wastewater treatment, the theoretical concept of SBR and ECR reactors and their applications in wastewater treatment, mechanisms of pollutant removal, the major reactions that happen at the electrodes in ECR, and design parameters considered in various studies in the literature. This chapter also reviews the various configurations of SBR reactors and technologies adopted for the treatment of Domestic Wastewater, Marigold Flower Processing Effluent, and Palm Oil Mill Effluent.

Chapter 2. Materials and Methodology

This chapter describes the different sites where this research was conducted and the existing processes. It includes a detailed discussion on the experimental setup, innovative concepts that have been incorporated into the design of the reactor, description of the process of installation of the same (**Figure 2**), the methodology adopted for the operation of the reactors (**Figure 3**), discussion on process optimization, and analytical methods adopted for the characterization of water and sludge samples.

This research was carried out using three different wastewaters at three different research sites:

1. Domestic wastewater (DWW) from M/s. Patancheru Envirotech Limited, Patancheru, Hyderabad, Telangana. A nearby canal carries sewage from the city of Hyderabad. Domestic wastewater from the canal was used in the first phase of the research.
2. Marigold flower processing wastewater (MWW) from M/s. Synthite Industries, Davanagere District, Karnataka. The effluent from the first stage anaerobic unit was collected and used in the second phase of this research.

3. Palm oil mill Effluent (POME) from M/s. Telangana State Oil Federation, Apparaopeta, Bhadrachalam, Kothagudem, Telangana, The effluent from the first stage anaerobic unit was collected and used in the third phase of this research.

The study at each site was divided into two phases. The experiment in phase-1 was carried out with SBR, and in phase-2, the ECR unit was connected in series when the SBR was operated at the lowest cycle time, that is, at the highest hydraulic and organic loading. The performance evaluation was carried out for both phases based on COD, TN, TP, and TSS removals. An LC-HRMS analysis was carried out to identify the organic compounds in the inlet and treated samples. In phase-2, the electrochemical reactor was used after the SBR system as a polishing step post-biological treatment. The process optimization was carried out using response surface methodology (RSM) to optimize the operational parameters for maximizing the treatment efficiency. Central composite design (CCD) was used for designing the experiment.



Figure 2. Photos of the installation process at one of the research sites

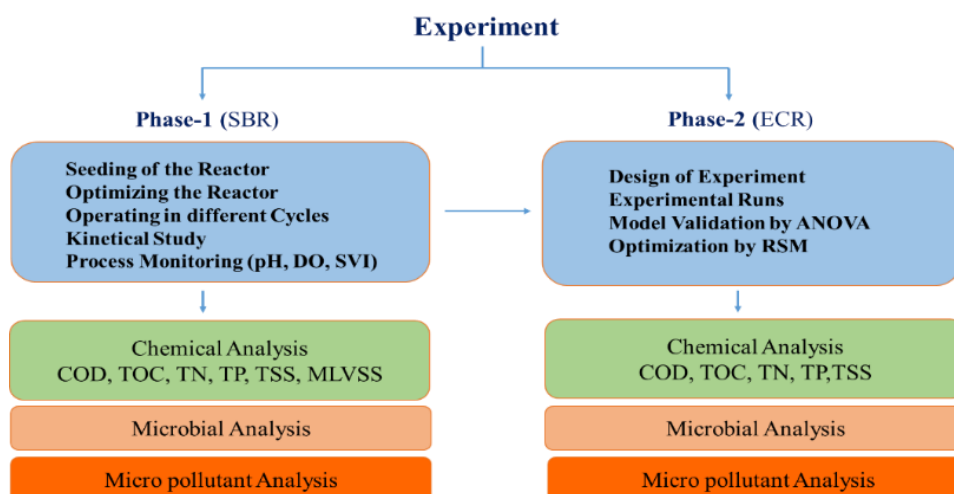


Figure 3. Pictorial representation of the experimental procedure

Chapter 3. Biological treatment of wastewater in an improved Sequential Batch Reactor

This chapter describes the characteristics of the wastewater, before and after biological treatment, and the operating cycles of the SBR. The characteristics of the inlet wastewaters are given in **Table 1**. The process monitoring of the SBR was done based on the parameters including pH, DO, MLSS, and Sludge Volume Index (SVI).

Table 1. Inlet wastewater characteristics for SBR

Parameter	DWW (Avg. Value)	MWW (Avg. Value)	POME (Avg. Value)	Unit of measurement
pH	7.7 ± 0.35	8.33 ± 0.18	7.50 ± 0.21	-
Color	Brownish	Dark Brown	Dark Brown	-
COD	135.9 ± 27.2	5750 ± 991	4899 ± 1599	mg/L
DOC	104.6 ± 49.7	980 ± 120	1160 ± 208	mg/L
T-N	38.6 ± 8.09	692 ± 60	506 ± 81	mg/L
Nitrate-N	0.67 ± 0.42	1.1 ± 0.3	1.2 ± 0.6	mg/L
Total phosphorous	1.36 ± 0.45	9.7 ± 1.1	312 ± 65	mg/L
Suspended solids	280.3 ± 91.7	1144 ± 166	11120 ± 1766	mg/L

The SBR was operated at four cycles and the performance was evaluated based on organic loading rate (OLR), food to microorganism ratio (F/M ratio), COD, TN, TP, and TSS removals for all three wastewaters. The effectiveness of two decanting mechanisms employed in this research was compared. **Table 2** shows the average values of the effluent parameters when decanter OT-S was used. The COD, TN, and TP removals for DWW were between 64-82%, 34-45%, and 40-45%, respectively; for MWW between 32-71%, 17-32%, and 40-52%, respectively; for POME between 36-81%, 20-52%, and 35-39% respectively, depending on the cycle duration. **Table 3** also shows the average values of the effluent parameters when membrane-based decanter (OT-M) was used. The COD, TN, and TP removals for DWW were between 82-95%, 36-44%, and 49-53% respectively; for MWW between 51-83%, 30-57%, and 52-78%; for POME between 52-86%, 37-71%, 45-50%, respectively. OT-M performed better as the membrane has a sieve effect, which can cut off colloidal substances and suspended particles [11]. Performance reduced at lower cycle times because of the lower availability of aeration time [12]. The performance of SBR was found to be good in higher HRT. It displayed a reduction in removal efficiency with decreased HRT or increased OLR.

When decanted through OT-S, the effluent TSS was found in the range of 54-65 mg/L for DWW, 129-153 mg/L for MWW, and 478-865 mg/L for POME, whereas TSS was completely removed when OT-M was used for all wastewaters. The length of the withdrawal period was strongly affected by the SBR effluent quality, especially in TSS removal with OT-S [13].

LC-HRMS analysis was carried out to screen the organic compounds in the wastewaters, and pathogen analysis was carried out to know the removal of pathogenic bacteria. LC-HRMS analysis of DWW indicated that ten out of nineteen micropollutants observed in this study were pharmaceutically active ingredients. The remaining nine compounds belonged to the pesticide family. The agro-based wastewaters, MWW and POME,

contained plant metabolites, herbicides, fungicides, and pesticides. The majority of the compounds were removed completely with the OT-M decanter, whereas the OT-S decanter could remove them partially. The material balance was performed to determine the percentage of inlet carbon converted to carbon dioxide, and kinetic studies were performed to determine degradation rates for all four-cycle timings for effluents.

Chapter 4. Polishing of biologically treated wastewater in an Electrochemical Reactor

This chapter describes the study of ECR treatment of the effluents after being treated in the SBR. The ECR was connected in series with the SBR when the SBR was operated at the lowest cycle time. The experiment was designed using CCD, and RSM was used to investigate the relationships between the dependent variable (pollutant removal) and the independent variables (operating parameters) and to optimize the process. pH (X_1), retention time (X_2 , min), and current (X_3 , amp) were chosen as independent process variables, while COD, DOC, TN, and TP removal efficiency were chosen as dependent variables. A total of 20 experimental runs were performed in duplicates, including 2^k factorial points, 2k axial points, and six central points following CCD ($k = 3$). The electrode area was kept constant at the highest limit of 765 sq cm for this study. Iron electrodes were used for the study. The response optimization and data processing were performed using statistical software Design Expert 11.1.2.0. High R-square values were obtained using the RSM technique.

The effluent from the SBR was fed to the ECR in a continuous mode of operation. Optimization study showed that at the optimal point, the COD, TN, and TP removal were above 90%, 67%, 61%, and 99% for DWW, 79%, 53%, and 99% for MWW, and 89%, 71% and 94% for POME, respectively. **Figure 4** represents surface plots of the MWW optimization study. The graphical representation of response surface plots and contour plots helped in understanding the interactions between the variables. Suspended solids were reduced to a level below the detection limit in all three wastewaters, which is shown in **Table 3**. LC-HRMS analysis was carried out to screen the organic compounds in the wastewaters, and pathogen analysis was carried out to know the presence of pathogenic bacteria. Most micropollutants were completely eliminated in the sequential SBR-ECR process. The SBR alone was ineffective in removing the pathogens, whereas the coupled system completely removed *Salmonella*, *Pseudomonas*, and *Staphylococcus aureus*. During electrochemical reactions, the charge on the cell surface is neutralized in the presence of $\text{Fe}^{2+}/\text{Fe}^{3+}$. This helps in bacterial floc formation and eventual precipitation along with the metal hydroxides. The final effluent still contains a considerable amount of COD in the case of MWW and POME, primarily in the soluble form. Therefore, MWW and POME need a tertiary treatment like ozonation and reverse osmosis for generating reusable water.

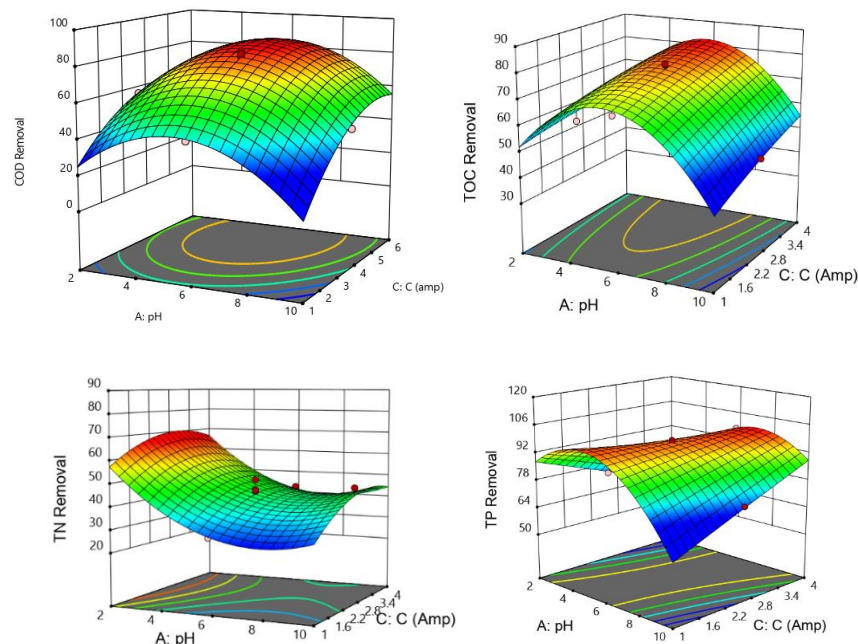


Figure 4. Response surface plots for the COD, DOC, TN, and TP removals for Marigold wastewater

Table 2. Outlet wastewater characteristics for SBR treatment

	Cycle time (h)	COD (mg/L)		TN (mg/L)		TP (mg/L)		Effluent SS (mg/L)	
		OT-S	OT-M	OT-S	OT-M	OT-S	OT-M	OT-S	OT-M
DWW	SBR (12 h)	24.2 ± 9.4	7.0 ± 5.8	21.5 ± 3.1	21.9 ± 2.2	0.9 ± 0.1	0.7 ± 0.1	53.6 ± 18.4	BDL
	SBR (8 h)	27.0 ± 5.5	8.4 ± 3.6	24.23 ± 6.0	24.3 ± 5.6	0.9 ± 0.1	0.9 ± 0.1	58.6 ± 22.7	BDL
	SBR (6h)	31.6 ± 5.0	11.8 ± 3.6	26.8 ± 5.5	26.9 ± 5.2	0.8 ± 0.1	0.8 ± 0.1	63.7 ± 16.4	BDL
	SBR (4 h)	49.3 ± 10.4	24.7 ± 5.8	18.7 ± 2.6	18.0 ± 2.4	0.4 ± 0.1	0.3 ± 0.1	64.8 ± 24.1	BDL
	After ECR	17.0	--	11	--	BDL	--	BDL	BDL
MWW	SBR (24 h)	1638 ± 231	971 ± 185	481 ± 34	478 ± 39	4.9 ± 0.5	4.8 ± 0.7	133 ± 26	BDL
	SBR 16 h)	1977 ± 436	1136 ± 282	441 ± 76	435 ± 74	4.8 ± 0.8	4.7 ± 1.2	153 ± 25	BDL
	SBR (12h)	3491 ± 303	2189 ± 345	587 ± 43	585 ± 49	5.2 ± 1	5.1 ± 1.1	130 ± 29	BDL
	SBR (8 h)	3472 ± 301	2502 ± 265	573 ± 35	575 ± 28	5.4 ± 0.9	5.3 ± 0.7	129 ± 35	BDL
	After ECR	1080	--	316	--	0.12	--	BDL	BDL
POME	SBR (48)	1129 ± 226	841 ± 164	237 ± 29	243 ± 19	210 ± 19	200 ± 16	478 ± 199	BDL
	SBR (36)	1707 ± 329	1240 ± 228	387 ± 48	373 ± 42	230 ± 20	224 ± 27	631 ± 167	BDL
	SBR (24)	2491 ± 433	1614 ± 302	402 ± 44	397 ± 53	199 ± 19	189 ± 18	820 ± 118	BDL
	SBR (12)	1551 ± 203	1162 ± 136	321 ± 46	324 ± 59	155 ± 24	149 ± 27	865 ± 99	BDL
	After ECR	240	--	110	--	19	--	BDL	

*BDL-Below Detection Limit

Chapter 5. Sludge Characterization and management

This chapter describes the sludge characterization studies and the conversion of sludge into a useful byproduct (activated carbon) in order to make the SBR-ECR combination a zero-waste treatment process. Sludge characterization and management is an important component in wastewater treatment. The sludge characterization helps to understand the mechanism of pollutant removal and to propose suitable management techniques including possible resource recovery and other valuable applications. XRD analysis was done to understand the mechanism involved in the process of removal of pollutants. The presence of compounds such

as phosphoric acid (H_3PO_4), iron hydroxide ($\text{Fe}(\text{OH})_3$) indicates the adsorption mechanism, and the presence of iron phosphate (FePO_4) indicates the precipitation mechanism. The analysis confirmed that in all SBR sludge samples, adsorption was the predominant mechanism. In the case of ECR sludge samples, chemical precipitation dominated. XRD analysis revealed no crystalline phase in the identified peaks in any of the samples. Few peaks in the FTIR spectra disappeared after ECR treatment which indicated the removal of organic matter while other peaks were attribute to the byproducts generated during the electrocoagulation process. This peak in the ECR sludge confirmed the formation of ferrous ion flocs during the electrocoagulation process. SEM images revealed that the SBR and ECR samples exhibited particles of random size and shape. When the image before and after ECR treatment was compared, some elements disappeared, and some decreased drastically. The iron content in the SBR sludge was 1.2%, 1.1%, and 1.7% for DWW, MWW, and POME, respectively. The corresponding values in the ECR sludge were 81.4%, 68.2%, and 48.5%. This increase was due to the use of iron electrodes in the ECR process. The SEM-EDX images of SBR sludge and ECR sludge for Marigold wastewater have been given in the **Figure 5**. The EDS results are in line with FTIR and XRD analysis. These analyses confirmed the removal of organic matter and the involvement of adsorption mechanism and chemical precipitation reaction during the SBR and ECR process. The SEM-EDS data shows the presence of potassium, silica, magnesium, etc. Therefore, the possibility of recovering these components in a cost-effective manner can be explored.

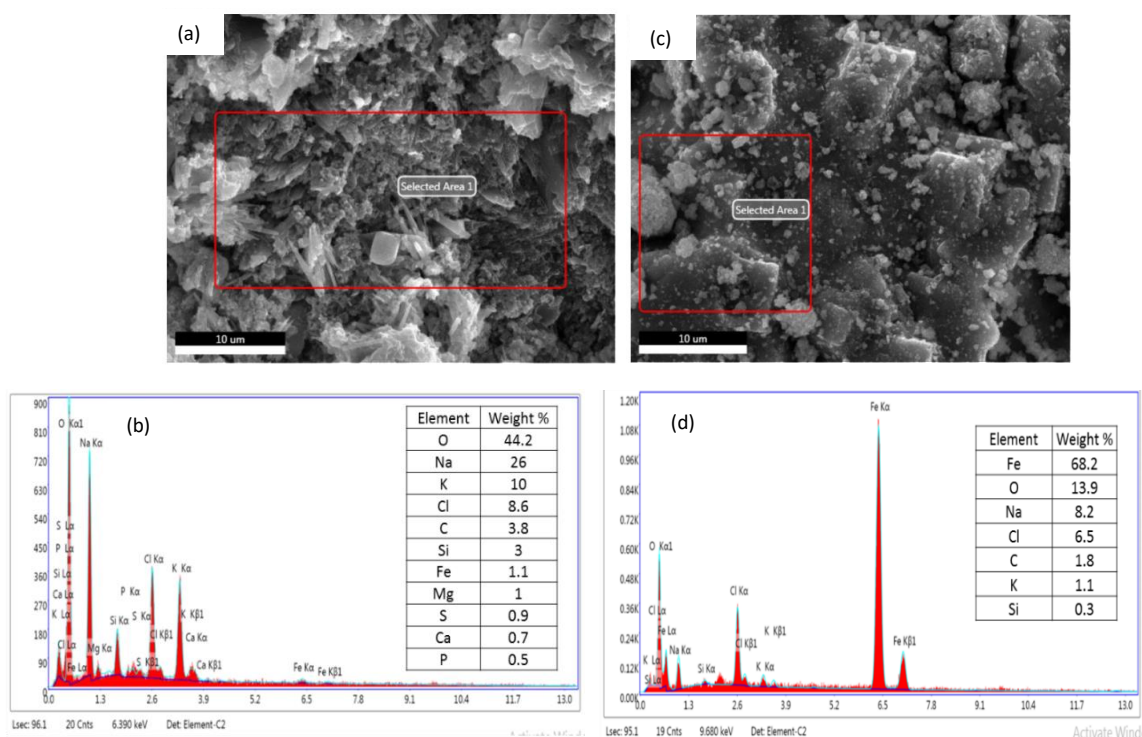


Figure 5. SEM-EDX images of (a-b) SBR sludge (c-d) ECR sludge for Marigold wastewater

The sludge obtained from the SBR during the treatment of POME effluent was used as the precursor material for activated carbon synthesis. The iron-rich sludge generated during the ECR process was used as the activating agent. This powdered mixture was pyrolyzed at a high temperature to synthesize sludge-derived

activated carbon (AC). Chemical activation of SBR sludge with Fe- rich Electrocoagulation (ECR) sludge yielded an activated carbon with good surface area (around 99.77 m²/g). The sludge and AC samples characterization were carried out using FTIR, XRD, SEM, and BET. The adsorption studies were conducted with sludge-based AC for color removal. Methylene blue was used as a model pollutant. Adsorption studies followed the Langmuir isotherm model, indicating multilayer adsorption on a homogenous surface irreversible adsorption. Adsorption kinetics followed pseudo-second-order kinetics.

Chapter 6. Conclusion

This chapter highlights the major outcome of this research and provides an overview of potential future research direction. According to this study, the SBR-ECR system is an excellent technology for removing recalcitrant chemicals and organic constituents from wastewater. The sequential process has the potential to be a part of a ZLD system. It can be stated that the SBR-ECR combination was able to reduce the time of treatment significantly. At optimized conditions, COD, TN, and TP removal were observed to be 90%, 61%, and 99%, respectively for DWW, 79%, 53%, and 99% for MWW and 89%, 71% and 94% for POME respectively. It was also observed that the anaerobically digested effluent contained a significant amount of color. In the SBR-ECR treatment, the polishing treatment can effectively remove color, as shown in **Figure 6**. The hybrid treatment scheme proposed in this study efficiently removed micropollutants and pathogens. The chapter also discussed the significance of sludge characterization in understanding the pollutant removal mechanism and proposing appropriate management techniques. The chapter summarized the potential design parameters (**Table 4&5**) for SBR and ECR reactors, which could be used for designing full-scale systems for treating domestic wastewater and agro-based industrial wastewater. The SBR-ECR setup can also be integrated into containerized wastewater treatment plants to treat effluent from small-and medium-scale industries and in-situ/decentralized treatment of domestic wastewater at the residential community level.

Table 4. Range of parameters suitable for SBR operation

Parameters	DWW	Agro-based wastewater
pH	7-9	7-9
DO	2.0-4.0	2.5-4.0
MLSS/MLVSS (mg/L)	4000/3000 to 6000/4000	6000/4500 to 7000/5000
OLR (kg COD/m ³ /d)	0.2-0.6	3-9
F/M (BOD/g MLVSS. d)	0.15 - 0.4	0.3-0.6
SVI (ml/g)	60-70	60-70
Cycle time (hours)	4-6	12-16
Power (kwh/m ³)	1.8-2.6	7.0-9.0

Table 5. Range of parameters suitable for ECR operation

Parameters	DWW	Agro-based wastewater
pH	6.0-7.0	5.5-6.6
Reaction time (min)	7.0-9.0	4.5-9.0
Surface Area/Volume ratio (m ² /m ³)	76.5	76.5
Energy Consumption (kwh/m ³)	6.8-8.8	69-106
Current (Amp)	1.0- 2.0	3.34-5.44

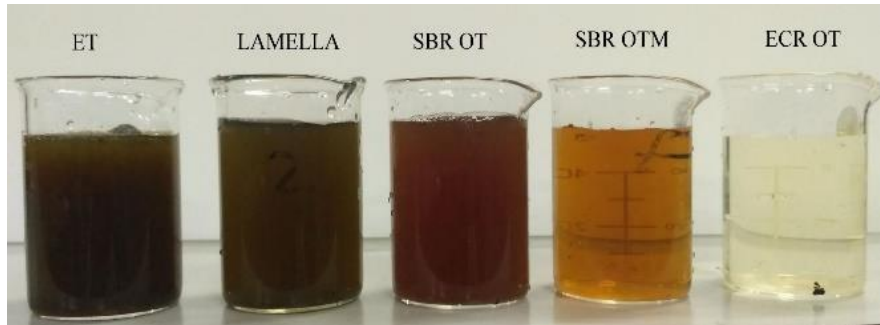


Figure 6. Color of the Marigold wastewater at different stages of treatment

4. List of Publications

4.1 Journals:

- **Akula, L. K.**, Gaddam, V. B., Damaraju, M., Bhattacharyya, D., Kurilla, K. K., “Domestic wastewater treatment in a coupled sequential batch reactor-electrochemical reactor process”. Water Environ. Res. 2020, Volume 93, Issue 6, 1–42. DOI: 10.1002/wer.1488
- **Akula, L. K.**, Oruganti, R.K., Bhattacharyya, D., Kurilla, K. K., “Treatment of Marigold Flower Processing Wastewater using a Sequential Biological-Electrochemical Process”. Journal of Applied Science and Engineering Progress. Vol 14, No 3 (2021), DOI: 10.14416/j.asep.2021.04.001
- **Akula, L. K.**, Bhattacharyya, D., Kurilla, K. K., “Treatment of Palm Oil Mill Effluent (POME) wastewater to reusable level by Novel SBR-ECR”.- (Manuscript under preparation)
- **Akula, L. K.**, Oruganti, R.K., Bhattacharyya, D., Kurilla, K. K., “ Preparation of sludge-based activated carbon from POME SBR sludge impregnated with the ferrous-rich ECR sludge and its adsorption performance for removal of methylene blue”. - (Manuscript under preparation)

4.2 Conferences:

- **Akula, L. K.**, Bhattacharyya, D., Kurilla, K. K. “Treatment of Palm oil Mill Effluent using a Sequential Biological-Electrochemical Process for Reuse”. Conference Name: **2nd International Conference on Advanced Technologies for Industrial Pollution Control (ATIPC-2020)** December 16-18, 2020.
- **Akula, L. K.**, Bhattacharyya, D., Kurilla, K. K., “Treatment of Marigold Flower Processing Wastewater in a Modified Sequencing Batch Reactor followed by a Bipolar-Mode Electrochemical Reactor”. Conference Name: **The 6th International Symposium towards the future of Advanced Researches in Shizuoka University (ISFAR-SU2020)**
- **Akula, L. K.**, Bhattacharyya, “Clear Water Hydrodynamic Behavior and Mixing Pattern in a Novel SAABMBR”. Conference Name: **URBANIZATION CHALLENGES IN EMERGING ECONOMIES ASCE INDIA CONFERENCE 2017.**

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