

Preface

Waste generated in a huge amount during the processing of crude oil into useful products such as petrol, diesel, liquefied petroleum gas (LPG), etc. in petroleum refineries has raised a concern worldwide. The direct discharge of waste in water bodies and land can cause harm to human beings, other living beings and environment. The waste generated is highly toxic and hazardous in nature. Therefore, the treatment of waste generated is necessary before the disposal/reuse. Mainly the two types of waste generated in petroleum refineries namely petroleum refinery effluent and the oily petroleum sludge has been discussed in this thesis. Multiple type of treatment methods are reported in the previous literature studied. The literature mainly reports the use of single type of metal salt for the treatment of effluent. I decided my research objective as the treatment of wastewater collected from the effluent treatment plant by using catalytic thermolysis and coagulation and flocculation process using the CuSO_4 , FeSO_4 , FeCl_3 , and 1:1 ratio (V: V) mixture of CuSO_4 and FeCl_3 as a catalyst and investigation of physicochemical characteristics of petroleum sludge for better remediation and reuse.

The catalytic thermolysis is performed for the treatment of refinery effluent using the CuSO_4 , FeSO_4 , FeCl_3 , and 1:1 ratio (V: V) mixture of CuSO_4 and FeCl_3 as a catalyst. The effects of parameters like temperature, pH, dosage of catalyst, and time are examined for COD, turbidity, and element reduction. The coagulation and flocculation has been experimentally studied for the treatment of refinery effluent by using the CuSO_4 , FeSO_4 , FeCl_3 , and 1:1 ratio (V: V) mixture of CuSO_4 and FeCl_3 as a coagulants. The independent parameters such as pH and dosage of coagulants were optimized using response surface methodology with central composite design technique considering final pH, reduction in COD, turbidity, TDS, and color as dependent variables. The physicochemical characteristics of petroleum sludge before and after Soxhlet

extraction have been investigated. Soxhlet extraction was performed using petroleum ether, hexane, toluene, and benzene as a solvent, among which hexane yields a maximum amount of liquid. The petroleum sludge before and after solvent extraction using hexane is characterized using proximate, ultimate, TGA/DTG/DTA, FTIR, XRD, and SEM-EDX analyses, while liquid extracted from petroleum sludge is characterized by GC-MS analysis.

For the ease of understanding to the readers, the present thesis entitled “Treatment of wastewater and Physico-chemical characterization of Sludge collected from Petroleum refinery” has been divided into six chapters. Chapter 1-Introduction- presents the general overview of the thesis, where the generation and treatment of petroleum refinery effluent and oily petroleum sludge in petroleum refineries has been discussed with the problem statement. Chapter 2-Literature Review- contains the detailed literature review of catalytic thermolysis, coagulation, and flocculation techniques for the treatment of petroleum refinery effluent. Besides, the physico-chemical characteristics of oily petroleum sludge previously studied has been summarized. Chapter 3- Catalytic thermolysis treatment of petroleum refinery wastewater collected from effluent treatment plant, *International Journal of Chemical Reactor Engineering*, 18(5-6), 20190210- describes the catalytic thermolysis of petroleum refinery effluent by using the CuSO_4 , FeSO_4 , FeCl_3 , and 1:1 ratio (V: V) mixture of CuSO_4 and FeCl_3 as a catalyst. The effects of parameters like temperature, pH, dosage of catalyst, and time were examined for COD, turbidity, and element reduction. The precipitated sludge left after catalytic thermolysis is characterized by using SEM-EDX, FTIR, and TGA/DTA techniques. Chapter 4- Pre-treatment of petroleum refinery wastewater by coagulation and flocculation using mixed coagulant: Optimization of process parameters using response surface methodology (RSM), *Journal of Water Process Engineering*, 36 (2020) 101317 – presents the study of coagulation and flocculation process for

the treatment of petroleum refinery effluents by using three coagulants CuSO_4 , FeCl_3 , and $\text{CuSO}_4+\text{FeCl}_3$ (both salt mixed in 1:1 ratio (V/V)). The response surface methodology with central composite design techniques was applied to find the optimum value of the independent variables selected for the analysis. The characteristics of precipitated sludge left after coagulation and flocculation were studied by using XPS, SEM-EDX, FTIR, and TGA/DTA techniques. Chapter 5- Physicochemical characteristics of hazardous sludge from effluent treatment plant of petroleum refinery as feedstock for thermochemical processes, *Journal of Environmental Chemical Engineering*, (2020) 103817- presents the physicochemical characteristics of oily petroleum sludge before and after the Soxhlet extraction using petroleum ether, hexane, toluene, and benzene as a solvent. Chapter 6 - Conclusion and recommendation for future work- presents the summary of the results and some suggestions for further research in this area. References are given at the end of the thesis.