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Catalytic Wet Air Oxidation of Industrial Organic Raffinate over Pt/Al_2O_3 Catalyst

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Abstract—*Catalytic wet air oxidation (CWAO) was performed for the treatment of toxic chemical industrial effluent by using alumina based Pt catalyst. The Pt catalysts were characterized by different analysis techniques such as Scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Transmission electron microscopy (TEM), X-ray diffraction (XRD), and thermo-gravimetric analysis (TGA). CWAO reactions were held in a 4-neck glass reactor at ambient conditions (room temperature (28^{o}C) and atmospheric pressure). Experiments were conducted to investigate the effect of catalyst particle size, different wt % of Pt, catalyst dosage and the air flow rate on COD (Chemical Oxygen Demand) removal. The 1 wt % Pt catalyst was optimized which corresponds to 55 % COD degradation at optimized conditions*.

1. INTRODUCTION

Chemical industry consist of various chemicals like chlor-alkali, inorganic chemicals, organic chemicals, dyestuffs and dye intermediates, agro chemicals and alcohol based chemicals. The effluents from chemical industry have high organic load which contain degradable or non-degradable bio-toxic organics included toxic ammonia, pyridine and β-picoline, which creates nuisance because of their malodorous and pungent smell [1-3]. Pyridine and its derivatives are volatile, toxic and flammable, with a pungent and unpleasant odor. They have very high half-life when released in the atmosphere. It is also mildly toxic by inhalation and exposure of it can cause depression, gastrointestinal upset, liver and kidney damage, nausea, anorexia, frequent urination and dermatitis [1-2].

So the treatment of organic raffinate is very necessary because of the toxic organic compounds and in order to meet the prescribed discharge limit. The main problem in reducing water pollution lies on how to remove toxic organic compounds, which are too concentrated for biological remediation but are too dilute for economical chemical or physical treatment, or incineration. Wet oxidation processes are considered to be attractive for removal of toxic compounds and organic loads in the range of about 10 to 100 g/l [4]. But it is operated at high temperature, high pressure and produce corrosive reaction environment. To reduce the severe temperature and pressure requirements of WAO, a catalyst is required. The use of a catalyst in conjunction with WAO is known as catalytic wet air oxidation (CWAO).

A lot of information is available on using CWAO for the treatment of synthetic solution containing single organic pollutant. But very scanty data is available for the real industrial effluent containing multi-component mixture of organic compounds.

It is proposed to explore the use of CWAO for treatment of industrial effluent. Therefore efforts were made in the present study to explore the potential of alumina based Platinum (Pt) catalyst for the treatment of industrial organic raffinate containing toxic constituents like ammonical nitrogen, pyridine etc.

2. MATERIAL AND METHODS

The industrial organic raffinate was collected from a nearby Chemical industry and was characterized for various parameters as shown in Table 1.

Table 1: Characteristics of organic raffinate

Parameters	Organic Raffinate
pH	8.8
COD (mg/L)	30000
TSS (mg/L)	720
TDS (mg/L)	9800
Turbidity NTU	10.6

The alumina based Pt catalyst was prepared by incipient wetness impregnation method and was characterized for surface morphology, BET surface area analysis.
The experiments were performed in a four neck glass reactor at ambient conditions of temperature and pressure

(atmospheric pressure and ~28^oC room temperature). The effect of operating parameters such as catalyst particle size, Pt loading, catalyst dosage and air flow rate was analyzed in terms of percentage COD removal. Initially the glass reactor was filled with 1 litre effluent and the known amount of catalyst was added to it. Then the air flow rate was started and the samples were withdrawn at regular time intervals. The COD of the samples were determined and analyzed by dichromate method (Open reflux, titrimetric method) (APHA, 1998).

2.1 Calculation

$$\text{COD (mg.l}^{-1}) = \frac{(A - B)\ X\ N}{\text{Vol. of sample (ml)}}\ X\ 8000$$

Where, A = Volume of FAS used for blank, ml
B = Volume of FAS used for sample, ml
N = Normality of FAS
COD removal efficiency, COD (%) was calculated by the following equation:

$$C_r(\%) = \frac{C_o - C_i}{C_o} \times 100$$

Where C_r is COD removal efficiency; C_o is initial COD and C_t is COD at a time t

3. RESULTS AND DISCUSSION

Experiments were conducted to determine the optimum conditions for COD removal efficiency by studying the effect of catalyst particle size, Pt loading, catalyst dosage, air flow rate (Fig. 1-5).

3.1 Catalyst particle size effect:

Three different sizes (50 µm, 100 µm, 500 µm) of the catalyst particles were used and the results are shown in Fig. 1. It can be noticed that maximum degradation was achieved for 50 µm size particles and the COD removal showed a decreasing trend with an increase in the size of the catalyst particles. This may be due to the mass transfer across the film surrounding the catalyst particle which is affected by the external surface area of the catalyst.

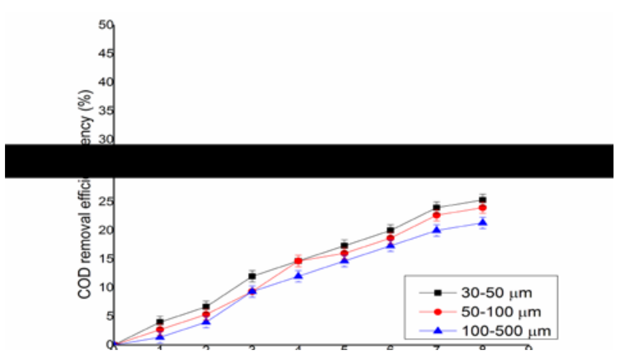

Fig. 1: Effect of catalyst particle size on COD reduction of organic raffinate

3.2 Pt loading effect

Experiments were performed by varying the platinum content (from 0.1 to 1.5 wt %) in the catalyst support and the results are shown in Fig. 2 for Pt/Al_2O_3 catalyst. It can be noticed that there is an increase in COD removal efficiency with an increase in Pt loading from 0.1 to 1 wt%. The maximum degradation is obtained with 1 wt % loading of platinum metal and no increase in COD removal efficiency was observed beyond 1 wt % Pt loading.

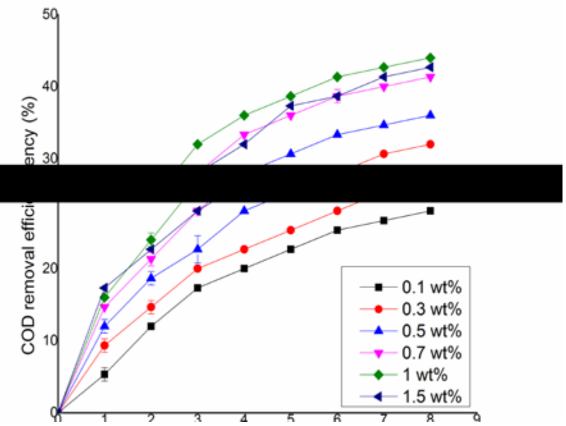

Fig. 2: Effect of Pt loading on COD reduction of organic raffinate at different conditions.

3.3 Catalyst loading effect:

Catalyst loading has significant effects on the COD removal efficiency. Catalytic oxidation was carried out with different catalyst dosage (catalyst amount/volume of solution. The COD removal efficiency increased with an increase in the weight of catalyst per unit volume of solution as shown in Fig. 3. But no increase in COD removal efficiency was observed beyond 3g/l catalyst dosage. Therefore, further experiments were performed with that catalyst dosage.

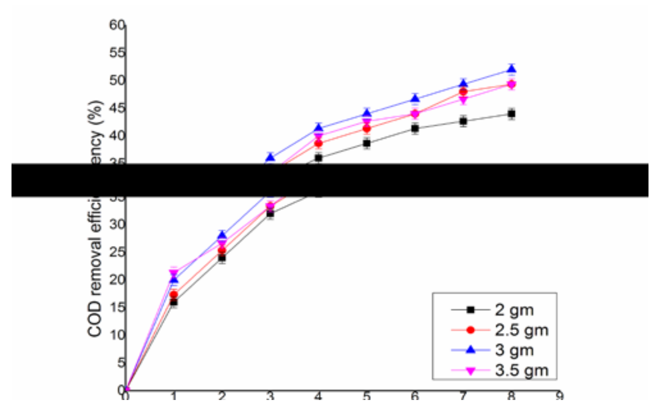

Fig. 3: Effect of catalyst dosage on COD reduction of organic raffinate

3.4 Air flow rate effect

The effect of air flow rate was observed for CWAO experiments of organic raffinate by varying air flow rate from

2 L/min to 4 L/min shown in Fig. 4. It can be noticed that the increase in air flow rate from 2 L/min to 4 L/min did not result in significant increase in the COD removal and 2 L/min air flow rate was optimized for CWAO experiments.

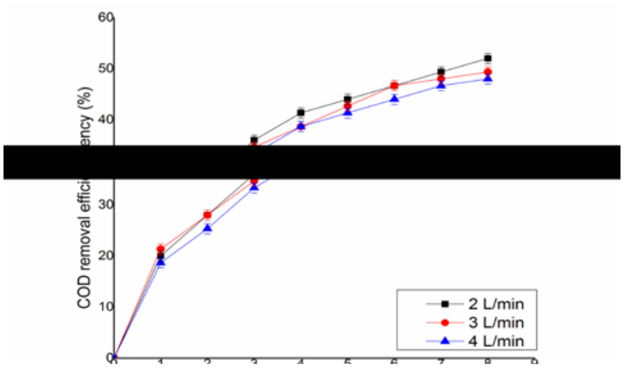

Fig. 4: Effect of air flow rate on COD reduction of organic raffinate at different conditions.

4. CONCLUSION

It can be concluded that catalytic wet air oxidation process has the potential for treatment of industrial organic raffinate which contains toxic organic constituents.

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