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RESEARCH ARTICLE

REMOVAL OF ORGANIC IMPURITIES BY PEROXIDE OXIDATION

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Abstract

Waste sulfuric acid is a big environmental problem in modern world. The amount of waste sulfuric acid produced is huge every year[1]. Many small and medium-sized businesses produce lots of waste acids, but they do not have an appropriate method to treat and recover them. Also due to norms set by pollution control boards across the countries, the waste Sulphuric acid cannot be dumped without proper treatment. This paper focuses on removal of organic impurities in waste Sulphuric acid by using H_2O_2oxidation process. This method is economical and feasible for most small and medium-sized industries / business.

Introduction:-

Sulphuric acid is used for regeneration of resins in DM water plant. However, the raw water in Rasayani Patalganga industrial area is highly contaminated with iron being its major contaminant[2].The high amount of contamination puts load on the resins of DM water plant in all the industries of this area. Storage of 98% sulphuric acid in HDPE containers for longer duration (more than a week) at 40-50°C is not recommended by the manufacturers of HDPE containers. Sulfuric acid is extremely heavy (Sp. Gr = 1.8) and will test the mechanical integrity of the storage container. The inherent weight of sulfuric acid requires a strong material that can withstand the static load pressure constantly pressing against the bottom of the storage tank. Sulfuric acid is an aggressive oxidizer and appropriate safeguards are needed to prevent the container's material from degrading, becoming brittle, and cracking—which could result in leaks or tank failure.The storing temperature of the chemical in HDPE is also very important. It is necessary to regularly verify that the temperature of container never exceeds 100 degrees Fahrenheit (37.7 Deg C). If you know that your container will be subject to higher temperatures, a specially designed tank with a thicker construction is required. In present investigation, the problem of organic impurities in sulphuric acid can be attributed tothe corona lock-down restrictions which resulted in longer than expected storage time of the acid in HDPE containers at moderately high temperatures of summer season (Approx. 40 Deg C) leading to development of colouration in acid and disintegration of the storage container.The reaction between HDPE and Con. Sulphuric acid is demonstrated by following reaction[3].

Due to the colour formation and residue generation in acid, the acid does not comply with acceptance norms. To overcome this problem, peroxide oxidation process was carried out. H_2O_2 is an efficient oxidant. It is widely used to decompose organic residue[4]. Experimental results have indicated that H_2O_2 can decompose organic residue in waste acids.

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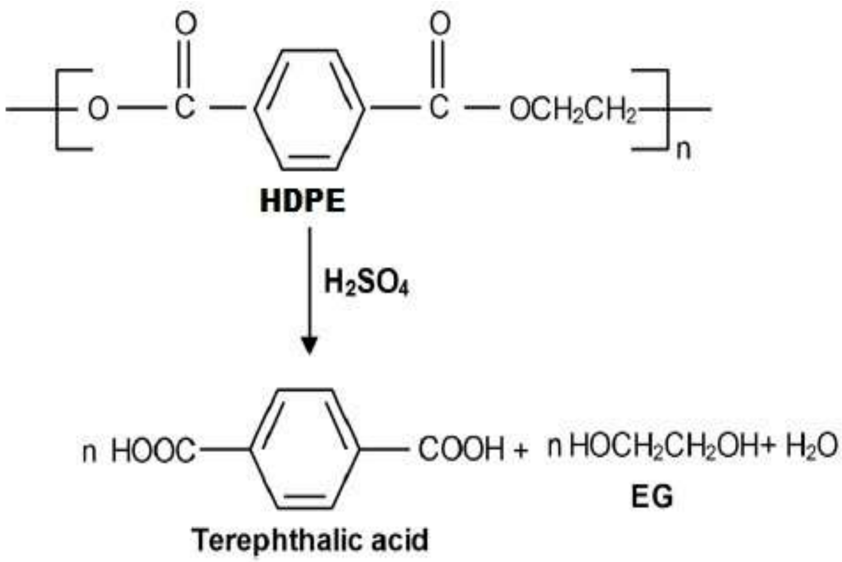

Experimenital

Materials and Methods:-

All the reagents used in this investigation were AR grade. The 98 % sulphuric acid which was delivered in HDPE containers was supplied by M/s Hindustan Petrochemicals, Mumbai. Hydrogen Peroxide (30% W/V) was obtained from M/s Thomas Baker, Mumbai. Classical titration method using 50 ml burette and 3-neck round bottom flask (Borosil) was used to carry out the experiment.

Procedure

100 ml waste sulphuric acid was titrated against varying amount of hydrogen peroxide under cooling and non-cooling conditions. Peroxide was added under cooling and non-cooling conditions until complete decolourization of sulphuric acid was observed. It was observed that when the solution was not cooled, the efficiency of reaction was more i.e., less amount of peroxide was required for decolourization and concentration of resultant acid was also on higher side. The results are discussed further. The setup for theexperiment is given below

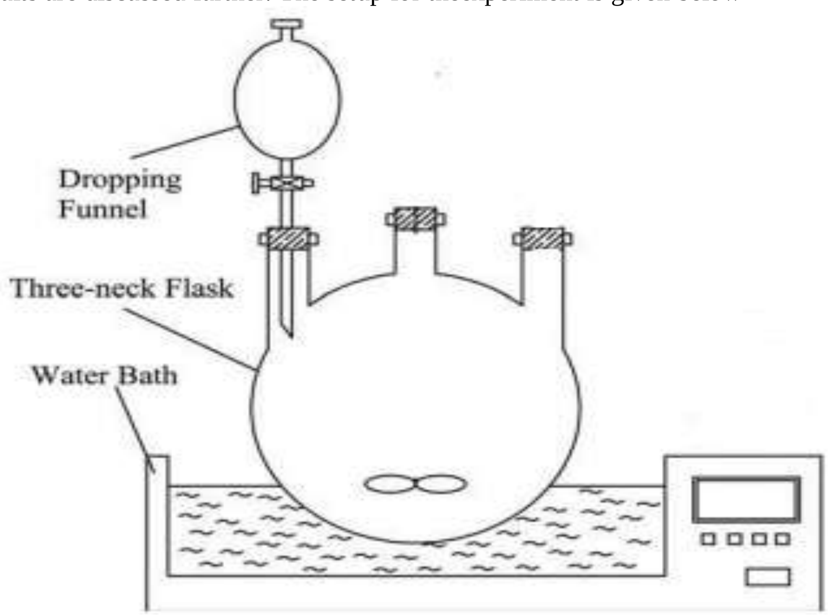

Fig. 1:- Setup for waste Sulphuric Acid Oxidation process.

Results and Discussions:-

Removal of organic impurities in waste sulphuric acid is attributed to the hydroxyl radicals generated upon photolysis of hydrogen peroxide in presence of sunlight[5]. Several studies in the past have proposed different reaction mechanisms for photolysis Of H_2O_2.

The present reaction can be divided into 4 main mechanisms;
1. Initiation mechanism.
2. Propagation mechanism.
3.Termination mechanism.
4. Decomposition mechanism.

This is followed by abstraction of organic impurity resulting in water formation.

Initiation:

K_1

$H_2O_2 + h\nu \longrightarrow 2\,{}^{\bullet}OH$

Propagation:

K_2

$H_2O_2 + {}^{\bullet}OH \longrightarrow {}^{\bullet}O_2H + H_2O_2$

K_3

$H_2O_2 + {}^{\bullet}O_2H \longrightarrow {}^{\bullet}OH + H_2O + O_2$

Termination:

K_4

${}^{\bullet}OH + {}^{\bullet}OH \longrightarrow H_2O_2$

K_5

${}^{\bullet}O_2H + {}^{\bullet}O_2H \longrightarrow H_2O_2 + O_2$

K_6

${}^{\bullet}OH + {}^{\bullet}O_2H \longrightarrow H_2O + O_2$

The powerful oxidizing hydroxyl radicals react with organic molecules, resulting in the removal of impurities.

Decomposition:

$RX + {}^{\bullet}OH \longrightarrow RH$ (Products)

$RX + {}^{\bullet}O_2H \longrightarrow RH$ (Products)

The ${}^{\bullet}OH$ radicals are capable of oxidizing organic compounds by the method of hydrogen abstraction

$RH + {}^{\bullet}OH \longrightarrow R^{\bullet} + H_2O$

Furthermore, **the H_2O_2 process has the additional advantage of preventing any sludge formation during the different stages of the treatment**. It can be carried out under ambient conditions and **leads to complete mineralization of organic carbon into CO_2**.

The experiment carried out can be summarized as per the following table:

Sr. No.	ml of H_2O_2	Cooling (Less than 30 Deg C)		Non-Cooling (60-70 Deg C)	
		H_2SO_4 %	Colour	H_2SO_4 %	Colour

1	5	90.28	Light Brown	92.42	Faint Yellow
2	10	87.62	Yellow	90.12	Colorless
3	20	85.35	Light Yellow	--	--
4	25	84.10	Faint Yellow	--	--
5	30	80.21	Colorless	--	--

Reaction temperature is the main factor influencing the oxidation rate in oxidation systems. In this experiment, it was observed that under non-cooling conditions where the solution temperature was in the range of 60-70 Deg C, the reaction was more vigorous and amount of peroxide required was also on lower side for recovery of acid, i.e., only 10 ml of peroxide was sufficient for recovery. Whereas, under cooling conditions, 30 ml of peroxide is required to achieve complete decolourization.

H_2O_2 was added dropwise and the feed rate of H_2O_2 was controlled by the stopcock. At the end of reaction, the amount of H_2O_2 used was measured as difference between the initial and final readings. During the H_2O_2 oxidation, the color of the waste acid solution changed rapidly. The color changed from black to light brown, yellow and finally to light yellow and clear in case of cooling conditions (less than 30 Deg C) and from black directly to light yellow and to clear in non-cooling condition (60-70 Deg C)

Practical Application
The recovered Sulphuric acidwas tested as per acceptancenorms. Following results were obtained.

Test Conducted	Recovered Acid	Waste Acid
Appearance	Faint Yellow to colorless	Black to Dark Brown
H_2SO_4, % Min	92.42	98.00
Residue on ignition, % max	0.012	0.45
Iron as Fe, ppm max	310	322

As per operation manual for DM water plant, 7.8 % Sulphuric acid (50 kg of 100 % acid in 330 liters of DM water) is required for regeneration of cations in DM water plant. However, using the recovered acid, only 310 liters of DM water will be required resulting in saving of 20 liters of water for each regeneration cycle.

Conclusion:-

This process of H2O2 oxidation could be used to eliminate the organic impurities in waste sulfuric acid. The performance of H2O2 oxidation on waste sulfuric acid was satisfactory andthe optimized parameters for effective oxidation can be summarized as 5 ml of H2O2 per 100 ml of waste Sulphuric acid in non-cooling conditions. The content of sulfuric acid in recovered acid was 92.42 % and the recovered acid could be reused in the original process directly.

Actual Photograph:

Fig.2:- Visual Comparison of waste and lab recovered Sulphuric acid.

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