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RESEARCH ARTICLE OPEN ACCESS

To design industrial protocol on various waste water treatment parameters

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Abstract:

The purpose of this study work is to provide an industrial procedure for testing various wastewater characteristics, with an emphasis on Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD). The research begins with a thorough analysis of current literature in order to gain insights and expertise on these critical water quality evaluation methodologies.

The protocol is meant to include complete methods for both the COD and BOD testing. The COD protocol provides a quick and broad measurement of organic pollution levels, but the BOD protocol gives a more ecologically appropriate evaluation of water quality by measuring the oxygen demand caused by organic matter biodegradation.A case study on the optimisation of COD levels in effluent samples from an ABC Manufacturing Plant is included in the paper. The effectiveness of several oxidising agents in lowering the amounts of organic pollutants in wastewater discharge is investigated.The paper also provides experimental datasets from the use of the Winkler technique to assess the concentrations of dissolved oxygen in pharmaceutical business wastewater. The Winkler technique is used to replace a flawed dissolved oxygen probe, emphasising the need of following standardised procedures for precise and trustworthy findings. This study offers insightful information about the usefulness of COD and BOD tests in real-world settings and highlights their significance in environmental monitoring and water resource management.

Keywords **—CHEMICAL OXYGEN DEMAND(COD), BIOLOGICAL OXYGEN DEMAND (BOD).**

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I. INTRODUCTION

In the modern world, there are severe problems with water contamination and its effects on the ecosystem. Modern wastewater treatment methods are being developed toreduce water pollution and enhance water quality. These technologies works by removing physical, chemical and biological impurities from wastewater. To create treated effluent, a fluid waste stream that is safe for the environment, and treated sludge solid waste. With the aid of increasingly modern technologies, it would even be able to reuse sewage effluent for drinking water. A major risk to the environment and to human health results from untreated wastewater with contaminants getting into surface and ground water supplies industrial wastewater treatments facilities conduct a water quality assessment by reduce the possible dangers from untreated wastewater entering freshwater resources.

There are numerous parameters that can be present in wastewater, depending on the source and composition of the wastewater.

1.2 List of parameters commonly tested in wastewater analysis:

- **Physical parameters:**

1. **Temperature:**

Temperature can be measured using a calibrated thermometer or a temperature probe.

2. **Color:**

Color can be evaluated visually by comparing the wastewater sample to a color reference chart or by using a spectrophotometer to measure the absorbance of light at specific wavelengths.

3. **Turbidity:**

Turbidity is often measured using a turbidimeter, which passes light through the water sample and detects the scattered light. The intensity of the scattered light is then correlated to the turbidity level.

4. **Total Suspended Solids (TSS):**TSS is determined by filtering a known volume of wastewater through a pre-weighed filter, drying the filter to remove moisture, and then reweighing it. The difference in weight before and after filtration represents the TSS concentration, usually reported in milligrams per liter (mg/L) or parts per million (ppm).
5. **Total Dissolved Solids (TDS):**

TDS is determined by evaporating a known volume of wastewater and weighing the residue. The result represents the concentration of dissolved solids in mg/L or ppm.

6. **Settleable Solids:**

Settleable solids are determined by allowing a specific volume of wastewater to settle in a calibrated Imhoff cone or a similar settling device. The settled solids are then reported as a volume or as a percentage of the original sample volume.

- **Chemical parameters:**

1. **pH:**

pH is measured using a pH meter or pH indicator strips. The sample is mixed with a pH-sensitive indicator, and the resulting color change or voltage reading is compared to a calibration curve to determine the pH value.

2. **Chemical Oxygen Demand (COD):**

COD is typically determined through laboratory tests using specific procedures such as the closed reflux method or the open reflux method. These methods involve oxidizing organic and inorganic substances in the sample with a strong oxidizing agent. The amount of oxygen consumed is then measured and reported as COD.

3. **Biochemical Oxygen Demand (BOD):**

BOD is measured by incubating a diluted sample of wastewater in a BOD bottle under controlled conditions (usually at 20°C) for a specified period, typically 5 days. The decrease in dissolved oxygen (DO) concentration during incubation is measured using a DO probe, and the BOD value is reported as the amount of oxygen consumed in mg/L.

4. **Total Organic Carbon (TOC):**

TOC is determined by oxidizing the organic carbon in the sample to carbon dioxide (CO2) using high-temperature combustion or chemical oxidation methods. The resulting CO2 is then quantified using infrared absorption or conductivity measurement techniques.

1.3 Background on oxygen demand testing:

Oxygen demand is an important parameter for determining the amount of organic pollution in water. The test is most frequently used to assess the performance of treatment methods and to evaluate the waste loadings of treatment facilities. Testing water samples from lakes and streams for organic pollution is one of the other uses. Oxygen demand testing assesses the impact of a variety of situations and substances, not the concentration of a particular chemical. Since oxygen demand is not a contaminant, fish or other organisms are not directly threatened by it. However, it can indirectly endanger life by lowering the amount of dissolved oxygen. There are three ways that are frequently used to gauge oxygen demand. Two quantify the direct demand for oxygen: BOD, or biochemical oxygen demand, and COD, or chemical oxygen demand.

1.4 Aim of the project:

The aim of this project is to design industrial protocol on various waste water parameters.

1.5 Objectives:

To review literature and design various parameter protocol;

- Chemical Oxygen Demand protocol
- Biochemical Oxygen Demand protocol

Literature review

Article 1: "Biochemical oxygen demand" volume II Environmental ecological chemistry p 327-330(2015)

This paper demonstrated "Biochemical Oxygen Demand" and it is written by Michael R. Penn, James J. Pauer and James R. Mihelcic.

It provides an overview of biochemical oxygen demand (BOD), which is a measure of the dissolved oxygen consumed by microorganisms during the oxidation of organic substances in water and wastewater. The article covers various aspects of BOD, including its theory, measurement methods, typical values for different types of waters and wastewaters, and its applications. It discusses the importance of BOD in evaluating water quality and the potential problems caused by high levels of BOD, such as oxygen depletion and negative impacts on aquatic life. The article also explains the different types of BOD, such as five-day BOD (BOD5), ultimate BOD (UBOD), carbonaceous oxygen demand (CBOD), and nitrogenous oxygen demand (NBOD). It describes the measurement procedures for BOD, including the BOD5 test, which is a widely used standardized test for quantifying the organic strength of wastewater. The article discusses the limitations of the BOD5 test and proposes alternative techniques and estimation methods. Furthermore the article explores the relationship between BOD and other parameters like chemical oxygen demand (COD) and theoretical oxygen demand (ThOD). It highlights the importance of BOD in surface water quality modeling and wastewater treatment modeling.[1]

Article 2: "Chemical Oxygen Demand (Titrimetric, High Level For Saline Waters)"Journal WPCF, Vol. 37, p 1716-1721(1965).

The study demonstrates how to perform experiments without making errors in this article suggests clearing the glassware containing organic matter with running blank procedure.

If the sample contains volatile material then the condenser should be rinse with highly acidic silver sulphate sulphuric acid solution while cooling the flask.[2]

Article 3: "Methods of sampling and test (physical and chemical) for water and wastewater"Google patents (Editorial Revision 2016)

Author:Veses, A., &Lafuente, J

In the preparation of this standard, considerable assistance has been derived from 1S0 6060: 1989 'Water quality - Determination of the chemical oxygen demand and standard methods for the examination of water and wastewater', 19th Edition-1995, published by the American Public Health Association, Washington, U.S.A.

It specifies two procedures one for high COD and other for low COD against a single procedure. Precision and accuracy both depends upon the COD value. For the high COD values (400 mg/l) precision up to 2 percent is expected from a good analyst. As the COD value goes on decreasing, precision also become poorer and poorer that is percentage goes on increasing. Precision for the low COD samples maybe improved by using alternate method where diluted reagents are used.[3]

Article 4: "The science of chemical oxygen demand"Standard methods for the examination of water and waste water, 15th Edition p 445-470 (2017).

This booklet no.9 written by Wayne Boyles describes the three tests i.e. BOD, COD and TOC and shows the comparison of this test with proper reagent used. In COD if we used alternate reagent instead of potassium dichromate what are the advantages and disadvantages. One of the main challenges when using alternate reagents is the potential for interference from certain organic compounds present in the sample. Different reagents may react differently with specific organic compounds, leading to variations in COD measurements. Researchers have conducted comparative studies to evaluate the performance and accuracy of alternate reagents compared to potassium dichromate. These studies have examined the effects of interfering substances and the overall reliability of the results obtained. Safety and Environmental Concerns:Studies have highlighted the hazardous nature of potassium dichromate and its potential adverse effects on human health and the environment. The search for alternative reagents stems from the need to reduce the risks associated with the use, storage, and disposal of potassium dichromate. Alternative Reagents Explored: Several alternative reagents have been investigated, including hydrogen peroxide (H2O2), potassium permanganate (KMnO4), potassium persulfate (K2S2O8), and various photochemical oxidation methods. These alternative reagents are generally considered safer and less toxic than potassium dichromate.[4]

Article 5: Chen, G., & Zhao, Y., "Wastewater treatment methods"Chemical Engineering Journal, 248, 661-671. (2014)

The present invention relates to a wastewater treatment method that effectively treats complex organic substances found in wastewater, such as carbohydrates, proteins, lipids, and microcrystalline cellulose, using bacteria. When foreign matter enters a water body, it can be decomposed and oxidized by microorganisms, consuming dissolved oxygen and affecting the self-purification capacity of the water. Various pollutants, including organic pollutants, heavy metals, sanitizing agents, and kitchen waste, can cause water pollution.

The current wastewater treatment methods often require advanced treatment units to meet water quality standards. In the case of organic compounds in wastewater, their oxidation mechanism can involve the addition or hydrogen atom abstraction by hydroxyl free radicals. The Fenton oxidation method, which uses hydrogen peroxide and ferrous ions under acidic conditions, is commonly used for the oxidation of organic compounds. However, excessive ferrous ions can lead to reduced removal rates of chemical oxygen demand (COD) and color during the coagulation stage.

The present invention aims to provide a wastewater treatment method that can effectively degrade complex organic substances, such as carbohydrates, proteins, lipids, and microcrystalline cellulose, by utilizing bacterial metabolism. The method involves two elementary operation units.

In the first elementary operation unit, an acidification tank and a fermenter are used. The wastewater undergoes acidification in the acidification tank, where acid-producing bacteria convert complex organic substances into organic acids, ethanol, hydrogen, and carbonic acid gas. The acidified wastewater is then transferred to the fermenter, where acetic acid Methane producing bacteria convert acetic acid into methane and carbon dioxide, degrading the COD. The treated wastewater and sludge can be further processed separately.

In the second elementary operation unit, a Fenton reaction unit is added before the acidification tank. The wastewater undergoes an oxidizing reaction in the Fenton reaction unit, and the resulting wastewater is then subjected to acidification and methanation processes in the acidification tank and fermenter, respectively.

By employing these wastewater treatment methods, the present invention enables the effective degradation of complex organic substances and the reduction of COD in wastewater. The operational parameters, such as temperature, pH, and hydraulic detention time, can be controlled accordingly for optimal treatment efficiency.[5]

Chemical Oxygen Demand (COD)

3.1 General Description

The Chemical Oxygen Demand (COD) test converts organic carbon to CO_2 and H_2O using a potent chemical oxidant in an acid solution. Chemical oxygen demand, according to its definition is a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by strong chemical oxidant. Utilizing titrimetric or photometric techniques, the amount of oxidant consumed is measured to calculate the oxygen demand. The test is not adversely affected by harmful compounds, and test results are available in 1-1/2 to 3 hours, allowing for quicker process management and evaluation of the water quality.

3.1.1 Advantages and Disadvantages of COD

- **Advantages of COD**

1. Fast results: COD tests can provide results within a few hours, making it suitable for rapid analysis and process control.
2. Broad range of measurement: COD can measure a wide range of organic compounds, including both biodegradable and non-biodegradable substances.
3. Useful for industrial wastewater: COD is particularly valuable for assessing the organic pollution load in industrial wastewater, where diverse and complex compounds may be present.

- **Disadvantages of COD**

1. Lack of specificity: COD does not differentiate between biodegradable and non-biodegradable compounds. Therefore, it may overestimate the actual oxygen demand since some compounds are not readily degradable by microorganisms.
2. Chemical interference: Certain substances present in the water, such as chlorides, can interfere with COD measurements, leading to inaccurate results.
3. Limited ecological relevance: COD does not provide direct information about the environmental impact of wastewater because it includes non-biodegradable substances.

3.2 Designed industrial estimation protocol on chemical oxygen demand

- **Objectives:** To find out Chemical Oxygen Demand (COD) of given waste water sample

- **Principle:**

In the presence of sulfuric acid, silver sulfate, and mercury sulfate, potassium dichromate oxidizes the organic content in the water sample to produce carbon dioxide (CO_2) and water. The amount of ferrous ammonium sulfate consumed in the blank and sample titrations differs, and this difference is used to compute the amount of potassium dichromate needed. The amount of oxygen (0_2) used to oxidize the wastewater's organic content is equivalent to the amount of potassium dichromate utilized in the reaction.

- **Apparatus:**

1. Volumetric flask
2. Reagent storing bottles
3. Measuring cylinder
4. Pipette
5. COD digestor with apparatus

- **Reagents:**

1. Standard Potassium dichromate 0.25N K_2Cr2O_7:Dissolve 1.225gm of K_2Cr2O_7 in the distilled water and dilute it in 100ml
2. Ferrous ammonium sulphate (Mohr's salt) $FeSO_4 (NH_4)_2.6H_2O$ 0.1N:3.9212gm is required for 0.1N in the distilled water with 2ml H_2SO_4 in 100ml of solution. [sulphuric acid is added in the Mohr's salt because ferrous ions in Mohr's salt undergoes hydrolysis conc.H_2SO_4 needs to be added to it during the preparation of its standard solution]
3. Mercury sulphate $HgSO_4$: 2gm in 100ml conc. sulphuric acid to make a mercury sulphate solution so it is used to avoid interference of chloride ions present in the sample.
4. Silver sulphate Ag_2SO_4: 1gm of silver sulphate is required for 100ml it acts as a catalyst dissolve in conc.sulphuric acid Conc.sulphuric acid 98%
5. Ferroin indicator (1,10-Phenanthroline iron(II) sulphate complex):Mix and dissolve 1.485gm of 1,10-phenanthroline monohydrate together with 700mg of ferrous sulphate ($FeSO_4.7H_2O$) in the distilled water and dilute it to 100ml of solution.

- **Procedure:**

a. Take 10 ml of sample water in the flask.
b. Put 5 ml of K_2Cr2O_7 and 15ml silver sulphate solution with 1ml mercury sulphate solution.
c. Attach the condenser to the mouth of the flask.
d. Process further to CO_2 digester or on heating mantle for 150°C to reflux the content up to 2hrs.

e. Cool the flask, detach from the unit and dilute its contents to 150ml by adding distilled water.
f. Add 2-3 drops of ferroin indicator solution & titrate against ferrous ammonium sulphate solution.
g. At the end point blue green color changes to reddish blue.
h. Repeat the same procedure for Blank titration.(instead of sample)

- **Calculations:**

$$\text{COD (mgl}^{-1}\text{or ppm)} = \frac{[(B\text{-}S) * \text{Normality of FAS} * 8000]}{V}$$

S = Volume of titrant used against sample.
B = Volume of titrant used against distilled water or blank sample.
N = Normality of titrant
V = Volume of sample taken in ml.

- **Result and Discussion:**

This protocol is referred for high cod for low cod change the concentration from 0.25N to 0.025N or to 0.0016N of oxidizing agent.

- **Caution:**
  In carrying out the following procedures, use proper safety measures, including protective clothing, eye protection, and a fume hood. Reagents containing heavy metals ($HgSO_4$ and Ag_2SO_4) should be disposed of as toxic wastes.

CASE STUDY ON COD

4.1 Report Summary:

The case study focuses on the optimization of the effluent sample from the ABC Manufacturing Plant by reducing Chemical Oxygen Demand (COD) levels. The objective is to minimize organic pollution and enhance the environmental sustainability of the plant's wastewater discharge.

The initial COD analysis of the effluent sample collected from the plant on May 7, 2023, revealed a value of 16,000ppm. Several experiments were conducted to determine the COD of different water sources, including tap water, effluent samples from various companies, pond water, and the high COD sample "Sewoclean" from Greyeast Technologies Pvt.Ltd.The experiments involved the use of different oxidizing agents, such as 0.1N and 0.025M Ferrous Ammonium Sulphate (FAS) and 0.5N and 0.25N Potassium Dichromate. The samples were subjected to reflux, titration, and centrifugation processes to determine COD levels accurately.

The results obtained from the experiments indicated varying COD values for different water sources. The effluent sample from the ABC Manufacturing Plant showed COD levels of 320ppm. Pond water exhibited COD values of 30ppm, while the high COD sample "Sewoclean" showed inconsistent results due to the presence of suspended particles and improper dilution.

In the optimization process, different techniques were employed, such as filtration and centrifugation, to improve the accuracy of COD measurements. Filtered samples and centrifuged samples yielded different COD values, indicating the influence of suspended particles on the results.

4.2 Experiments performed data

1. **Experimental data:1**
   Date: 5-04-23

- **Aim:** To determine chemical oxygen demand of tap water.
- **Effluent Sample Details:**
  Source: greyeast technologies pvt. ltd Tap water
  Objectives: To test Organic matter present with in the tap water
  Due to some instrumental error failed to complete the experiments.

2. **Experimental data:2**
   Date: 6-04-23

- **Aim:** To determine chemical oxygen demand of effluent and tap water.
- **Effluent Sample Details:**
  Source: greyeast technologies pvt. ltd tap water and xyz manufacturing company effluent
  Objectives: To test content oxygen demand with in the tap water as well as abc pharmaceutical company.
  Due to Broken equipment failed to complete the experiments.

3. **Experimental data:3**
   Date: 7-04-23

- **Aim:** To determine chemical oxygen demand of effluent by 0.1N FAS.
- **Effluent sample details:**
- Source: greyeast technologies pvt. ltd tap water and xyz manufacturing company effluent
- Objectives: To test content oxygen demand with in the tap water as well as abc pharmaceutical company.

Figure 4.1 Blank sample and effluent sample after reflux.

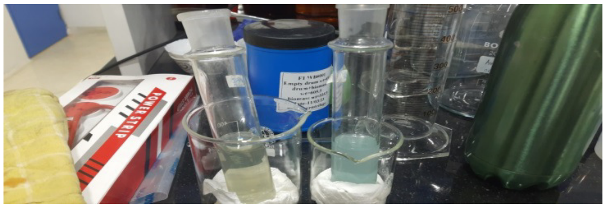

Figure 4.2 Blank sample and effluent sample after titration.

Result and Conclusion:

Result: The COD of the effluent sample is measured to be 240 ppm.

Due to time limitations, the experiment could not be completed fully.

4. **Experimental data:4**
   Date: 10-04-23

- **Aim:** To determine chemical oxygen demand of tap water and effluent by 0.1N ferrous ammonium sulphate.
- **Effluent sample details:**
  Source: greyeast technologies pvt. ltd tap water and xyz manufacturing company effluent
  Objectives: To test content oxygen demand with in the tap water as well as abc pharmaceutical company.

Figure 4.3 Blank sample, tap water and effluent sample after reflux.

Figure 4.4 After titration with FAS

Results: COD of the sample effluent is 320ppm,Due to the low organic content in tap water, the COD value could not be determined accurately.
Conclusion: The effluent sample shows a significant COD value, indicating a higher oxygen demand compared to tap water. Further analysis and investigation are required to assess the organic content in tap water accurately.

5. **Experimental data:5**
   Date: 11-04-23

- **Aim:** To determine cod of pond water by 0.025M FAS
- **Sample details:**
  Source:Achole pond water sample
  Objectives: To test content of COD
- **Preparation of Reagent:**

1) Add 0.613gm potassium dichromate previously dried to 60^0C for atleast 2hrs in oven into 80ml distilled water then shake the flask well to dissolve the content and make up the solution to 100ml and mix well.
2) 0.025M ferrous ammonium sulphate: Dissolve 0.98gm of ferrous ammonium sulphate in 2ml conc.sulphuric acid and make up volume to

100ml it is recommend to prepare solution before 10-20min before titration because ferrous ammonium sulphate resist oxidation by air.

- **Procedure:** procedure is same as listed above on 2nd page.
  Results in error
  No end point

6. **Experimental data:6**
   Date: 12-04-23

- **Aim:** To determine COD of pond water and sample effluent by 0.025M
  Effluent sample details:
  Source: Achole pond water sample and xyz manufacturing company effluent
- Objectives: To test chemical oxygen demand with in the tap water as well as abc pharmaceutical company.
  Results in 180ppm for effluent and for pond water is 30ppm

7. **Experimental data:7**
   Date: 12-04-23

- **Aim:** To determine cod of effluent by 0.025M FAS

Figure 4.5 Blank and achole pond sample in elenmeyer flask

Figure 4.6 Before end point

Figure 4.7After end point

Results: COD of Achole pond water is 30ppm.

8. **Experimental data:8**
   Date: 13-04-23

- **Aim:** To determine cod of achole pond water by 0.025M FAS
  Repeated to reverify
  Results 30ppm

9. **Experimental data:9**
   Date: 13-04-23

- **Aim:** To determine high cod sample that is sewoclean by 0.025M
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd
- **Result:** High content of organic matter so potassium dichromate present in the sample is less so the sample has no end point.

10. **Experimental data:10**
    Date: 14-04-23

- **Aim:** To determine high cod sample that is sewoclean by 0.025M
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd

Figure 4.8 After removing the cod digestor

Figure 4.9 Aliquot before titration

Results: no end point colour changes drastically to black due to high organic matter with suspended particles.

11. **Experimental data:11**
    Date: 15-04-23

- **Aim: To determine high cod sample that is sewoclean with 0.25N**
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd
  Results: Error due to high organic matter potassium dichromate oxidizes completely.

12. **Experimental data:12**
    Date: 16-04-23

- **Aim: to determine high cod sample with 2.5N oxidizing agent for sewoclean**
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd
- **Preparation of reagent:** Potassium dichromate used in this run is 2.5N take 12.25gm in 100ml distilled water.
  Ferrous ammonium sulphate used 1N about 39.2gm of FAS adding 20ml distilled water and make up volume upto 100ml distilled water.
  Result in variation in value due to improper dilution
  For 1:10 it gives 80000ppm and another it gives 40000ppm and for 1:100 it results in 2,40,000
  Discussion: As we have increase the concentration oxidizing agent remains in the sample so we conclude that high concentration is needed.

Figure 4.10 sample before titration

13. **Experimental data:13**
    Date: 18-04-23

- **Aim:** To determine the cod of sewoclean by 0.5N oxidizing agent.
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd
- **Preparation:** Potassium dichromate for 0.5N we used 2.45gm of potassium dichromate and 0.2N ferrous ammonium sulphate we used 7.84gm
- **Results:**Highly diluted sample results
  1:10 samples: 19,200ppm
  1:100: 1,60,000ppm
  1: 1000: 2,40,000ppm
  Results in colour changes to greenish blue and at end point it changes to brick red.
  Conclusion: Due to suspended particles present in the sample so it vary in the result

Figure 4.11 Samples before titration after reflux

Experimental data:14
Date: 19-04-23

- **Aim:** To determine COD of sewoclean using 0.25N potassium dichromate and 0.1N FAS.
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd

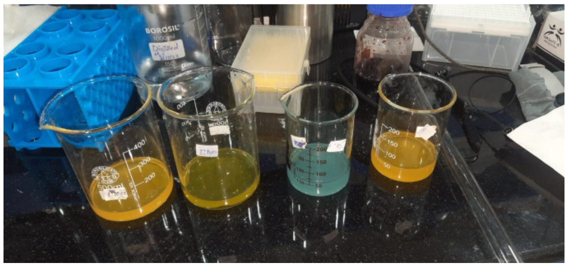

Figure 4.12 Samples before titration

Results:
1:100 = 49,600ppm
1:1000 = 80,000ppm
Discussion: Some errors due to suspended particles vary in preparation of sample

14. Experimental data:15

Date: 20-04-2023

- **Aim:** Reverification experiment to determine COD of sewoclean.
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd

Result:
In this experiment we taken filter samples and unfilter samples
1:100filtered and unfiltered samples were taken
1:1000 unfiltered is taken,
Result: 1:100F = 1:100filtered = 46,720ppm
1:1000UF = unfiltered = 80,000ppm
1:100UF = unfiltered = 72,000ppm

15. Experimental data:16

Date: 22-04-2023

- **Aim:** Reverification experiment by centrifuge to determine COD of sewoclean.
- **Sample details**: Sewoclean from greyeast technologies pvt.ltd
- **Results:**

In this experiment we centrifuge the sample at 4000rpm for 10min
1:100 dilution aliquot: 48000ppm
1:1000 dilution aliquot: 48800ppm
Statistical data:

- **Formula:**

Steps to calculate standard error without excel are:

1. Calculate the average(μ)
2. Calculate standard deviation using formula:
3. SD sample :$\sigma = [\sum(x-\mu)^2/n-1]^{1/2}$
4. Standard error: $\sigma/n^{1/2}$

16. Experimental data:16

Date: 8-05-2023

- **Case study:** Optimization of Effluent sample from ABC Manufacturing Plant
- **Objective:** The objective of this case study is to optimize the effluent sample from the ABC Manufacturing Plant by reducing Chemical Oxygen Demand (COD). The optimization process aims to minimize organic pollution levels and improve the environmental sustainability of the plant's wastewater discharge.
- Effluent Sample Details:

1) Source: ABC Manufacturing Plant wastewater discharge
2) Sample Collection Date: May7, 2023
3) Test Methods: Standard COD methods
4) Initial COD Analysis:

a. Sample Collection: A representative effluent sample was collected in a clean, airtight container.
b. COD Analysis: The collected sample was analyzed for COD using the standard methods.
c. Results: Initial COD: 16000ppm

When the effluent has a Chemical Oxygen Demand (COD) concentration of 16,000 ppm, it indicates a high organic load in the wastewater.

d. Discussion:COD digester becomes damaged during an experiment, it is crucial to halt the experiment to ensure the reliability and accuracy of the results.

Biological Oxygen Demand (BOD)

5.1 General description

The BOD test most closely resembles aerobic waste treatment and the aquatic ecosystem among the three test techniques used to estimate oxygen demand (COD, TOC, and BOD). In this experiment, microbes consume organic substances as food while also ingesting oxygen. The typical

BOD test calculates the sample's oxygen consumption over a five-day period. The test's lengthy execution time means that the results only provide historical information and do not allow for an efficient process control system or a quick evaluation of the water quality. The test has limitations when used in some situations, such as industrial wastewaters, which frequently contain heavy metal ions, cyanidesandother chemicals hazardous to microorganisms. When hazardous compounds poison microorganisms, they are unable tooxidize waste, in which case the BOD test becomes an ineffective measure of organic pollution.

5.1.1 Advantages and Disadvantages of BOD

- **Advantages of BOD**

1. Reflects actual biodegradable organic content: BOD specifically measures the oxygen demand resulting from the biodegradation of organic matter, providing a more ecologically relevant assessment of water quality.
2. Environmental significance: BOD results can indicate the potential impact of wastewater on the receiving water bodies and the effectiveness of natural self-purification processes.
3. Standardized method: BOD testing follows standard protocols, allowing for consistent comparison of results among different laboratories.

- **Disadvantages of BOD**

1. Long testing time: BOD tests require incubation periods of several days (usually 5 days) to measure the oxygen demand accurately. This makes it time-consuming and less suitable for immediate process control.
2. Limited range: BOD tests have a limited measuring range, and samples with high organic loads may require dilution to fall within the applicable range.
3. Variability in results: BOD measurements can be affected by factors such as temperature, microbial activity and sample handling, leading to variability in results.

5.2 Designed industrial estimation protocol on biological oxygen demand

- **Objectives:** To find out Biological Oxygen Demand (BOD) of given waste water sample.

- **Principle:**

The manganese sulphate combines with the dissolved oxygen in the sample to form a white precipitate of manganese hydroxide under alkaline circumstances (by adding alkali-iodide). This produces a brown precipitate of manganese hydroxide.

Manganese switches to its divalent state in an acidic environment and releases iodine. Utilising starch as an indication, this release iodine is titrated against sodium thiosulphate.

- **Apparatus:**

1. Volumetric flask
2. BOD bottles
3. Measuring cylinder
4. Pipette

- **Reagents:**

1. $MnSO_4$ (Manganese sulphate) : Dissolve 48g of MnSO□·3H□O in 100ml of distilled water. Filter if necessary.
2. Alkali- Iodide: Dissolve 20g of NaOH and 3g KI in 100ml distilled water.
3. Conc. H_2SO_4 :
4. Starch Indicator (1%): 1g of starch dissolves in 100ml distilled water.
5. 0.025N Sodium thiosulphate: of sodium thiosulphate in 200ml distilled water.

- **Procedure:**

1. Collect the waste water sample.
2. Collect 300ml of waste water sample in BOD bottle using pipette without making air bubble.
3. Add 2 ml of $MnSO_4$ to the BOD bottle carefully by inserting the pipette just below the surface of water.
4. Add 2ml of alkali-iodide reagent in the same manner. Stopper the bottle immediately.
5. Mix well by inverting the bottle 2-3 times, a brownish cloud will appear in the solution as an indicator of the presence of oxygen.
6. Allow the precipitate to settle leaving approx. 150ml clear supernatant.

7. Add 2ml of conc. H_2SO_4 carefully without forming air bubbles; mix the solution well to dissolve the precipitate.
8. Take 200ml solution from this in Erlenmeyer flask and titrate against $Na_2S_2O_3$ using starch as an indicator. (Note: Color change from bluish greenish to colorless)
9. For blank use distilled water.
10. Keep one bottle for determination of initial Dissolved oxygen (DO) level on 1st day and incubate 2nd bottle at 20°C for the determination of DO level on 5th day for waste water sample and Blank both.

- **Calculations:**

DO(mgl^{-1}or ppm)O_2 = [V_T * N of $Na_2S_2O_3$ * Equivalent wt.O_2] *DF
Volume of sample used during titration
BOD (mgl^{-1}or ppm) = DO_i-DO_f
Dissolved oxygen (DO_i)
Dissolved oxygen (DO_f)

CASE STUDY ON BOD

6.1 Report Summary:

The case study focuses on the determination of dissolved oxygen (DO) in the effluent of a pharmaceutical company using the Winkler method. The study consists of three experimental datasets conducted on different dates. The initial experimental data using a DO probe failed to provide accurate readings, leading to the adoption of the Winkler method as an alternative.

In the second and third experiments, the Winkler method was employed to determine the DO levels in two different effluent samples. The procedure involved collecting the waste water samples, adding specific reagents, allowing precipitate settling and performing titration. The results obtained from the experiments indicated the DO levels (DOi and DOf) and the corresponding biochemical oxygen demand (BOD) values for each sample. The percentage reduction in BOD was also calculated.

Data Analysis:

In the second experiment, Effluent 1 showed a 42.9% reduction in BOD for the 1% sample and a 73.3% reduction for the 2% sample. Effluent 2 exhibited an 84% reduction in BOD for the 1% sample and a significant 94% reduction for the 2% sample.

In the third experiment, Effluent 1 had a 28.7% reduction in BOD for the 2% sample, while Effluent 2 demonstrated a reduction of 4.3% for the 1% sample and a substantial reduction of 80.65% for the 2% sample.

6.2 Experiment performed data

1. **Experimental data:1**
   Date: 9-05-2023

- Aim: To determine dissolved oxygen of xyz pharma effluent dissolved oxygen probe
- Procedure: Collect the waste water sample.
  1) Collect 300ml of waste water sample in BOD bottle using pipette without making air bubble.
  2) Calibrate DO probe as per manufacture.
  3) Take DO intial from day 1 and DO from day 5

  Result: Experiment failed

  Conclusion: DO probe failed to take readings due to faulty probe so alternate method is used to find dissolved oxygen.

2. **Experimental data:2**
   Date: 16-05-2023

- Aim: To determine dissolved oxygen of xyz pharma effluent of 2 plant effluent by winkler method
- Procedure:
  1. Collect the waste water sample.
  2. Collect 300ml of waste water sample in BOD bottle using pipette without making air bubble.
  3. Add 2 ml of MnSO4 to the BOD bottle carefully by inserting the pipette just below the surface of water.
  4. Add 2ml of alkali-iodide reagent in the same manner. Stopper the bottle immediately.
  5. Mix well by inverting the bottle 2-3 times, a brownish cloud will appear in the solution as an indicator of the presence of oxygen.
  6. Allow the precipitate to settle leaving approx. 150ml clear supernatant.
  7. Add 2ml of conc. H2SO4 carefully without forming air bubbles; mix the solution well to dissolve the precipitate.

8. Take 200ml solution from this in Erlenmeyer flask and titrate against Na2S2O3 using starch as an indicator. (Note: Colour change from bluish greenish to colourless)
9. For blank use distilled water.

- Note-Keep one bottle for determination of initial Dissolved oxygen (DO) level on 1st day and incubate 2nd bottle at 20°C for the determination of DO level on 5th day for waste water sample and Blank both.
- **Results:**

Table 6.1 Effluent 1

sample	DO$_i$	DO$_f$	BOD(mg^{-1})	%reduction
1%	140ppm O$_2$	80ppm O$_2$	60	42.9
2%	30ppmO$_2$	8ppmO$_2$	22	73.3

Table 6.2 Effluent 2

sample	DO$_i$	DO$_f$	BOD(mg-1)	%reduction
1%	180ppm O2	32ppm O2	168	84
2%	200ppm O2	10ppm O2	170	94

- **Conclusion:**
  The experiment result can vary due to formation of bubbles so this concludes that final dissolved oxygen may be incorrect.

3. **Experimental data:3**
   Date: 27 -05-2023

- Aim: To determine dissolved oxygen of xyz pharma effluent of 2 plant effluent by winkler method
- Procedure: Collect the waste water sample.

1. Collect the waste water sample.
2. Collect 300ml of waste water sample in BOD bottle using pipette without making air bubble.
3. Add 2 ml of MnSO4 to the BOD bottle carefully by inserting the pipette just below the surface of water.
4. Add 2ml of alkali-iodide reagent in the same manner. Stopper the bottle immediately.
5. Mix well by inverting the bottle 2-3 times, a brownish cloud will appear in the solution as an indicator of the presence of oxygen.
6. Allow the precipitate to settle leaving approx. 150ml clear supernatant.
7. Add 2ml of conc. H2SO4 carefully without forming air bubbles, mix the solution well to dissolve the precipitate.
8. Take 200ml solution from this in Erlenmeyer flask and titrate against Na2S2O3 using starch as an indicator. (Note: Colour change from bluish greenish to colourless).

- Note: Keep one bottle for determination of initial Dissolved oxygen (DO) level on 1st day and incubate 2nd bottle at 20°C for the determination of DO level on 5th day for waste water sample and Blank both.
- **Results:**

Table 6.3 Effluent 1

sample	DO$_i$(ppm)	DO$_f$(ppm)	BOD (ppm)	%reduction
5%	21.6 ppmO$_2$	Error	-	-
2%	14 ppmO$_2$	10 ppmO$_2$	4	28.7

Table 6.4 Effluent 2

sample	DO$_i$	DO$_f$	BOD(ppm)	%reduction
1%	116 ppmO	111ppm O$_2$	5	4.3%
2%	15.5 ppmO$_2$	3 ppmO$_2$	12.5	80.65%

The experiment result can vary due to formation of bubbles so this concludes that BOD may be incorrect.By deviating from this protocol and using a fume hood with room temperature, the conditions for the BOD test are altered, which can lead to inconsistent and potentially inaccurate results. Therefore, it is important to adhere to the recommended protocol and maintain the appropriate temperature conditions when conducting BOD experiments to ensure the reliability of the results.

II. CONCLUSIONS

Series of experiments were conducted using various water sources, including tap water, effluent samples from different companies, pond water, and the high COD sample "Sewoclean" from Greyeast Technologies Pvt. Ltd. Different oxidizing agents and processes such as reflux, titration, and centrifugation were employed in the experiments. These findings underscore the importance of mitigating the influence of suspended particles in COD analysis for accurate and reliable results. By implementing filtration and centrifugation, the optimization process aimed to enhance the effectiveness of wastewater treatment strategies and minimize the organic pollution associated with the ABC Manufacturing Plant's effluent.

The study consists of three experimental datasets conducted on different dates. The initial attempt to measure DO using a DO probe proved inaccurate due to calibration or handling, leading to the adoption of the Winkler method as an alternative approach.

Data analysis revealed significant reductions in Biochemical Oxygen Demand (BOD) values for both effluent samples in the second experiment.

These findings demonstrate the effectiveness of the Winkler method in accurately determining DO levels and provide insights into the performance of the wastewater treatment processes in reducing organic pollution. The substantial reductions in BOD values indicate positive outcomes in terms of environmental sustainability and compliance with regulatory standards.

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