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COLLEGE OF GRADUATE STUDIES

ENVIRONMENTAL EVALUATION FOR TREATED WASTEWATER IN KHARTOUM REFINERY

التقويم البيئي للمخلفات السائلة المعالجة في مصفاة الخرطوم

A Thesis Submitted in Partial of the

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Master of Science in Environmental Engineering

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الآية

قال الله تعالى:

"اللَّهُ نُورُ السَّمَاوَاتِ وَالأَرْضِ مَثَلُ نُورِهِ كَمِشْكَاةٍ فِيهَا مِصْبَاحٌ الْمِصْبَاحُ فِي زُجَاجَةٍ الزُّجَاجَةُ كَأَنَّهَا كَوْكَبٌ دُرِّيٌّ يُوقَدُ مِن شَجَرَةٍ مُّبَارَكَةٍ زَيْتُونَةٍ لّا شَرْقِيَّةٍ وَلا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ نُّورٌ عَلَى نُورٍ يَهْدِي اللَّهُ لِنُورِهِ مَن يَشَاء وَيَضْرِبُ اللَّهُ الأَمْثَالَ لِلنَّاسِ وَاللَّهُ بِكُلِّ شَيْءٍ عَلِيمٌ (35)"

صَدَقَ اللهُ العَظِيم

سورة النور: الآية (35)

DEDICATION

I dedicate my dissertation work to my family and many friends.
A special feeling of gratitude to my loving parents, whose words of encouragement and push for tenacity ring in my ears. My sisters and brothers have never left my side and they are very special. I also dedicate this dissertation to my many friends and who have supported me throughout the process. I will always appreciate all what they have done, for helping me develop my technology skills, proofreading.

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ABSTRACT

The environmentally adequate disposal of oily wastewater is a current challenge to the petroleum industry. Currently, more consideration has been focused on the treatment techniques of oily wastewater. Therefore, oily wastewater treatment has become an urgent problem, and it must be explored and resolved by every refinery company. Oil refinery wastewaters contain many different chemicals at different concentrations for example: ammonia, sulfides, phenol and hydrocarbons. The study considered Khartoum Refinery as study area, treated wastewaters were qualified using standard laboratory tests for the following properties: Phenol Content, Ammonia Nitrogen, Suspended Solids, Sulfide content, Chemical Oxygen Demand(COD) and Biological Oxygen Demand (BOD) , the average results of oxidation pond (discharge point) samples were compatible with the Sudanese Standards and the Chinese standards in: Phenol Content which is reduced to 0.2 mg/l; Ammonia Nitrogen is reduced to 1.7 mg/l .However, the results were in agreement with one of the two standards ,sulfides is reduced to 7.6 mg/l , Suspended Solids value are reduced to 43.3 mg/l, Biological Oxygen Demand is reduced to 33.3 mg/l, Chemical Oxygen Demand is reduced to 142.7 mg/l. The results obviously showed the effectiveness of the treatment methods adopted by the Khartoum Refinery for decreasing levels of the contaminants in discharged wastewaters to meet the standards. More research and development work are recommended to adopt new combined treatment processes for Khartoum Refinery wastewater, to maximize the efficiency while decreasing cost of treatment.

المستخلص

يمثل التخلص الملائم بيئيًا من المخلفات السائلة الزيتية تحديًا لصناعة البترول، في هذه الدراسة تم التركيز بشكل أكبر على تقنيات معالجة المخلفات السائلة الزيتية لذلك أصبحت معالجة المياه العادمة الزيتية مشكلة ملحة ، ويجب استكشافها وحلها من قبل كل شركة مصفاة. تحتوي مياه الصرف في مصافي النفط على العديد من المواد الكيميائية المختلفة بتركيزات مختلفة على سبيل المثال: الأمونيا والكبريتيد والفينول والهيدروكربونات ،اتخذت الدراسة مصفاة الخرطوم كمنطقة دراسة ، وقد تم تقييم المخلفات السائلة المعالجة باستخدام الاختبارات المعملية القياسية للخصائص التالية: محتوى الفينول ، نيتروجين الأمونيا ، المادة الصلبة العالقة ، محتوى الكبريتيد ، طلب الأكسجين الكيميائي ، طلب الأكسجين الحيوي ، متوسط نتائج عينات بركة الأكسدة(نقطة التصريف) كانت متوافق مع المواصفات السودانية والمواصفات الصينية في: محتوى الفينول الذي انخفض إلى 0.2 ملجم / لتر،تم تقليل النيتروجين الأمونيا إلى 1.7 ملجم / لتر،ومع ذلك كانت النتائج متوافقة مع أحد المعيارين في كل من الكبريتيدات التي تم تخفيضها إلى 7.6 ملجم / لتر ،انخفضت القيمة الصلبة المعلقة إلى 43.3 ملجم / لتر ، انخفض الطلب على الأكسجين الحيوي إلى 33.3 ملجم / لتر ، انخفض الطلب على الأكسجين الكيميائي إلى 142.7 ملجم / لتر، اظهرت النتائج بشكل واضح فاعلية طرق المعالجة المعتمدة من قبل المصفاة لخفض مستويات الملوثات في مياه الصرف الصحي المصرفة للوفاء بأحد المعايير، اوصت الدراسة بإجراء المزيد من الأبحاث والتطوير لتبني عمليات معالجة جديدة مشتركة لمياه الصرف الصحي في مصفاة الخرطوم ، لتحسين الكفاءة مع تقليل تكلفة المعالجة.

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LIST OF ABBREVIATIONS

KRC	Khartoum Refinery Company
CNPC	China National Petroleum Company
MEM	Ministry of Energy and Mining
CPU	Crude Processing Unit
RFCC	Residuum Fluid Catalytic Cracking
CRU	Catalytic Reformer Unit
CDU	Crude Distillation Unit
DHT	Diesel Hydro Treating Unit
CCR	Continuous Catalytic Reforming
LPG	Liquefied Petroleum Gas
COD	Chemical Oxygen Demand
BOD	Biological Oxygen Demand
PAH	Polycyclic Aromatic Hydrocarbons
BTEX	Benzene, Toluene, Ethyl benzene and Xylene
DAF	Dissolved Air Flotation
PAC	Poly Aluminum Chloride
TSS	Total Suspended Solids
ASTM	American Standard Testing Methods
GB	China National Standards

LIST OF SYMBOLS

V	Sample volume in ml
C_N	Concentration of standard hydrochloric acid titration solution
V_1	Volume of standard hydrochloric acid solution consumed for titrating sample in ml
V_2	Volume of standard hydrochloric acid solution for titrating blank sample in ml
S^{-2}	Sulfide concentration in mol/l
X	Suspended matter of water sample (mg/l)
W_1	Weight of weighting bottle and filter paper in (g)
W_2	Weight of weighting bottle, filter paper and suspended matter in (g)
V_w	Volume of water sample in (ml)
A	Ferrous ammonium sulphate solution used for distilled water in ml
B	Ferrous ammonium sulphate solution used for sample in ml
C	Normality of ferrous ammonium sulphate solution

CHAPTER ONE

CHAPTER ONE
INTRODUCTION

1.1. General:

Refinery uses water at every stage of the refining process. The most common water used is for cooling hydrocarbons; however, water is also used for cleaning and other purposes. Water is classified as 'waste water' if it could have come in contact with hydrocarbons or other contaminants.

Petroleum refining utilizes large quantities of water for desalting, distillation, thermal cracking, catalytic and treatment processes to produce useful products. Refining process generates wastewater. Discharge of untreated petroleum refining wastewater into water bodies results in environmental and human health effects due to release of toxic contaminants.

The wastewater from Petroleum industries and refineries mainly contains oil, organic matter and other compounds. The composition of effluent in refinery wastewater depends on the crude quality. It varies with the operating conditions in the refinery, non-hydrocarbon substances are removed and the oil is broken down into its various components and blended into useful products.

Water production may lead to the following consequences: damage to the facility, cost of separation, cost of disposal and environmental damage. Oily wastewater pollution is mainly manifested in the following aspects:

1. Affecting drinking water and groundwater resources, endangering aquatic resources.
2. Endangering human health.
3. Atmospheric pollution.
4. Affecting crop production.
5. Destructing the natural landscape.

The treatment of this wastewater can be carried out by physical, chemical and biological treatment processes. Treatment of petroleum wastewater has two stages, firstly, pre-treatment stage to reduce grease, oil and suspended materials. Secondly, an advanced treatment stage to degrade and decrease the pollutants to acceptable discharge values.

1.2 Research Background

Khartoum Refinery Company (KRC), ongoing production in 16 May 2000. It is a joint venture between (CNPC) China National petroleum Company and the Sudanese Ministry of Energy and Mining (MEM). The proprietorship is separated 50% for each party. The refinery can process 2.5 million tons of crude oil each year, and the annual production of petroleum products is 2.26 million. It contains mainly: crude processing unit (CPU), residuum fluid catalytic cracking (RFCC) unit, catalytic reformer (CRU), and diesel hydrogenation unit. Their capacities are, 2.5 million tons annually, 1.8 million tons per year, 150000 tons per year, and 500000 tons per year respectively. Other supporting facilities such as water treatment lab, product storage tanks and transfer systems, and power plant are part of the refinery. The refinery receives its crude oil supply from a 1600 km pipeline that goes through the refinery to the port of the Sudan in the Red Sea (Garelnabi, M., A., 2009).

1.3 Problem statement

Contamination of water and lands by petroleum chemicals is a global issue and is of particular concern in developing countries. In addition, the release of untreated or partially treated petroleum refinery wastewaters into the environment leads to pollution of the terrestrial and aquatic ecosystems, an unaesthetic environment, and losses of farmlands, fishes, portable water and means of the livelihood particularly in the oil polluted community.

Khartoum refinery generates Wastewater by the catalytic hydrocracking and hydroskimming unit, which include a crude distillation unit, a de-sulfuring unit and a reforming unit. Removal of pollutants produced by refinery plant is requirement for reuse of water in cooling water systems, re-boiling to produce steam which used in most of refinery units and obtains to environmental standards because refinery wastewater contain high organic matter, when discharged into the aquatic environment, required 2 mg per litter from dissolved oxygen for normal life, results in decreased dissolved oxygen by the bacteria. Moreover, release of obnoxious odors to the urban atmosphere around refinery.

The study aims to examine the treated wastewater from Khartoum refinery to obtain safe discharge into around environment.

1.4. Research objectives

1.4.1 General objective

Characterization of wastewater from the Khartoum Refining Company (KRC) at the point of its discharge into the oxidation basins.

1.4.2 Specific objectives

1. Examination of the efficiency of the wastewater treatment plant in Khartoum Refinery in terms of: phenol content, ammonia nitrogen, suspended solid content, sulfide content, chemical oxygen demand (COD) and biological oxygen demand (BOD) by comparing with Chinese and Sudanese standards using water samples from different points of the treatment plant.
2. Represent the relation between samples taken using the laboratory test results for each of: phenol content, ammonia nitrogen, suspended solid content, sulfide content, chemical oxygen demand (COD) and biological oxygen demand (BOD).

CHAPTER TWO

CHAPTER TWO
LITERATURE REVIEW

2.1 Introduction:

The petroleum refining industry converts crude oil into a lot of refined products, including liquefied petroleum gas, gasoline, kerosene, aviation fuel, and diesel fuel, fuel oils, lubricating oils, and feed stocks for the petrochemical industry. Typically, petroleum refining activities start with receipt of crude oil for storage at the refinery, include all petroleum handling and refining operations, and they terminate with storage prior to shipping the refined products from the refinery (Babiker ,2003).

2.1.1The Main Production Units in Refinery:

A. Crude Distillation Unit (CDU), capacity: 2.5 mt/y, containing of an electric desalter and crude distillation products refining.
B. Residual Fuel Oil Catalytic Cracking Unit (RFCC), capacity :1.8 m t/y, containing of reaction and regeneration, distillation, absorption and stabilization, energy retrieval, Sulphur and mercaptan removal units
C. Reforming Unit, capacity: 150000 t/y and containing of pretreatment and reforming units.
D. Diesel Hydro Treating Unit (DHT), capacity: 500000 t/y, containing of hydrogen recovery and diesel hydrogenation units.
E. Sour Water Stripping Unit, capacity: 400000 t/y.
F. Delayed Coking Unit (DCU).
G. Gasoline Diesel Hydro Treating (GDHT).
H. Continuous Catalytic Reforming (CCR).
I. Utilities include: a power plant, a waste water plant, an air
J. Separation and compression unit and a river water purification plant (1500 m^3/h).

(Liquefied Petroleum Gas (LPG) Demand, Supply and Future Perspectives for Sudan Synthesis report of a workshop, 2010)

Figure (2.1): Khartoum Refining Production Units (Mohamed, 2015)

2.1.2 Main Products:

The Khartoum Refining output oil products such as gasoline, jet fuel, diesel (naphtha), fuel oil, liquefied gas (LPG), kerosene and benzene (mogas). Among them the gasoline is unleaded, the diesel is of high quality with low Sulpher, low aromatics and light color. Due to its low Sulphur content, Liquid Petroleum Gas (LPG) is a clean fuel satisfying the environment protection requirements. (Khogali. H.A, 2016)

Figure (2.2): Khartoum Refining Main Products (Mohamed, 2015)

The petroleum refining industry employs a wide variety of physical and chemical treatment processes. A refinery processing flow scheme is largely determined by the composition of the crude oil feedstock and the chosen final petroleum products. Typical processing and auxiliary units in refineries are presented below :(IPIECA Operations Good Practice Series, 2010).

1. Separation processes, include:
   - ❖ Atmospheric distillation
   - ❖ Vacuum distillation

- ❖ Light ends recovery (gas processing)

2. Petroleum conversion processes, contain:
   - ❖ Cracking (thermal and catalytic)
   - ❖ Reforming
   - ❖ Alkylation
   - ❖ Polymerization
   - ❖ Isomerization
   - ❖ Coking
   - ❖ Visbreaking
3. Petroleum treating processes, involve:
   - ❖ Hydrodesulfurization
   - ❖ Hydrotreating
   - ❖ Chemical sweetening
   - ❖ Acid gas removal
   - ❖ DE asphalting
4. Feedstock and product handling, such as:
   - ❖ Storage
   - ❖ Blending
   - ❖ Loading
   - ❖ Unloading
5. Auxiliary facilities, contain:
   - ❖ Boilers
   - ❖ Wastewater treatment
   - ❖ Hydrogen production
   - ❖ Sulfur recovery plant

Large amounts of water are used in refining processes, especially for cooling, distillation, hydrotreating and desalination systems, so it is clear that filtered wastewater can be broadly classified as clean or unclean wastewater. In most modern refineries, these various wastewaters are directed into separate sewage systems. There are at least two different independent sewers in most petroleum refineries: one that deals with rainwater and runoff and one that deals with all process water and water from utility units. However, in more efficient refineries, the treated water streams are usually divided into more than one stream depending on the nature of the wastewater, thus reducing the load on the wastewater treatment plants, increasing the efficiency of each treatment unit and expanding the possibilities for wastewater reuse in various refining units (Hassan, 2008).

2.2 Petroleum refining wastewater:

Petroleum refining industries extract large volumes of freshwater in the process of refining crude oil and as cooling agent. Consequently, large volumes of wastewater are generated as a result of production, storage, and distribution and processing of petroleum, or by accidents due to spills from water/fuel mixtures, oil well drilling, leaks from underground storage or water collected from secondary containment and sumps .The characteristics of petroleum wastewater from different refineries vary from one region of the globe to another depending on the region in which the crude oil was drilled, type of crude oil, its chemical composition, the different processes and the employed treatment mechanism. This has significant impact on the character and quantity of contaminants entering a given refinery wastewater treatment system. High rates of consumption of petroleum and its refined products will continue to generate effluents from petroleum refining processes, which, when discharged into water bodies would result in environmental pollution. The effects of the discharge of effluents include eutrophication, accumulation of toxic compounds in biomass and sediments, loss of dissolved oxygen in water, contamination of drinking water and groundwater resources, thus endangering aquatic resources and human health as well as destruction of the natural land scape. The types of wastewater stream generated by the petroleum refining industry can be classified into desalter water, cooling tower, spent catalyst, spent caustic, water used for flushing during maintenance and shut down, sour water and other residuals from desalting, catalytic cracking, a stripping steam, sanitary, lube oil and asphalt. These wastes pose major problems that are a challenge faced by the petroleum industry, imposing the need to recover oil as well as to prevent the discharge of oily wastewater into the environment (Mustapha,2018).

The types of wastewater generated in refining petroleum include refinery wastewater, brackish oilfield produced water, heavy-oil produced water, and petroleum hydrocarbon contaminated water, sour water, and produced water and diesel contaminated wastewater. Produced water is the largest volume of wastewater generated by the petroleum industry. It is described as water from an oil well after its separation from oil in American Petroleum Institute (API) separators. Refinery wastewater is the wastewater generated from refining crude oil and manufacturing fuels, lubricants and petroleum intermediates. Other types of petroleum-contaminated wastewaters may include ballast water from ships, storm water and runoff from roads (Ventra et al., 1998).

2.3 Petroleum contaminants:

Petroleum-contaminated wastewaters contain different types of organic and inorganic pollutants with varying levels of contamination .Petroleum wastewater is characterized by a range of pollutants including organics, such as dispersed oil, oil and grease and heavy oil (viscosity > 100 mPas) (Ji et al. 2007), polycyclic aromatic hydrocarbons (PAH), phenols and inorganics such as ammonia (NH_3) and heavy metals. Some crude oils contain small quantities of metals that may require special equipment for refining the crude. In addition, oil and gas may contain Sulphur and carbon dioxide that needs to be removed before marketing. Wastes containing petroleum compounds, nutrients and other toxic compounds should be properly treated prior to discharge into the receiving water bodies because these substances may pose serious hazards to the environment as well as their immediate damages to the organisms. The toxicity of petroleum refinery effluent has been reported in many studies. However, the toxicity depends on a number of factors, including quantity, volume and variability of discharge (Diya alddeen et al., 2011).

2.3.1 Organic pollutants:

The discharge of wastewater with a high organic matter content into the aquatic environment results in the depletion of oxygen Organic pollutants produced by industrial activity such as BTEX (benzene, toluene, ethylbenzene and xylene) (Fountoulakis et al., 2009). Oil and grease (O&G) clog drain pipes and sewer liners, causing unpleasant odors and also corrode sewer lines under anaerobic conditions. In addition, O&G in wastewater can cause depletion of dissolved oxygen and loss of biodiversity in the receiving water bodies. Polycyclic aromatic hydrocarbons are highly toxic, carcinogenic and mutagenic to microorganisms, organisms and humans (Zheng et al., 2013).

2.3.2 Heavy metals:

Metal contamination is a major environmental problem, especially in the aquatic environment. Unlike organic pollutants, metals in wastewater are not degraded through biological processes. Many heavy metals are toxic both in elemental and soluble form. The most toxic metals are Cadmium (Cd), lead (Pb), mercury (Hg), Silver (Ag) and arsenic (As). Several authors have reported the presence of heavy metals in petroleum contaminated wastewater as well as their hazardous effects (Calheiros et al. 2008; Hashim et al. 2011; Qasaimeh et al. 2015).

2.3.3 Nutrients:

Nitrogen and phosphorus compounds in discharged wastewater may adversely contribute to eutrophication, depletion of oxygen and toxicity to humans, aquatic life and bacteria and as well as acceleration of the corrosion of metals and construction materials (Diya alddeen et al., 2011).

2.4 Refinery Wastewater Treatment Processes:

Petroleum refinery waste waters vary in quantity and quality from refinery to refinery, treatment processes include:

- Physical Treatment
- Chemical Treatment
- Biological Treatment

2.4.1 Physical Treatment:

Consists of temporarily holding the wastewater in a quiescent basin where heavy solids can settle to the bottom while oil, grease and lighter solids float to the surface. Raw wastewater inlet big vessel contain screen openings of uniform size is used to remove large solid such as plastics, cloth, polythene etc. which may damage process equipment, reduce the effectiveness of the contaminate streams.

.The settled and floating materials are removed and the remaining liquid may be discharged or subjected to secondary treatment, mostly Physical treatment stage include:

- Screening:

The influent wastewater is strained to remove all large substances carried in the wastewater stream. This is most commonly done with an automated mechanically raked bar screen, a manually cleaned screen may be used. The raking action of a mechanical bar screen is typically paced according to the accumulation on the bar screens and/or flow rate. The solids are collected and later disposed in a landfill or incinerated.

- Grit removal:

Pretreatment may include a sand or grit channel or chamber where the velocity of the incoming wastewater is carefully controlled to allow sand, grit and stones to settle (Nelson, 1969).

- Sedimentation:

In the sedimentation stage, wastewater flows through large tanks, commonly called "primary clarifiers" or "primary sedimentation tanks". The tanks are large enough that sludge can settle and floating material such as grease and oils can rise to the surface and be skimmed off. The main purpose of the primary sedimentation stage is to produce both a generally homogeneous liquid capable of being treated biologically and a sludge that can be separately treated or processed. Primary settling tanks are usually equipped with mechanically driven scrapers that continually drive the collected sludge towards a hopper in the base of the tank from where it can be pumped to further treatment stages (Operation Manual for Water Purifying Treatment Unit, 2004).

2.4.2 Chemical Treatment:

Dissolved air flotation (DAF) is a chemical wastewater treatment process that clarifies wastewaters by the removal of suspended matter such as oil or solids. The removal is achieved by dissolving air in the wastewater under pressure and then releasing the air at atmospheric pressure in a flotation tank or basin. The tank contains chain-type slag skimmer, the coagulant for air flotation uses basic aluminum chloride in addition with demulsifying agent. It is to be added through dosing pump. After mechanical mixing and chemical reaction wastewater enters the separation stage. The tiny oil drops, suspended substances, emulsified oil and other colloidal objects in wastewater attach themselves to floccules and float to liquid surface with the help of released air bubbles, where it may then remove by a skimming device (Operation Manual for Water Purifying Treatment Unit, 2004).

2.4.3 Biological Treatment:

The biological treatment section which uses Anoxic / Aeration (A/O) biological treatment process for serial operation through two stages, the first anoxic stage and the second aerobic stage, activated sludge plants encompass a variety of mechanisms and processes that use dissolved oxygen to promote the growth of biological floe that substantially removes organic material.

The process traps particulate material and can, under ideal conditions, convert ammonia to nitrite and nitrate and ultimately to nitrogen gas.

In aerobic treatment, the wastewater passes through the carbonizing stage, where the COD in wastewater is oxidized into Carbon Oxide (CO^2) and water, and then it passes through the nitrifying stage, where the Nitrogen Ammonia (NH^3-N) in wastewater is oxidized into Nitrogen Oxide (NO^2-N) salt under the action of Nitrosamines and into the Nitrogen Ammonia (NH^3-N) salt under the action of nitrobacteria. The wastewater after nitrification flows back to the anoxic stage for denitrification. With the help of denitrifying bacteria (heterotrophic facultative anaerobic bacteria) in sludge, Nitrogen Ammonia (NH^3-N) is reduced into gas nitrogen (N^2) and released under anoxic atmosphere. Through A/O system treatment, organic substances, ammonia nitrogen and total nitrogen are removed from wastewater. (Operation Manual for Water Purifying Treatment Unit, 2004).

2.5 Discharge of treated wastewater:

The oily slurry from oil separator, Dissolved air flotation ponds and remained activated sludge from biological reaction system is dewatered by centrifuge dry machine. Then it is transported to dumping area to be buried. The waste oil from this plant is recovered by DCU unit. The treated wastewater passes down through a filter, the suspended matter is caught in the pores. When the pressure drop through the filter becomes excessive, the flow through the filter is reversed for removal of the collected solids loading. (The backwash cycle occurs approximately once a day, depending on the loading).Then treated wastewater discharged into oxidation ponds. In the oxidation ponds, there exist certain micro-organisms and some water-plants such as reeds and cattails which can decompose and absorb remaining pollutants. The self-purification effect of oxidation ponds plays an essential role in natural purification eco-system. The water in oxidation ponds is vaporized into atmosphere and irrigated to trees' area around KRC. Through the vaporization and irrigation, the water quantity balance between influent and effluent of oxidation ponds is carried out (Operation Manual for Water Purifying Treatment Unit, 2004).

Figure (2.3) represent typical waste water treatment processes in Khartoum Refinery Company (KRC).

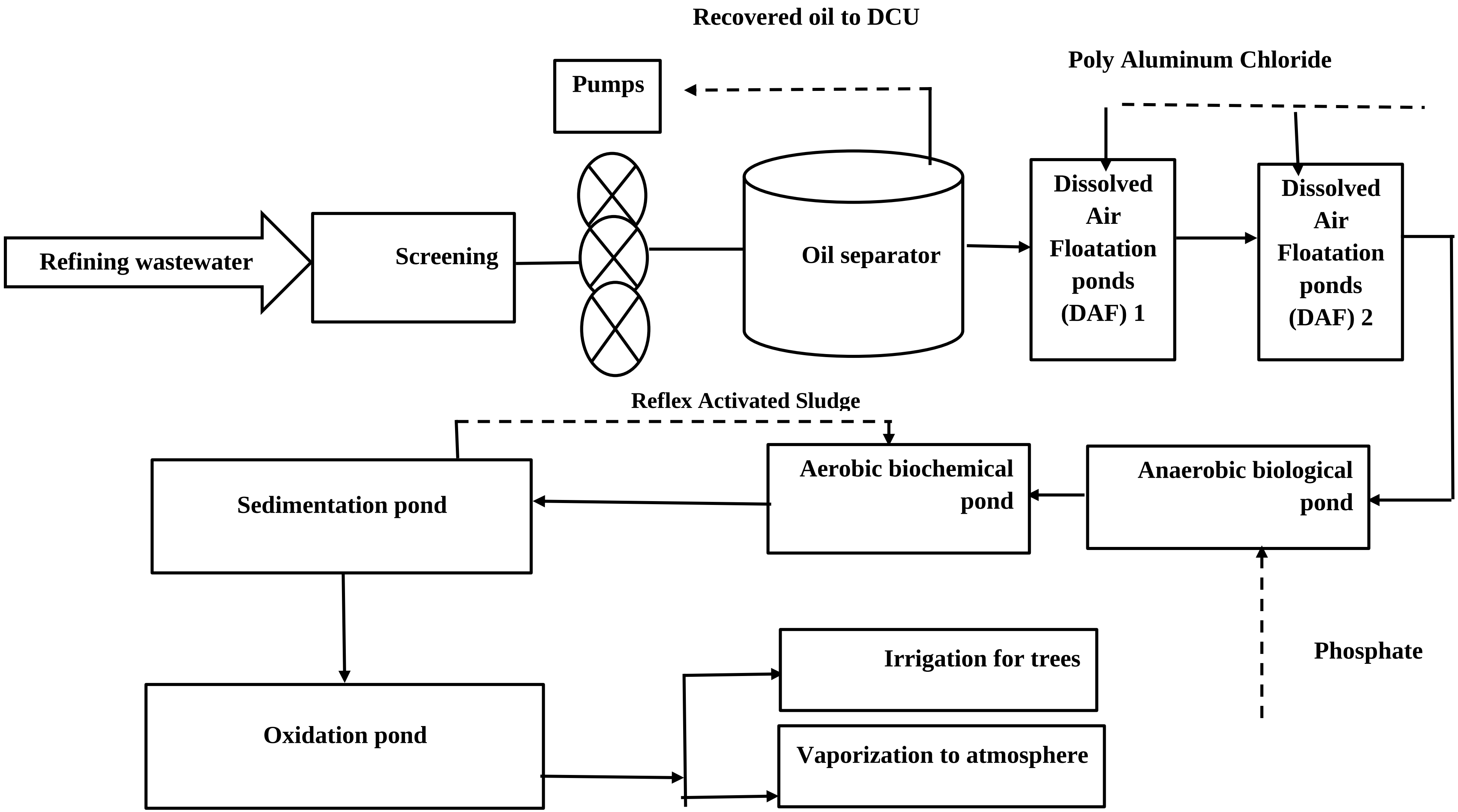

Fig (2.3): waste water treatment process flow chart (Operation Manual for Water Purifying Treatment Unit, 2004)

2.6 Previous Studies:

(Hassan, R, H, 2008) studied treatment of liquid waste resulting from a petroleum refinery and determined the characteristics of liquid waste generated. The sources of wastewater pollutants identified, suitable treatment technologies, and its effect on the environment. Khartoum refinery was considered as a study case, liquid waste produced was studied, the performance of waste treatment unit evaluated. Laboratory tests were conducted to identify the characteristics of wastes in each stage. The effluent drain to the environment was studied and compared with the permissible limits. The analysis carried include: oil content, phenol content, power of Hydrogen, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Ammonia Nitrogen, Sulfide, Dissolved Oxygen, Ortho-phosphate Content, Suspended Solid.

(Hajomer. S.H, 2016) Investigate wastewater in Khartoum Refining and associated pollution problems, the treatment processes of the refinery have been reviewed, Analysis and comparison of treated waste water properties with local and international specifications were conducted. By using standard tests in Khartoum Refinery Company to test pH, COD, BOD, Phenol Content, Oil content, NH_3-N and Suspended Solids Sulfide Content. The results of study were analyzed and compared to the local Sudanese Standards as well as to the International Standard.

(Alnour .Y . A, 2010) A wastewater treatment plant performance has been evaluated after a three years period of work, and the efficiency of the station approximated through the following variables: average removal of the biological oxygen demand, chemical oxygen demand, suspended solids and oils, by taking water samples before and after treatment, every week for two months, analysis of samples was conducted in the central laboratory at Khartoum Refining Company. The determination of the station efficiency revealed that the station is working well, treated water characteristics are in conformity with the specifications set by the World Organization Health. It is also proved to be suitable for use in irrigation.

(Elemam .O. E, 2009) studying of major environmental issues of Khartoum Refinery Company (KRC) were conducted. Studies covered the impact of waste in (gaseous – liquid solid) states. Assessment of the environmental impacts from KRC, alternate methods of waste management in addition to developing the present applied methods. Mobile and online analyzer were used to measure the contents of the released gases (H_2S– SO_2 – CO – NOX). Results show compliance with standard criteria from Sudanese Standard and Metrology Organization specify for refineries. Liquid state wastes from process units were studied at the wastewater treatment plant. Samples were taken from the last stage pre-disposal, analysis of (pH – oil content – SS sulfide -phenol -COD – BOD) showed an increase in COD content, others comply with standards.

(Saad.S. A, 2011) Produced water samples were collected from oil fields and analyzed for chemical and physical properties (BOD, TDS, pH, COD... etc.) and the crude oil content of untreated water was analyzed by Gas Chromatograph. Current treatment practices were studied and evaluated carefully. Chemical and physical composition of samples showed normal environmental values of many parameters (BOD, TDS, pH, COD) but the Sodium Content is high.

(Hammoodi. F ,2012) studied and analyzed oily wastewater characteristics originating from old-processing plant of North Oil Company in Iraqi and to found a suitable and simple method to treat the waste so it can be disposed safely. Oily wastewater pollutants were found to be within Iraqi and EPA standards.

(Li Yu, 2013) summarized development status of treatment methods from six aspects, which contains flotation, coagulation, biological treatment, membrane separation technology, combined technology and advanced oxidation process.

CHAPTER THREE

CHAPTER THREE
MATERIALS AND METHODS

3.1 Introduction

The wastewater treatment unit of Khartoum Oil Refinery was started up in October 1999 after its construction had been completed by China Petroleum Engineering Company. The desired capacity of the unit is 300 m^3/ h, including alkaline containing sewage 40 m^3/h and sulfur containing sewage 37 m3/h. The wastewater after treatment is transported to the oxidation pond outside the plant. Capacity of oxidation pond: 1260X650X0.5m, the designed rate of evaporation: 17.1mm/d. The oily wastewater is pumped into oil separator it is a physical method, effective for removing suspended hydrocarbons. The first step in the removal of hydrocarbons from water is usually by gravity separation. Through properly designated separator tanks with skimmers, most free oil and unstable oil emulsions can be separated from the water. It is the most economical method to remove large amounts of free oil from water after passing the water through large tanks to allow the phases to separate. Then it flows into two stages Dissolved Air Floatation pond (DAF). It is a mechanical and chemical method by using air stripping from compressors to evaporate organic compounds (VOC s) and by adding chemicals poly aluminum chloride (PAC) in order to remove dissolved hydrocarbons. Afterwards, it moves into biological reaction ponds which used to remove low levels of dissolved hydrocarbons from wastewater streams. Biological treatment contains of mixing oxygen and nutrients with water in a tank. The bacteria then destroy the organic compounds as well as alter the chemical form of the heavy metals, Sulphur, phenols, nitrogen and ammonia. And through nitrification and de-nitrification effects, a part of ammonia is turned into nitrogen to atmosphere. Finally, it enters sedimentation ponds where water and activated sludge are separated. Sand or gravel filters are common media used in this process. The oily slurry from oil separator, dissolved air flotation ponds and remained activated sludge from biological reaction system is dewatered by centrifuge dry machine. The waste oil from this plant is recovered by DCU unit. The treated waste water forward to oxidation ponds. The water in oxidation ponds is vaporized into atmosphere (Petroleum Training Center ,2004).

3.2 Field Materials:

In order to evaluate efficiency of wastewater treatment unit in Khartoum refinery, randomly three samples collected from six different points in wastewater treatment unit as shown in figure (3.1).

Fig (3.1): field samples

3.3 Sampling points:

350 ml of wastewater collected from below sample points:

1. House one (inlet).
2. Dissolved Air Flotation.
3. Activated sludge reflux pond.
4. Sedimentation pond.
5. Auto filter.
6. Oxidation Pond (outlet).

3.4 Methodology:

The methodology followed in this research is experimental lab work to measure:

- Phenols.
- Nitrogen and Ammonia.
- Sulfides.
- Total suspended solids (TSS).
- Biological oxygen demand (BOD).
- Chemical oxygen demand (COD).

3.4.1 Hydroxyl Benzene (phenol) content Experiment:

Principle of experiment:

Distil off sample and separate from disturbance matter and fixed agent by distillation. Distilled sample reacts with tetra aminoantipyrine in the medium with PH 10±0.2 and existence of Potassium ferri-Cyanide to produce orange antipyrine dye. After developing color, absorbency will test at wavelength of 510nm with 30mm thick cell, expressed in phenol content mg/l.

Preparation of sample:

0.2g of active Carbon powder which has been activated at 200°C for 30 minutes added to wastewater sample after sufficiently shaking up, then filtered with filter paper of double layers and at medium speed. Sodium hydroxide and drop of Potassium permanganate solution were add to make solution purple, then transferred solution into glass vaporizer for distillation to collect distillate.

Instruments:

- Ultraviolet spectrophotometer.
- Separating funnel = 500ml.
- Funnel diameter is 60mm.
- Measuring flash.

Fig (3.2): Ultraviolet spectrophotometer

Test steps: -

250ml sample transferred into 500ml glass vaporizer, several glass balls were add to prevent bumping, and several drops of (0.5g/l) methyl orange indicator were add. pH Adjusted to equal 4 using Phosphorous acid solution, then added 5ml of Copper sulfate solution . the condenser connected and heated for distillation till 225ml distillate is produced, then heating stopped and cooled , 25ml distilled water which prepared previously were add into distillation flask, then distillation continued till 250ml distillate is produced. 50ml of distillate putted into a 50ml color comparison tube, 0.5ml of Buffer solution with pH= 10.7 added with shaking , 1ml of amonioantipyrine solution added with shake up, 1ml of Potassium ferri-cyanide solution added , shake up sufficiently, then settled for 10 minutes.

At wavelength of 510nm with 20nm cell taking blank reagent as reference, the absorbency of the solution tested (China National Standards -GB/T 7491-1987).

Equation:

Phenol content (mg/l) = $\frac{\text{absorbance (Reading)}*196.15*0.11}{\text{V}}$ **Equation (3.1)**

V≡ sample volume in ml.

3.4.2 Ammonia Nitrogen Experiment:

Principle:

Control the sample pH at the range of 6-7, Magnesia adding to make the sample alkalescent. Ammonia which is distilled off is absorbed by boric of receiver. Methyl red-methylene blue will take as indicator, titrate ammonium of distillate with standard acid solution.

Instruments:

- Distillation flask of 500-800ml
- Blow out prevention nozzle
- Vertical condensation tube

Test steps:

50ml of boric acid (H_3PO_3) indicator solution has been taken into receiver of vaporizer to ensure the outlet of condensation pipes is below boric acid solution level; 50ml of sample putted into the distillation bottle. 3 drops of Bromothymol ($Cr_7H_{28}O_5Br_2S$) (0.5g/l) blue indicator were add, with 1 mol/l Sodium hydroxide or 1% hydrochloric acid, then water added to make the volume of liquid in the distillation flask at about 350ml. Light Magnesia (MgO) was add and a few of explosion proof glass ball into distillation flask, immediately distillation flask was connect with condensation tube. Vaporizer has been heated to make the collecting speed of distillate at about 10ml/m, distillation has been stopped when effluent was about 200ml.

distillate has been titrated with 0.10mol/l standard hydrochloric acid solution to reach purple end point, the consumption (V_1) has been recorded, a blank test has been conducted according to test steps of sample, 250ml of water has been used instead of sample (China National Standards - GB 5750-85-04).

Equation:

$C_N = \frac{(V_1 - V_2)*C*14.01*1000}{V_0}$ **Equation (3.2)**

Where:

V_0 ≡sample volume in ml.

V_1 ≡ volume of standard hydrochloric acid solution consumed for titrating sample in ml.

V_2 ≡ volume of standard hydrochloric acid solution for titrating blank sample in ml. C_N ≡ concentration of standard hydrochloric acid titration solution.

3.4.3 Sulfide Experiment: (Iodimetry Method):

Principle:

Sulfide will be react with Zinc acetate to produce settling of white Zinc. Sulfide dissolves this settling in acid when react with standard Iodine, sodium hyposulfite will be used to titrate the excessive amount of iodine.

Instruments:

- Acid burette. - Iodine flask.
- Vacuum pump, Buckner filter.
- Middle-speed quantitative filter paper.

Test steps:

10ml of 10% Zinc acetate solution and 5ml of 1mol/l Sodium hydroxide solution have been poured into a sample-taking bottle of 250ml, 250ml of water sample has been taken into this bottle to determine sulfide of water sample.

100ml of water sample with immobile liquid, have been filtered using middle-speed filter paper and vacuum. Sediment has been washed with distilled water. Settling and filter paper have been putted into an Iodine flask of 250ml, 50ml of distilled water has been added. Filter paper has been broken up sufficiently using Agitating, 5.0ml of sulfuric acid solution and 10ml f 0.05mol/l Iodine water have been added to flask .flask has been covered with bottle block immediately and sealed with distilled water, settled in a dark place for 5 minutes, then titrated with 0.05mol/l sodium hyposulfite to light yellow, 1ml of 1% fecula indicator has been added, titration has been continued till blue disappears completely. Consumption V2 (ml) of Sodium hyposulfite has been recorded, blank test has been made in the same way. (China National Standards - GB 5750-85-08)

Equation:

$$S^{-2}\ (\text{ml/l}) = \frac{(V_1 - V_2) * C * 14.01 * 1000}{V} \qquad \textbf{Equation (3.3)}$$

Where:

S^{-2} ≡ Sulfide concentration in mol/l.

V_1 ≡ volume of Sodium hyposulfite standard solution for blank test (ml).

V_2 ≡ volume of Sodium hyposulfite for titrating water sample (ml).

V ≡ volume of water sample (ml).

3.4.4 Determination of Suspended Solid Experiment (Weighting Method):

Principle:

Filtration a proper amount of water and then drying the residue to constant weight which is the content of suspended matter.

Instruments:

- Analysis balance: reciprocal sensibility of 0.001mg.
- Drying box with constant temperature.
- Dryer.
- Glass funnel.
- Measuring bottle: high type.
- Low-speed filter paper.

Analysis steps: -

A filter paper has been pleated into cross shape, then it has been rolled into a cylinder and putted into a weighting bottle, then putted in an oven of 105 ± 5°C dry to constant weight. Water sample of 100-1000ml with a sampling bottle has been taken and putted all of water samples into measuring cylinder and record its volume. Filter paper, which has constant weight out of the weighting bottle has been putted into a funnel. Moisture with little amount of water to make it attached to the funnel. water sample has been filtered, then sampling bottle and measuring cylinder have been washed with little amount of water, wash water has been poured into the filter paper for filtering, When the water drips have been disappeared from funnel, filter paper has been taken out carefully and pleated then it has been putted into the original weighting bottle, it has been dried to constant weight according to the conditions stipulated. (China National Standards - GB 5750-85-09).

Equation:

$$X = \frac{(W_2 - W_1) * 1000 * 1000}{V_W} \qquad \textbf{Equation (3.4)}$$

Where;

X≡ suspended matter of water sample (mg/l).

W_1≡ weight of weighting bottle and filter paper in (g).

W_2 ≡ weight of weighting bottle, filter paper and suspended matter in (g).

V_w ≡ volume of water sample in (ml).

3.4.5 Biological Oxygen Demand (BOD) Experiment (Dilution Method):

Principle:

Determination of BOD by measuring the rate of oxygen uptake by micro-organisms in a sample of water at a fixed temperature (20°C) and over a given period of time in the dark.

Instruments:

- Respirometer and incubator.
- Amber bottles with seal caps.
- Stopcock grease.
- Magnetic stirrers.

➢ Reagents:

- Lithium hydroxide, or potassium hydroxide pellets.

Analysis steps:

The incubator's temperature has been adjusted at (19-21)℃, a magnetic stirrer has been putted in the clean empty bottle. The bottle has been closed using grease in the cup. (1-2) pellets of the hydroxide have been putted in the cup carefully. The screw of the sample bottle's cap have been fasten .the track has been turned on and BOD range selected. The final reading has been taken after 120 hours (5 days) (ASTM D6238).

3.4.6 Chemical Oxygen Demand (COD) Experiment (Titration Method):

Principle:

These test methods cover the determination of the quantity of oxygen that certain impurities in water will consume.

Instruments:

- Reflux apparatus.
- Round bottom flask.
- 500-ml Erlenmeyer
- Volumetric cylinder.
- Glass beads.

Analysis steps:

20ml of sample has been placed in round bottom flask; 0.4ml of mercuric sulphate, and 10ml potassium dichromate solution have been added. Carefully 30ml sulphric acid –silver sulphate solution have been added with mixing after each addition. A few of clean glass beads have been added to prevent bumping and attack the flask to condenser. Then mixture has been refluxed for 2 h Cool, and the condenser has been washed with approximately 25ml of distilled water. The content has been transferred to 500-ml Erlenmeyer flask, the reflux flask has been washed 4 to 5 times with distilled water. The mixture has been diluted to approximately 140ml. The excess dichromate has been cooled and titrated with ferrous ammonium sulphate solution, (2 or 3) drops of the ferroin indicator has been added. The sharp color change from blue –green to brown has been taken as end point. 20ml of distilled water instead of the sample, together with the reagent, has been refluxed and titrated in the same manner (ASTM D1252).

Equation:

COD mg/l = $\frac{(A-B)*C*800}{\text{sample volume}-\text{ml}}$ **Equation (3.5)**

Where:

A≡ ferrous ammonium sulphate solution used for distilled water in ml.

B≡ ferrous ammonium sulphate solution used for sample in ml.

C≡ Normality of ferrous ammonium sulphate solution.

CHAPTER FOUR

CHAPTER FOUR
RESULTS AND DISCUSSION

4.1 Results

Table (4-1) represents results of three samples from each sample point in treatment system.

Table (4.1): Results of Samples

Samples		**phenol content/mg/L**	**Ammonia Nitrogen/ mg/L**	**Sulfide/ mg/L**	**Total Suspended Solids/ mg/L**	**Biological Oxygen Demand (BOD) / mg/L**	**Chemical Oxygen Demand (COD)/ mg/L**
House one	**Sample I**	45.90	04.90	18.80	130.0	28.00	860.0
	Sample II	40.83	03.50	08.56	72.00	39.00	800.0
	Sample III	54.66	04.90	08.24	150.0	32.00	886.0
Dissolved Air Flotation (DAF2)	**Sample I**	30.57	05.50	14.32	80.00	29.00	449.0
	Sample II	53.91	02.90	11.60	96.00	42.00	770.0
	Sample III	55.35	03.90	08.40	120.0	35.00	920.0
Activated sludge reflux pond	**Sample I**	34.90	05.30	12.64	120.0	32.00	660.0
	Sample II	42.30	03.70	12.32	130.0	39.00	900.0
	Sample III	02.50	03.70	13.10	130.0	27.00	150.0
Sedimentation pond	**Sample I**	01.80	03.70	10.30	88.00	34.00	173.0
	Sample II	01.11	02.60	08.90	118.0	44.00	144.0
	Sample III	00.74	02.50	10.80	130.0	25.00	100.0
Auto filter	**Sample I**	00.69	02.40	08.80	44.00	29.00	145.0
	Sample II	00.42	02.30	07.20	109.0	46.00	80.00
	Sample III	00.17	02.20	08.16	67.00	36.00	130.0
Oxidation Pond	**Sample I**	00.30	01.20	07.60	52.00	32.00	120.0
	Sample II	00.13	02.30	06.50	50.00	40.00	148.0
	Sample III	00.20	01.60	08.60	28.00	28.00	160.0

The results of the study include the numbers obtained from laboratory tests conducted on wastewater samples from the water treatment plant in the Khartoum Refinery, and all experiments were conducted in the Environment Research Laboratory, the reference for the Ministry of Environment, and the location in Al-amarat Street No. 57.

The relation between samples results from each sample point investigated using simple charts which show the trended relation between the samples for each parameter. The next figures describe relation between samples results for each analyzed parameter.

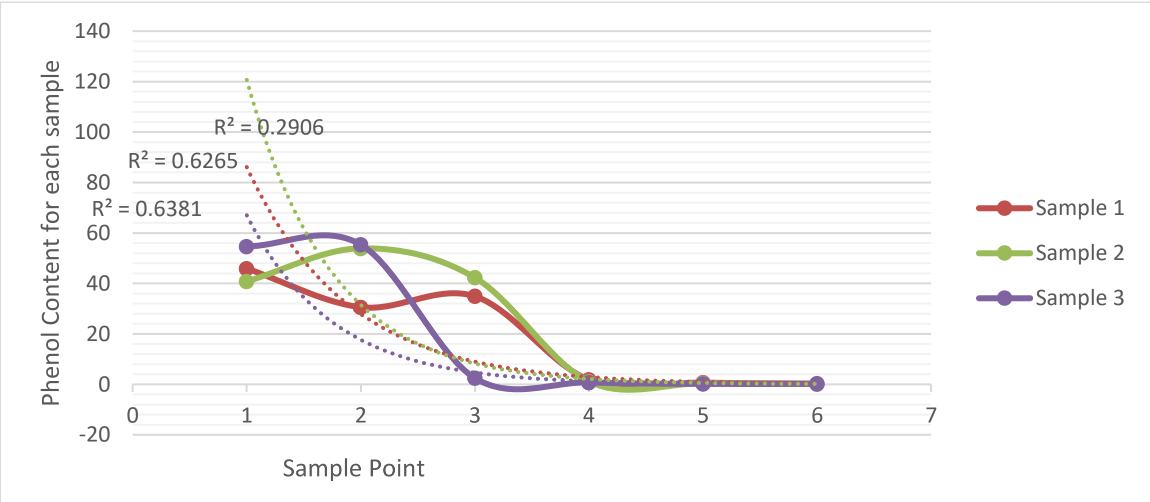

Figure (4.1): Relation between results of the Phenol Content in samples

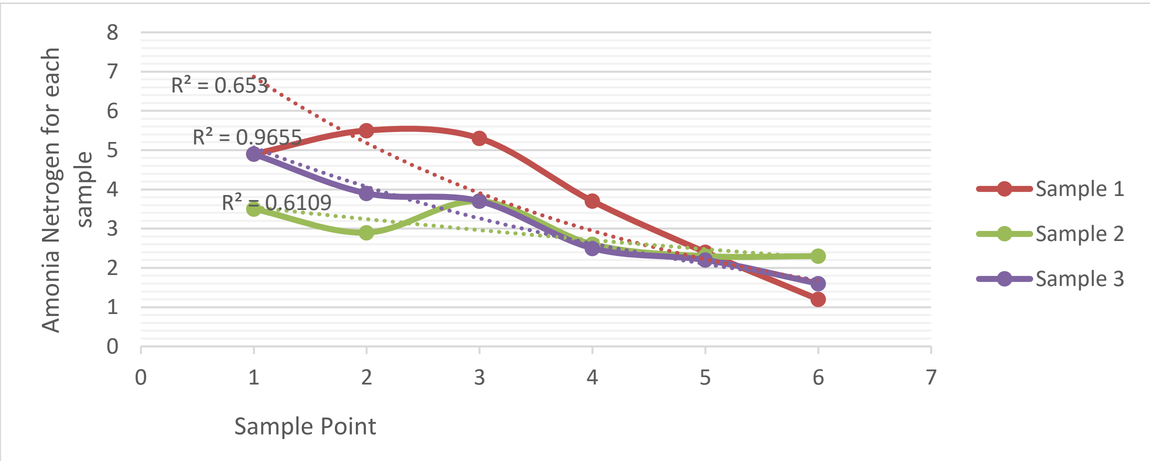

Figure (4.2): Relation between results of the Ammonia Nitrogen in samples

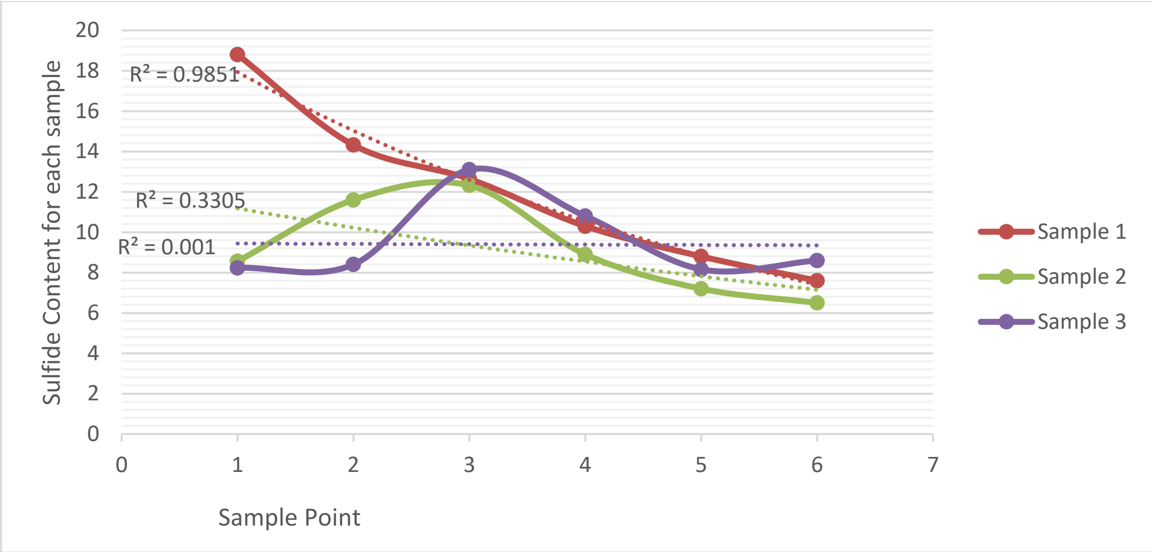

Figure (4.3): Relation between results of the Sulfide Content in samples

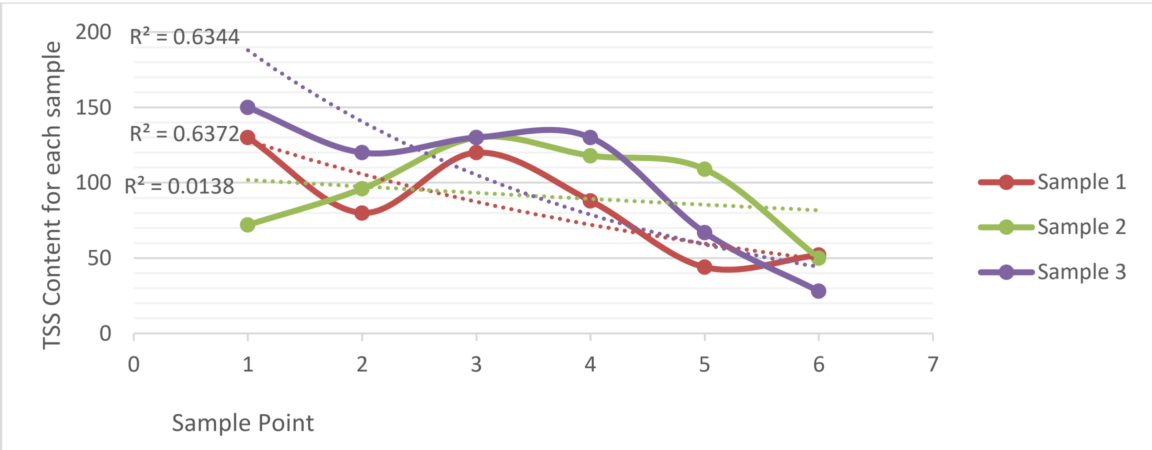

Figure (4.4): Relation between results of the Total Suspended Solids in samples

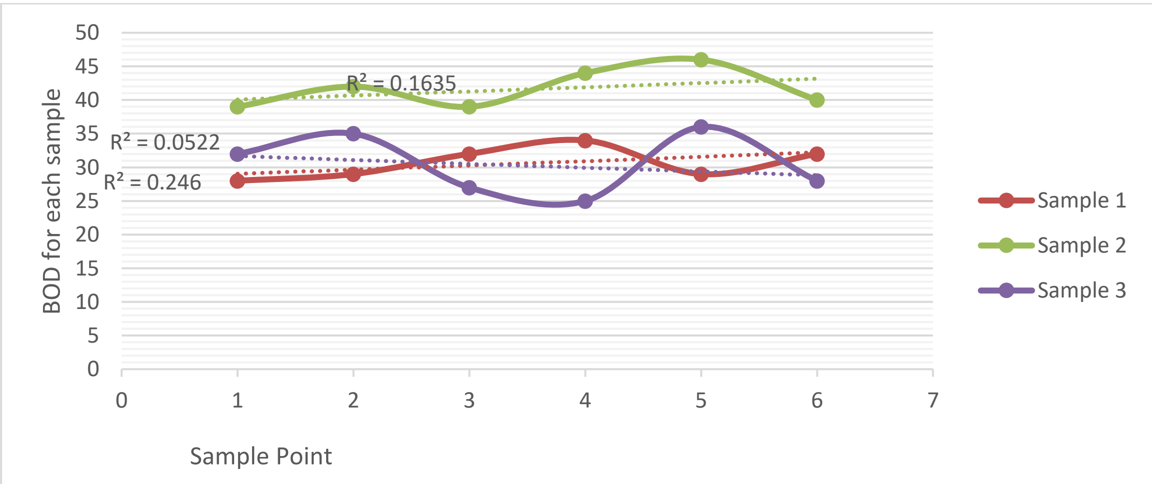

Figure (4.5): Relation between results of the Biological Oxygen Demand in samples

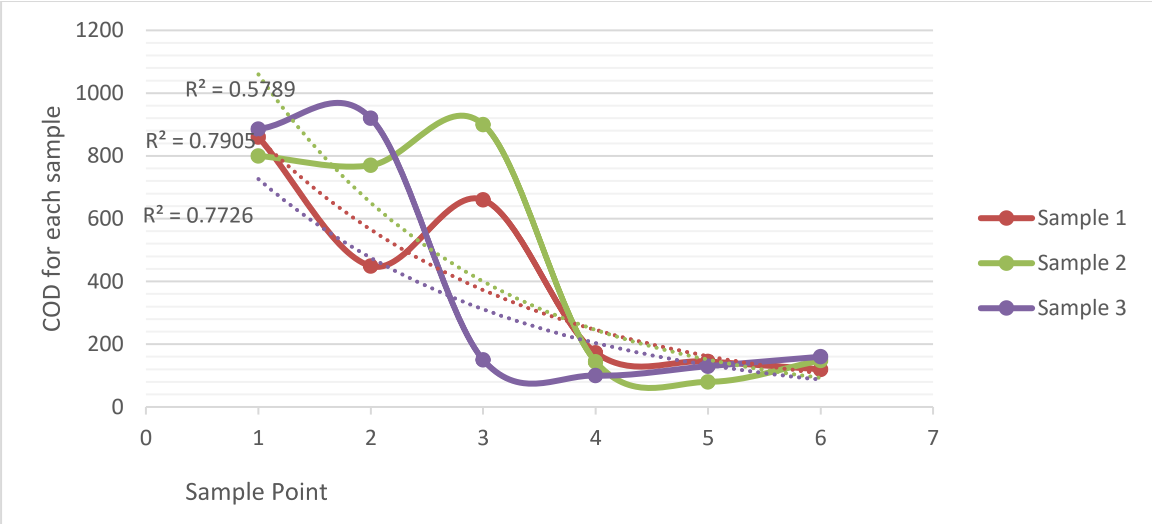

Figure (4.6): Relation between results of chemical Oxygen Demand (COD) in samples

The Average results of all samples calculated using Simple Arithmetic Mean and compared with Chinese Standards and the Sudanese Standards limits of Liquid Wastes Resulting from Petroleum Refining, the comparison represented in table (4-2).

Table (4.2): The Average Results of all Samples vs. Chinese Standards and the Sudanese Standards

Parameter	**Average Results of Samples**						**Maximum limit in Chinese standards**	**Maximum limit in Sudanese Standards**
	House one	**Dissolved Air Flotation (DAF2)**	**Activated sludge reflux pond**	**Sediment-ation pond**	**Auto filter**	**Oxidation Pond**		
phenol content/mg/L	47.10	46.60	26.60	01.20	00.40	00.20	00.50	10.00
Ammonia Nitrogen/ mg/L	4.40	4.10	04.20	02.90	02.30	01.70	10.00	15.00
Sulfide/mg/l	11.90	11.40	12.70	10.00	08.10	07.60	01.00	01.00
Total Suspended Solid/ mg/L	117.30	98.70	126.7	112.0	73.30	43.30	30.00	70.00
Biological Oxygen Demand (BOD) / mg/L	33.00	35.30	32.70	34.30	37.00	33.30	30.00	30.00
Chemical Oxygen Demand (COD)/ mg/L	848.7	713.0	570.0	139.0	118.3	142.7	150.0	100.0

The average results of all Samples represented VS. Chinese Standards and the Sudanese Standards limits in next figures.

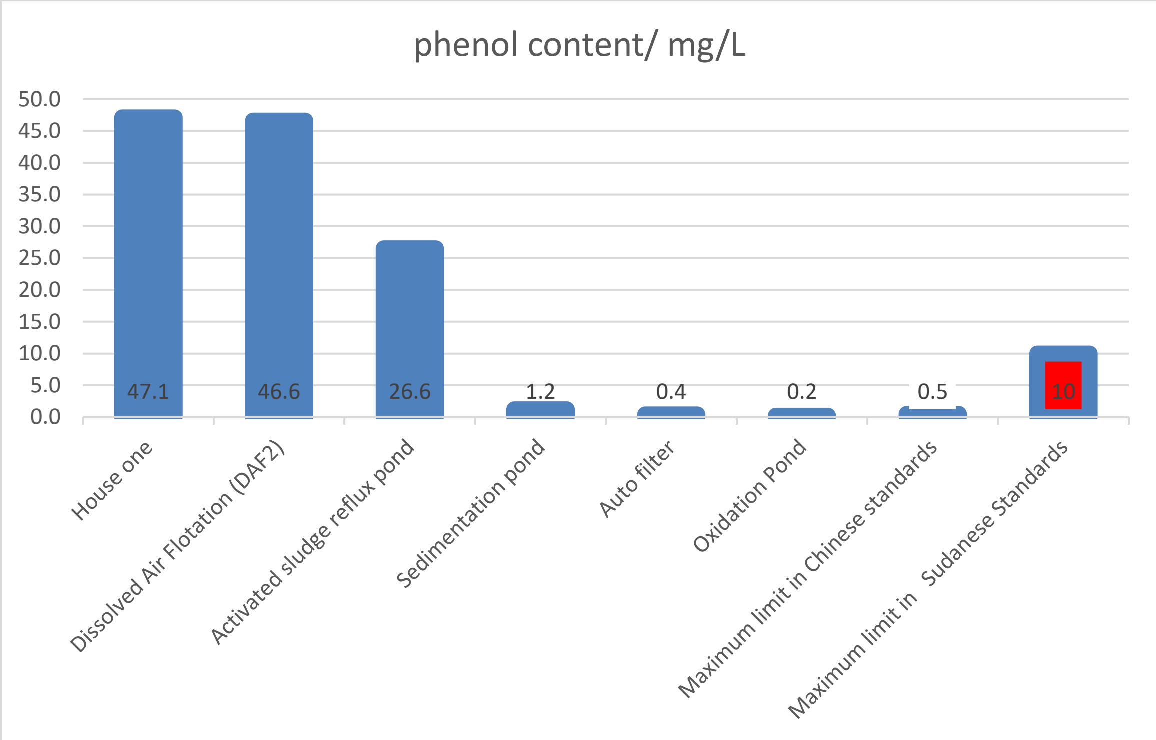

Figure (4.7): Average phenol content VS. Chinese Standards and the Sudanese Standards limits

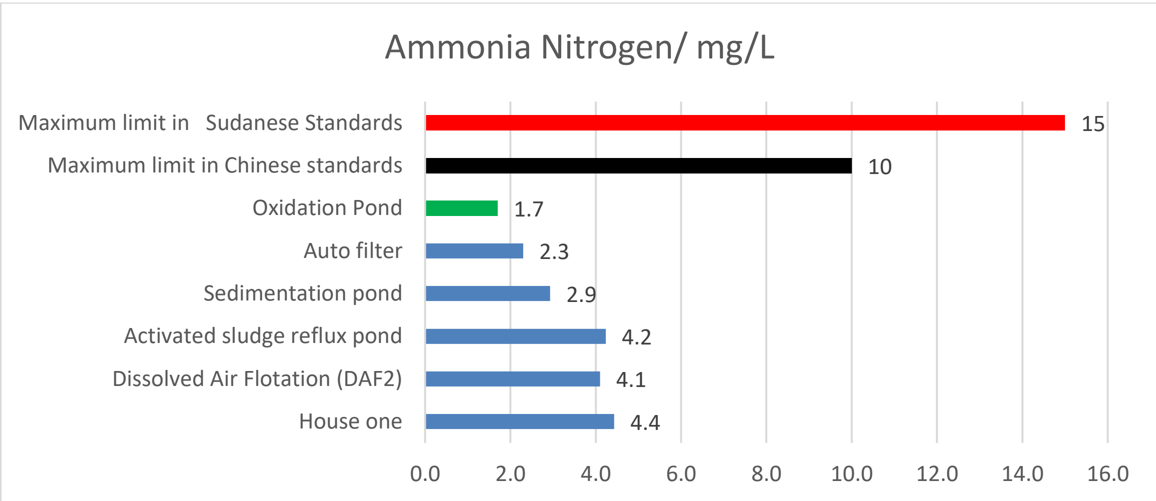

Figure (4.8): Average of Ammonia Nitrogen VS. Chinese Standards and the Sudanese Standards limits

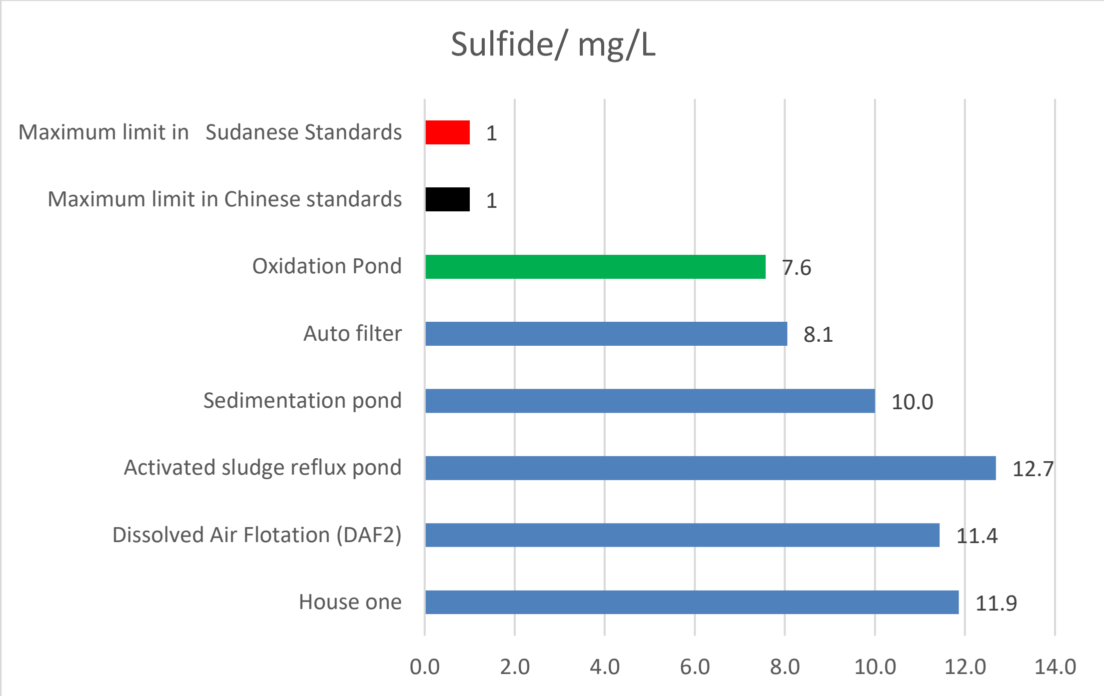

Figure (4.9): Average of Sulfide content VS. Chinese Standards and the Sudanese Standards limits

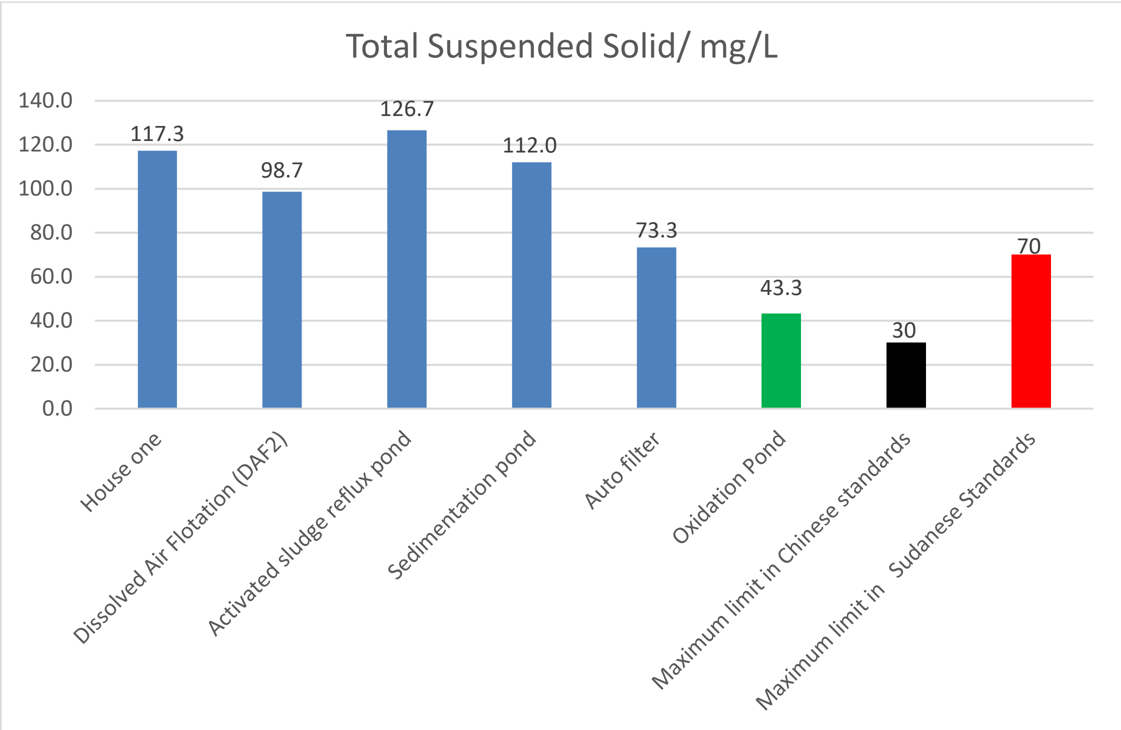

Figure (4.10): Average of Total Suspended Solid VS. Chinese Standards and the Sudanese Standards limits

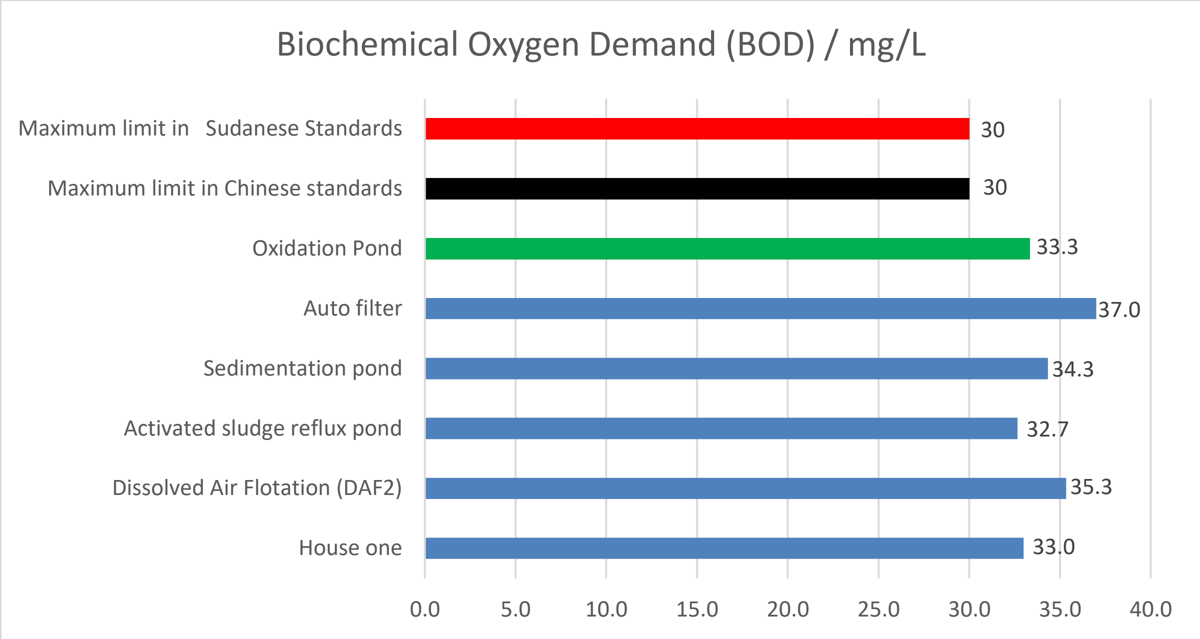

Figure (4.11): Average of Biological Oxygen Demand (BOD) VS. Chinese Standards and the Sudanese Standards limits

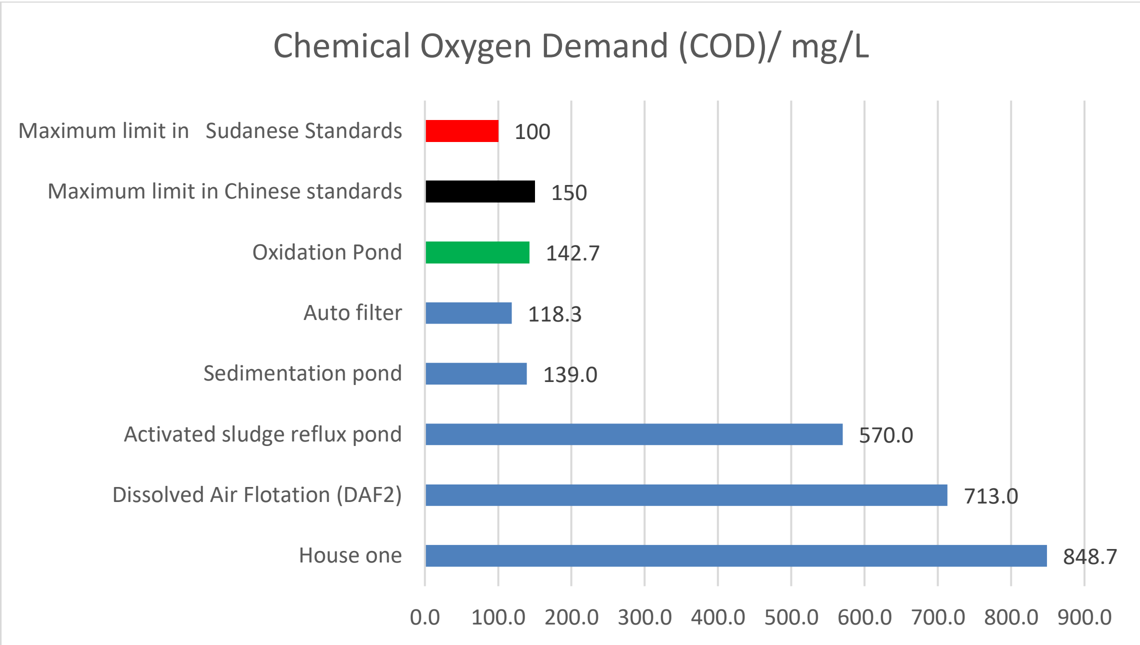

Figure (4.12): Average of chemical Oxygen Demand (COD) VS. Chinese Standards and the Sudanese Standards limits

4.2 Discussions

- ❖ The change of parameters through treatment system will be discussed in the light of the Chinese Standards and the Sudanese Standards limits of Liquid Wastes Resulting from Petroleum Refining using average result of samples. The analysis of samples and their comparison with local and international standards were done and it is noticed that:
- ❖ Phenol Content: The Phenol Content values of wastewater are reduced 47.1 mg/l at House one (inlet) to 0.2 mg/l at Oxidation Pond through treatment system, so that the system performance was significantly effective by decreasing the phenol concentration.
- ❖ Ammonia Nitrogen: The ammonia Nitrogen values of wastewater are reduced from 4.4 mg/l at House one (inlet) to 1.7 mg/l at Oxidation Pond through treatment system, which showing ammonia removal efficiency.
- ❖ Sulfide Content: Several problems arise from high sulfate concentrations in water such as corrosion of water transport systems and of concrete structure. The sulfide values of wastewater reduced from 12.7 mg/l at Activated sludge reflux pond to 7.6 mg/l at Oxidation Pond, which is not within permissible limits of Sudanese and International standards due to conversion of sulfates to hydrogen sulfide by bacteria under anaerobic conditions which require more consideration to enhance treatment processes.
- ❖ Suspended Solids: The Suspended Solids values of wastewater are reduced from 126.7 mg/l at Activated sludge reflux pond to 43 mg/l at Oxidation Pond which is not within permissible limits of Chinese standards although it acceptable to Sudanese standards .
- ❖ Biological Oxygen Demand: The BOD values of wastewater are reduced from 37 mg/l at Auto filter to 33.3 mg/l at Oxidation Pond which is not within permissible limits of Sudanese and International standards. According to these results, it can be inferred that biological reactions in the anaerobic pond can convert particularly cyclic hydrocarbons such as phenol, to biodegradable organic compounds. Indeed, in such process the possibility of increasing the BOD is provided. Therefore, the BOD values increased in the system and thus it seemed that the BOD removal efficiency in anaerobic ponds were low. In other words,

removal of BOD in facultative and aerobic ponds was higher than in anaerobic ponds.

- Chemical Oxygen Demand: The COD values of waste water are reduced on the average from 848.7 mg/l at House one (inlet) to 142.7 mg/l at Oxidation Pond this value is satisfying the Sudanese and International standards , reduction of COD was mainly due to the degradation of most of the organic matter in refinery wastewater.

CHAPTER FIVE

Chapter Five
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The study is concluded to:

- The average results of oxidation pond (discharge point) samples were compatible with the Chinese standards and the Sudanese Standards in: Phenol Content; ammonia Nitrogen. However, the results were in agreement with one of the two standards in sulfides; Suspended Solid; Biological Oxygen Demand; Chemical Oxygen Demand.
- The results obviously showed low effectiveness of the treatment methods adopted by the refinery to decrease levels of the contaminants in treated wastewaters to the allowable, harmless concentrations.
- The graphic representation of the sampling results showed an acceptable relation between the samples taken from the six sampling points, which indicates the regularity of the processing stages of all the quantities entering the treatment plant.

5.2 Recommendations

In practice, the attention should be paid to the use of chemicals in refinery wastewater treatment, after this study, analysis and review of the existing methods of treatment in the refinery carried out and it is recommended to:

- ❖ More development required in term of sulfide removal in raw wastewater in order to prevent the damages of sulfide to treatment processes such as corrosion of concrete sewer pipes, release of obnoxious odors to the urban atmosphere, safety hazards to sewer workers due to the toxicity of sulfide gas.
- ❖ Re-habitation of mechanical cleanings for top water at sedimentation ponds to enhance performance of auto filters that follow sedimentation stage.
- ❖ More research and developments should be carried out to adopt a new combined treatment processes for Khartoum refinery wastewater, to maximize the efficiency with decreasing cost of treatment.
- ❖ Samples from treatment plant should be subjected semiannually to be analysis by second party laboratory to ensure the accuracy of the Khartoum Refinery Company (KRC) laboratory.

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