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Determination of Metal ions, Phenols, and NO3-, NO2- in Industrial Wastewater of Al-Dura and Al-Najaf Refinery

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Abstract--- *Determination of the proportion of selected metal elements (Fe, Mn, Co, Cu, Cr, Ni, Mo, Pb, Cd) using a flame absorption spectroscopy device and the spectroscopy of Graphite furnace, and Phenols so as Free Radical (NO3-, NO2-) using ultraviolet spectroscopy device in the industrial wastewater of Al-Dura (A) and Al-Najaf (B) refinery before and after entering the treatment units, taking into consideration the sampling time Varying (more than separated) and setting the temperature during the drawing of the model and conducting a statistical study of the results reached.*

Keywords--- *Heavy Metal, Industrial Wastewater, Al-Dura Refinery, Treatment, Al-Najaf Refinery.*

I. INTRODUCTION

At present, water studies are one of the basic necessities that must be done because the quality of water has become limited in quantity and quality of agricultural production. The main purpose of the treatment process is to obtain treated water to the extent that the risks to public health and the environment are at an appropriate and acceptable level. Regardless of the type of treatment plant, it reduces suspended solids and removes chemical contents that may have adverse effects on crops. As well as biological components (pathogens), which are the main sources of concern for public health (1, 2).The contaminated water wastewater has led over time to many problems, namely environmental pollution, as the pollution of the Tigris and Euphrates rivers has increased, organic and inorganic pollutants and toxic substances, and hence its significant impact on the water and biological balance and its harmful effects on the health of Human, from which it has been proven that fish that live in water contaminated with lead compounds, contain in their bodies lead compounds with dangerous concentrations that harm the health of those who eat them. (3).What causes water pollution is the entry of organic matter, chemicals, fats, nitrogen, fertilizer, insecticides, and other materials. Wastewater from the inorganic chemical and oil industries leads to special problems in processing and subtraction as some of these special chemicals cause the taste and smell of water and are difficult to remove(4).

Pollution of Water with Heavy Metals, Phenols and Free Radical

Heavy metals are commonly used in industry and toxicity generally affects animals and aerobic and anaerobic processes, all of which form a number of undesirable properties that affect humans and the environment, they form non-biodegradable chemical compounds (5). Because of its high solubility in aquatic environments, organisms can

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absorb heavy metals; once the food chain is entered, large concentrations of heavy metals may accumulate in the human body (6). Nitrogen is found naturally in groundwater and rainwater (7).Nitrates do not affect dangerously or healthily unless they are exceeded to the natural limit or converted to nitrite (8). Nitrite is composed of oxygen oxidation of ammonium by nitrite bacteria; it is an intermediate compound for the complete oxidation of nitrogen to nitrate (9) the presence of nitrite as a result of nitrate reduction and then converted by nitrosamines, which in turn be carcinogenic (8). Phenols are widely found in oil refineries, coal-fired plants, petrochemicals, polymeric resins, coal distillation, pharmaceuticals, insecticides, textile industry (10). Because of their tendency to oxidize easily to the quin one roots, which tend to be more reactive, catechol tends to cause DNA damage, destroy some proteins in the body and disrupt the transfer of electrons in membranes that transmit energy (11).

II. STUDY AREA

AL-Dura refinery lies at the Eastern South township of Baghdad, in the close of Tigris River. The total area of the refinery is 250 ha .The abundant palm trees are nearby to the refinery along the rim of the river. It’s one of the old petroleum refineries in Iraq (12) Fig (1). AL-Najaf refinery is located north of the province of Najaf within the administrative borders of Al-Haidariyah within the Middle Euphrates region. The refinery was founded in 2005 with a capacity of 320 dunums Fig (2). Wastewater Refineries can generate a significant amount of wastewater that has been in contact with hydrocarbons. Wastewater can also include water rejected from boiler feed water pretreatment processes (or generated during regenerations). Wastewater can also refer to cooling tower blow downstream, or even once-through cooling water that leaves the refinery. Once-through cooling water typically does not receive any treatment before discharge. Cooling tower blow down water and wastewater from raw water treating may or may not receive treatment at the wastewater treatment plant (WWTP) before discharge. Contaminated wastewater is typically sent to either a wastewater treatment plant that is located at the facility, or it can be pretreated and sent to the local publicly owned treatment works or Third-party treatment facility for further treatment. Water that has not been in direct contact with hydrocarbons or which has only minimal Contamination can be a source for reuse and is discussed in the section on ‘Recycle and reuse Typically, a purge stream is removed from the water purification systems in order to prevent the buildup of contaminants. This purge stream is sent to wastewater treatment and is replaced by fresh make up water (13.14).

Figure 1: Study area Al-Dura Refinery

Figure 2: Study area Al-Najaf Refinery

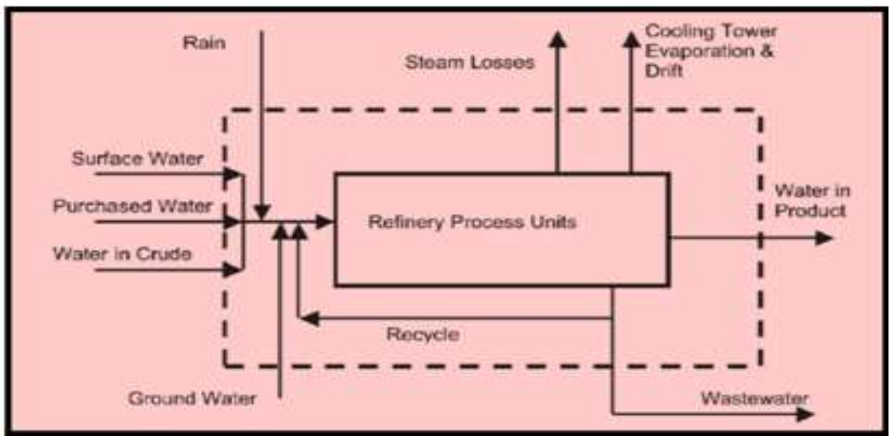

Figure 3: Shows a Typical Example of the Water Balance in a Refinery

III. WASTEWATER TREATMENT PROCESS

The amount of wastewater discharged (750) cubic meters / hour and the type of liquid waste is sewage, industrial and also cooling water.(3) Industrial water is treated in several stages:

1. The Initial Treatment is Called (Mechanical Processing Stage): Water is treated at this stage by removing floating oil, oils and sediments heavier than water according to the principle of density difference between different oil substances in contaminated water and then withdraw the remaining water and send it to the next stage. The main purpose of this process is to remove hydrocarbons (HC) and a part of the emulsified hydrocarbons. The resulting water will be sufficiently free of oil to go to the physiochemical stage. The other purpose of this process is to remove sand and gravel, which can hamper the assembly of oil minutes in the physiochemical process (15-17).

2. Secondary Treatment Called (Physico-Chemical Processing Phase): This stage can be defined as a combination of the first two processes of the Flocculation process by the addition of chemicals and the second process of Floc Sludge Physical Separation consisting of the first process and the separation either by Filtration or Flotation. Objectives of the physiochemical process: Reduction and reduction of Hydrocarbon in water, Deposition

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of heavy toxic metals, Oxidation and Sedimentation Sulfur by iron salts, Water Treatment (17-19).

3. Biological Phase and Final Settling Basin: It is one of the most important stages of treatment wastewater because it achieves the basic purpose of water treatment before putting it to the river is the removal of toxic substances and affect the health of human beings and these organisms and most of organic solvents used in the units of refineries such as (furfural and phenol and aldehyde) we use bacteria to get rid of these substances Which is a food for these organisms are analyzed and oxidized and converted into solid and non-toxic materials under certain conditions of the degree and pH and the proportion of contaminants of electromagnetic radiation that occurs in atoms, molecules and other chemical species (20, 21).

Absorption or emission is associated with changes in the energy status of the reactive chemical species that characterize them, which is why spectroscopy can be used in qualitative and quantitative analysis (22, 23). Measuring atomic absorption spectrum (AAS) is an analytical method for estimating the amount of more than 70 different elements in a solution or solid samples. The procedure is based on the dissolution of elements by various decomposition techniques such as flame (FAAS), electro thermal (ETAAS), hydride or cold vapor and each decomposition technique has its advantages and binds it (24), at FAAS group we will deal only with optical spectroscopy. In optical spectroscopy, light is dissolved on components that have different wavelengths. flame are used in this technique is air flame/acetylene, type of flame depends on the thermal stability of the analyzed material and forms its potential compounds with the accompanying flame(25). Temperature formed in the acetylene flame in the air is about 2300 degrees Celsius (26, 30). GF-AAS atomic absorb spectrometer is a sensitive and precise technique. The advantages of GF-AAS include shorter sample preparation time, increased sample productivity from simultaneous multi-element analysis, reduced amount of chemical waste, reduced sample size requirements, increased linear focus range, and use of a more precise digestion procedure (26-28).

IV. MATERIALS & METHODS

Apparatus: All spectrophotometric measurements were performed using Atomic Absorption spectroscopy AA-7000 (Shimadzu Company) and Graphite furnace GFA-7000A (Shimadzu Company).At ministry of science and technology.

Sample Collection and Sample Preparation: 34 water samples were collected (10 water samples before treatment, 12 water samples after treatment from each basin in Al-Dura refinery) and (6 water samples were collected before and after treatment from each basin in Al- Najaf refinery).from November 2018 to March 2019. Two liters of the water samples collected from Refinery into a plastic bottle that had been pre-washed with detergent and de ionized water. Each sampling point and identification labels were fixed on each water sample collected. The samples of heavy metals were acidified with concentrated nitric acid (29).

Standard Solution: Original standard solutions were (1000µg/ml in 2% HNO_3) for interested elements. Four standard solutions were prepared by dilution from original standard stock solution using general dilution law ($N_1 V_1 = N_2 V_2$). It must prepare a series of concentrations from the highest one reaching the values required for calibration curve performs. Series of concentrations which prepared were as follows:- (1000µg/ml → 100µg/ml → 10µg/ml → 1µg/ml). The value 1µg/ml is equal to 1000ng/ml, so complete preparation from this solution reaching the

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required concentration of the standard calibration curve as follow:- (1000ng/ml → 100ng/ml → 5ng/ml, 10ng/ml, 20ng/ml, 30ng/ml)

Trust Test (T-Test): Statistical used to know if there are different between result or not. In order to establish a comparison between the two kinds of elements, phenol, and free radical (i.e untreated and treated), an individual t-test was done. The tow hypotheses are:

1. Null hypothesis H_0:$\mu_{untreated} = \mu_{treated}$ there is no difference between the treated and untreated samples
2. Alternative hypothesis H_1 : $\mu_{untreated} \neq \mu_{treated}$ indicates that the means (D) are not the same, where H_1 is the alternative probability. Therefore the acceptance or rejection may be according to the two probabilities below. Either: $|t| < t_{(0.025),n}$ .accept H_0 which includes that the means of the two types of beads are insignificant.

Or another probability $|t| > t_{(0.025),n}$. reject H_0 and accept H_1 that the means of two types of beads are different from each other.

The Equation applied to calculate confidence is:

$$\pm t = \frac{\mu - D}{S} * \sqrt{n}$$

μ=Mean of sample before treatment, **D**=Mean of sample after treatment,

S=Stander deviation before treatment, **n**=total number of samples.

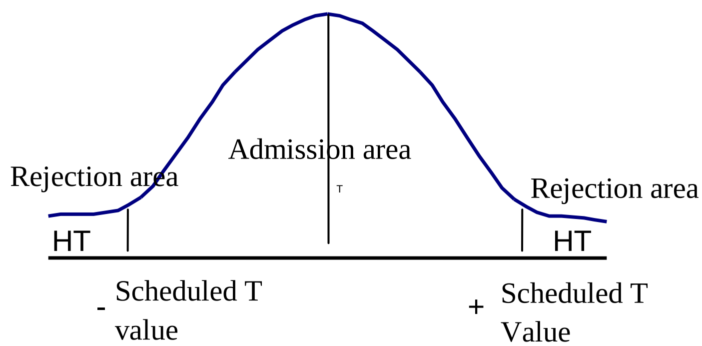

Table 1: The Range and Equation Used to Measure the Standard Solutions of Elements

Ion	**L-R***	**R****	**r²**	**r %**	**Equation**
Fe	20(ppb)	1	1	100	Abs=0.00469Conc-0.01315
Mn	4(ppb)	0.9981	0.9962	998.1	Abs=0.05846Conc+0.0059910
Cr	20(ppb)	0.9997	0.9994	999.7	Abs=0.013571Conc-0.006715
Co	20(ppb)	0.9998	0.9996	999.8	Abs=0.002524Conc-0.088135
Ni	4(ppb)	0.9982	0.9964	998.2	Abs=0.18541Conc+0.02515
Cu	20(ppb)	0.9990	0.998	999	Abs=0.010759Conc+0.00685
Mo	1 (ppm)	0.9993	0.9986	999.3	Abs= 0.52315Conc+0.026356
Pb	20(ppb)	0.9991	0.9982	999.1	Abs=0.020056Conc+0.0263
Cd	0.8(ppm)	0.9996	0.9994	999.6	Abs=0.52103Con+0.025687
L-R**=Linear Rang ,*R=** rang					

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V. RESULTS & DISCUSSION

In Table (2) that there is a clear difference in the results of the area (A) after treatment as there is a 63% treatment rate for contaminated water. It was also found that the ratio of iron and manganese elements is high and sensitive to the device of the flame atomic absorption spectrometer FAAS, while the rest of the element did not show any results in this mechanism so it was sensitive by the spectrometer of GFAAS. It was noted that the lead rate is high compared to the rest of the elements and the cobalt ratio is non-existent or below the specified level, but is generally within the limits of environmental parameters. The results of the region (B) were sensitive to the FAAS and treatment rate of 69%, the concentrations on the river are still higher than the level of environmental parameters.

In the Table(3) correlation coefficient for the elements (Fe, Mn, Ni, and NO2-) with a negative signal, that means inverse relationship between the concentration of elements before and after treatment and its ratio between (0.02-0.3) that means the efficiency of the processor for these elements is relatively weak. Elements (Pb, Cr) the correlation coefficient (0.6-0.7) means that the processor efficiency of these elements is very strong. By comparing T calculated of samples (Mn,Phenols,NO3-) with a scheduled value T(0.025),n-1, we find that the calculated value is higher than the scheduled, i.e. outside the point of acceptance of the hypothesis theoryH0, i.e. in the alternative rejection area. The theory rejects the hypothesis H0:µuntreated = µtreated and accepts the alternative theory H1: µuntreated ≠ µtreated with a probability of 0.95, Applied, there is a moral difference in treatment for these Samples .While By comparing calculated T of samples (Fe, Cr, Co, Ni, Cu, Mo, Pb, Cd and NO2-) with a scheduled value T(0.025),n-1, we find that the calculated value is Lower than the scheduled, i.e. inside the point of acceptance of the hypothesis theoryH0. The theory accepts the hypothesis H0:µuntreated = µtreated and rejects the alternative theory H1: µuntreated ≠ µtreated with a probability of 0.95, so there isn't a moral difference in treatment for these Samples.

In the Table (4) the correlation coefficient results are positive signal that means extreme relationship between the water concentration before and after treatment. But the values of elements (Pb, Co, Mn, and Fe) between (0.01-0.3) means that processor efficiency for these elements is relatively weak. While the (Cd, Cu, Ni, Cr, Phenols, NO3-,and NO2-) correlation factor (0.8-0.9)of elements, the processor efficiency of these elements is very strong.

The calculated T of samples (Fe,Mn,Ni,Cu,Mo,Cd,NO2- , NO3-) with a scheduled value T(0.025),n-1, we find that the calculated value is higher than the scheduled, i.e. outside the point of acceptance of the hypothesis theoryH0, i.e. in the alternative rejection area. The theory rejects the hypothesis H0:µuntreated = µtreated and accepts the alternative theory H1: µuntreated ≠ µtreated with a probability of 0.95, there is a moral difference in treatment for these Samples .While By comparing calculated T of samples (Cr, CoPb, and Phenols) with a scheduled value T (0.025),n-1, we find that the calculated value is Lower than the scheduled, i.e. inside the point of acceptance of the hypothesis theoryH0. The theory accepts the hypothesis H0:µuntreated = µtreated and rejects the alternative theory H1: µuntreated ≠ µtreated with a probability of 0.95, there isn't a moral difference in treatment for these Samples.

The Table (5) used a t-test Two-Sample Assuming Unequal Variances Type because of the different type of refineries, so this type of measurement was chosen for a difference in the contrast values. It was noted that by

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comparing the calculated T values of the Samples (Fe, Mn, Co, Phenols, and NO2-) with T(0.025) we find that the calculated T value is higher than the T(0.025) (outside the acceptance area of the hypothesis theory H0),i.e. located in the rejection area. The theory rejects the hypothesis H0:μuntreated = μtreated and accepts the alternative theory H1: μuntreated ≠ μtreated with a probability of 0.95, there are treatment teams for Al- Najaf refinery because it is the highest average.

The rest of the elements show that the comparing calculated T values of the Samples (Cr, Cu, Ni, Mo, Pb, Cd, and NO3-) with T(0.025), we find that the calculated T value is lower than the T(0.025) ,i.e inside the acceptance area of the hypothesis theory H0. The theory accepts the hypothesis H0:μuntreated = μtreated and rejects the alternative theory H1: μuntreated ≠ μtreated with a probability of 0.95, that is, there is no high efficiency difference between the Refineries for these elements.

Table 2: Mean Results of the Elements for the AL-Dura (A) and Al- Najaf (B) Refineries before and after Treatment

Ion mean	**Fe**	**Mn**	**Cr**	**Co**	**Ni**	**Cu**	**Mo**	**Pb**	**Cd**	**Phenols**	**NO_3^-**	**NO_2^-**
(A) before	1.5	0.13	15.7	0.99	12.4	5.18	2.05	24.2	2.23	2.66	18.8	0.04
(A) after	0.003	0.003	4.31	0	6.87	1.03	1.73	7.35	2.56	0.21	5.38	0.12
(B) before	31.4	124.7	21.39	12.06	10.8	16.3	20.3	57.6	10.9	5.96	5.07	3.53
(B) after	11.04	52.9	6.01	2.81	3.2	2.21	3.98	9.4	3.38	6.52	6.73	4.13

Table 3: Result of T-Test: Paired Two Sample for Means and Pearson Correlation Distribution of AL-Dura Refinery (A)

Sample	**n**	**μ**	**D**	**Pearson Correlation**	**T cal.**	**$T_{(0.025),n-1}$**
Fe	8	1.50	0.003	-0.398	2.186<<2.365	
Mn	8	0.13	0.003	-0.302	3.794>>2.365	
Cr	10	14.2	4.70	0.707	1.293<<2.262	
Co	10	0.79	0		1<<2.262	
Ni	10	12.4	6.87	-0.106	1.318<<2.262	
Cu	10	5.18	1.03	0.036	1.522<<2.262	
Mo	10	1.85	1.73	0.314	0.017<<2.262	
Pb	10	24.2	7.35	0.640	0.870<<2.262	
Cd	10	2.23	2.56	0.190	1.782<<2.262	
Phenol	10	2.66	0.207	0.545	9.287>>2.262	
NO_3^-	10	18.7	5.377	0.546	5.928>>2.262	
NO_2^-	10	0.039	0.121	-0.154	1.257<<2.262	

Table 4: Result of T-Test: Paired Two Sample for Means and Pearson Correlation Distribution of AL-Najaf Refinery

Sample	**n**	**μ**	**D**	**Pearson Correlation**	**T cal.**	**$T_{(0.025),n-1}$**
Fe	6	31.37	11.04	0.396	9.462>>2.57	
Mn	6	124.71	52.98	0.010	3.456>>2.57	
Cr	6	21.39	6.01	0.894	2.423<<2.57	
Co	6	12.063	2.81	0.291	2.478<<2.57	
Ni	6	10.81	3.2	0.882	2.789>>2.57	
Cu	6	16.28	2.21	0.879	2.948>>2.57	
Mo	6	20.28	3.98	0.573	4.951>>2.57	
Pb	6	57.58	9.44	0.376	2.061<<2.57	
Cd	6	10.98	3.39	0.807	4.077>>2.57	
Phenols	6	5.96	6.52	0.891	0.628<<2.57	
NO_3^-	6	5.07	6.73	0.888	4.236>>2.57	
NO_2^-	6	3.53	4.13	0.987	4.570>>2.57	

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Table 5: The Difference between the Efficiency of the Al-Dura Refinery Processor (A) and Najaf Refinery (B) Statistically

Samples	Mean(A)	Mean(B)	$T_{cal.}$	$T_{(0.025),n-1}$
Fe	0.0028	11.035	12.99>>2.570	
Mn	0.003	52.981	5.369>>2.570	
Cr	4.309	6.011	0.963<<2.119	
Co	0	2.813	4.029>>2.570	
Ni	6.865	3.2	1.703<<2.306	
Cu	1.020	2.205	1.626<<2.364	
Mo	1.734	3.981	2,347<<2.446	
Pb	7.345	9.441	0.461<<2.364	
Cd	2.562	3.385	0.582<<2.446	
Phenols	0.207	6.523	3.619>>2.570	
NO_3^-	5.377	6.736	1.373<<2.228	
NO_2^-	0.122	4.133	5.127>>2.570	

Table 6: The Iraqi and Global Environmental (World Health Organization) Determinants of the Measured Elements and their Comparison with the Results of al Dura (A) and al Najaf (B) Refinery

sample	EARE (A)	EARE (B)	MOH. IQ	WHO[30]
Temp.	20.25	30.33	35	5 degree< than mid
Fe(ppm)	0.002	11	5	0.3
Mn(ppm)	0.003	53	80	0.5
Cr(ppm)	0.004	6	0.1	0.05
Co(ppm)	UDL*	3	0.05	0.02
Ni(ppm)	0.01	3.2	0.2	0.02
Cu(ppm)	0.001	2	0.2	0.002
Mo(ppm)	0.002	4	0.01	NA**
Pb(ppm)	0.01	9	0.1	0.01
Cd(ppm)`	0.003	3	0.01	0.003
Phenols(ppm)	0.207	7	0.05	0.02
NO_3^-(ppm)	5.377	8	50	NA
NO_2^-(ppm)	0.121	4	50	NA
***UDL**=under detect limit**,** ****NA**=not available.				

In Table (6) shows the values of concentration of elements discharged to the river in an area (A) and (B) compared with the determinants of the Iraqi Ministry of Environmental Health (MOH.IQ) and the global environmental health determinants, it’s clear that the concentration of the area (A) is lower or within the permissible limits due to the lack of concentration found in the mainly contaminated water. Area (B) despite the efficiency of the processor, a high percentage of elements are thrown in to the river beyond the permitted environmental limits.

VI. CONCLUSION

Drawing samples from the refinery before and after the treatment unit during separate periods, taking into account the standard conditions of temperature and acidity and determination of the proportion of heavy elements using flammability absorption (FAAS) and GFAAS. Comparing the results with Najaf refinery to know the efficiency of the processor in each refinery. It was found that the percentage of the elements in the industrial wastewater of the Dura refinery is low compared with al-Najaf refinery, and the percentage of the elements in the industrial wastewater of the al-Najaf refinery is high compared with Ministry of health (MOH.IQ). Conducting

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evaluative statistical methods for the results reached.

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