

Assessment of Industrial Wastewater Treatment efficiency with Eco-friendly coagulants-A Case Study

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Abstract- Every industry generates wastewater with different pollutants. The volume of wastewater and pollution load depend on the process of industry and the installed capacity. Generally petrochemical industry, textile industry, food industry and paper industry generate huge volume of wastewater with high pollution load. The aim of this project is to assess the treatment efficiency of petrochemical industry wastewater with eco-friendly coagulants. The study was focussed on the pollutants like pH, total suspended solids, chemical oxygen demand, Nitrate, Phosphate, and Oil and Grease by using eco-friendly coagulants Moringa oliveira seeds and Ocimum Sanctum (Tulsi) leaves with organic solvent Methylene Chloride (CH₂Cl₂). The maximum percentage of reduction of physicochemical parameters of wastewater with respect to coagulant dosages of the natural coagulant M. oleifera seeds and Osmium Sanctum (Tulsi) leaves with Methylene Chloride. The maximum percentage of reduction of pH is 1.2 % at 50 mL dosage of natural coagulant and 45 mL dosage of organic solvent, phosphate is 5 %, Nitrate is 96.3%, Oil and Grease is 99.1 % and the maximum percentage of reduction of COD is 93.9 %. The treatment increased with the increase of dosage. The results are encouraging.

Keywords: Coagulation; COD; Moringa; Nitrate; Oil and Grease; Osmium Sanctum (Tulsi); Phosphate;

I. INTRODUCTION

As per the new regulations, the industry should adopt recycle and reuse technologies to conserve water resources. The zero liquid discharge (ZLD) is the new concept. The industry is investing good amount of money to develop technically feasible and economically viable technologies. The aluminium sulphate (Alum) and ferric chloride were used for chemical coagulation for decades. Later poly aluminium chloride was used as an effective coagulant than alum and ferric chloride. At present most of the industries are using anionic polymers and cationic polymers for coagulation. These polymers 1 millilitre is equal to 1 kilogram alum. The aim of this study is to replace the chemical coagulants with natural eco-friendly coagulants.

A study was conducted with eco-friendly coagulants of Moringa Oleifera, Tamarina indica and Strychnomous potatorum to treat textile industry wastewater. A dosage of 10 ml, 20 ml, 40 ml, 60 ml and 80 ml was added and studied the pollutants removal efficiency. The Moringa Oleifera seeds have shown better results among other coagulants (Muralimohan, 2014).

The small scale industries in India are disposing their influents in urban sewage manholes and urban gutters (Nikam et al. 2012). It has a negative impact on activated sludge process. The toxic chemicals from the industries inhibit biological treatment. It has been observed all over the world that industrial waste water discharges non-biodegradable toxic chemicals and heavy metals into the surface water bodies (Varna 2018). The researchers identified good number of natural coagulants and studied the treatment efficiency. Moringa Oleifera seeds are utilized as an absorbent and coagulation for wastewater treatment because it is distinguished by containing a high level of antimicrobial activity, strong coagulating properties for sedimentation of suspended undesired particles, inexpensive and effective (Emmanuel, 2016). The Cicer arietinum seed at a dosage of 1.5 mg/l, the COD and oil removal efficiency was 95.2%, and 83.8% respectively (Khader EH et.al.2018). The Moringa Oleifera seed powder was used for river water clarification. Experiments were conducted with 50 mg/l, 100 mg/l, 150 mg/l, and 200 mg/l dosages. It was observed that the turbidity, BOD, alkalinity, hardness, TDS, chloride and sulphate were reduced considerably (Karri.V.et.al. 2015). A comparative study was conducted as Moringa oleifera and alum as coagulants. The removal efficiency of COD, turbidity and TSS by using Moringa oleifera was 38.60%, 63.70% and 62.05% respectively. The alum dosage at the rate of 40 mg/L removed COD, turbidity, and TSS by 51.72%, 92.16%, and 85.26% respectively (Dhruva, R. Suresh, B., 2016). Good numbers of studies have shown that Moringa Oleifera is highly effective as a eco-friendly coagulant for turbidity removal (Golestanbagh, 2011). The Moringa Oleifera seed is effective as an eco-friendly coagulant to remove heavy metals and bacteria. It does not have any negative impacts on environment (Shan et.al.2016).The Moringa Oleifera seed extract has a tendency of adsorption and charge neutralization. The optimum dosage was 10 mg/l. That is why its turbidity removal efficiency is high (Nikam et al 2012).

II. METHODOLOGY

The good quality of Moringa Oliveira seeds and Osmium Sanctum (Tulsi) leaves were collected from neighboring farms from local trees and it was dried by exposing it to sunlight for approximately two weeks. The Osmium sanctum was dried for 2 days to protect the plant phytochemicals or antimicrobial constituents inside the seeds and leaves the direct daylight was avoided. The seeds were placed in an automatic hot air oven at 35°C for 5 hours to make sure that they were dried and free from all moisture content. (Dehghani & Alizadeh, 2016). In order, to increase the solubility of the seeds, they were powdered to decrease the particle size to approximately 600 µm. From the powder, the seed extract was prepared and stored in a sterile air-tight container in a dark place to prevent oxidation and for further use. The images of Moringa Oleifera seeds and Osmium Sanctum shown in fig 1.



Fig 1: Eco-friendly coagulants Moringa Oleifera seeds and Osmium Sanctum (Tulsi) leaves

III. PHYSIOCHEMICAL ANALYSIS OF PETRO-CHEMICAL INDUSTRY INFLUENT:

The Petrochemical influent samples were analyzed for pH, COD, TSS, Phosphate, Nitrate and Oil and Grease content to estimate the initial concentration. All measurement methods of the parameters were based on the standards for water and wastewater experiments were performed at the laboratory of Oman Refineries and Petrochemicals Industry Company.

3.1. Preparation of Coagulants Stock Solution:

Before starting the treatment process, the stock solutions were prepared. 1 g each of Moringa oliveira seeds and Ocimum Sanctum (Tulsi) leaves, were added to 100 mL of distilled water and mixed rapidly for 20 min using a magnetic stirrer to enhance water extraction of the coagulant proteins. To obtain a homogeneous solution free of suspended materials, the resulting suspension was passed through a filter paper of 0.45-micron filter paper. The coagulation procedure was adopted for a mixture of these two powders. The coagulation process was done with 15 mL, 20 mL, 25 mL, 30 mL, 35 mL, 40 mL, 45mL and 50 mL respectively for the remaining 8 samples of wastewater. (Murali Mohan & Palanisamy, 2014)

To obtain reliable and reproducible results FP4 Portable Flocculator apparatus was used in the experiment that allows standard conditions to be adopted for the jar test. The jar test was conducted for all the samples with 500-mL and 1000-mL beakers. The sample was mixed homogenously then the beakers were filled with about 500 mL of wastewater sample. It was carried out as a batch test to coagulate samples of PETRO CHEMICAL wastewater. At a time 4 samples processed in parallel with four stirring rods adjustable in height without tools and central light to facilitate reading. Different concentrations of prepared stock solution with organic solvent were applied into each beaker. The coagulant dosages were 10 mL, 15 mL, 20 mL, 25 mL, 30 mL, 35 mL, 40 mL, 45mL and 50 mL and for organic solvent Methylene Chloride (CH₂Cl₂) dosages were 5 mL, 10 mL, 15 mL, 20 mL, 25 mL, 30 mL, 35 mL, 40 mL and 45 mL were added into all samples together. The samples were coagulated for 30 minutes with 200 rpm. After coagulation the samples were filtered and analyzed for COD, Nitrate, Phosphate, TSS, Oil and Grease and finally check of pH. (Varma, 2018)

3.2. Measurement of Oil and Grease Content:

This test method covers the determination of oil and grease and nonpolar material in wastewater by an infrared (IR) determination of dimer/trimer of chlorotrifluoroethylene (S-316) extractable substances from an acidified sample. Included in this estimation of oil and grease are any other compounds soluble in the solvent. This method covers the range of 5 to 100 mg/L and may be extended to a lower or higher level by extraction of a larger or smaller sample volume collected separately.

3.3. Calculation the Percentage of Removal Efficiency:

In this study, was examined the efficiency of coagulant products such as Moringa Oleifera, Ocimum Sanctum (Tulsi) and Methylene Chloride (CH₂Cl₂) in the removal of, PH COD, TSS, Nitrate, Phosphate and Oil and Grease. The removal efficiency (% Removal) was calculated from the following formula:

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$$\% \text{Removal} = (\text{Cin} - \text{Cout}) / (\text{Cin}) \times 100$$

Where, Cin and Cout = COD, TSS, Nitrate, Phosphate, pH and Oil and Grease contents of wastewater (mg/L) before and after coagulation treatment, respectively .

3.4 Calculation of the Amount of Coagulants Required for One Cubic Meter of PETRO CHEMICAL Wastewater and the Cost of Treatment:

For the treatment of one cubic meter of PETRO CHEMICAL industry wastewater that equal to 1000 L, the amount of coagulants required are 1 kg of Moringa Oleifera seeds powder is equal to 2.0 OMR and 1kg of Tulsi leaves is equal to 1.0 OMR baissa, total cost= 3.000 OMR. Instead of 1 g Moringa Oleifera seeds powder and 1 g of Tulsi leaves powder that used to coagulate of 500 mL of PETRO CHEMICAL wastewater, the cost of treatment = 2.5 baissa and for 1000 ml= 5 baissa. So, by this the cost of the treatment of 1 m³ equal to 5 OMR.

IV. RESULTS AND DISCUSSIONS

4.1 Preliminary Characteristics of Petroleum Refinery Influent:

The initial concentration of PETRO CHEMICAL industry effluent was shown in Table .1.

Table 1: Average analysis of petro chemical industry influent sample.

Parameter	petroleum refinery influent
COD (mg/L)	425
TSS (mg/L)	125
pH	5.6
Nitrate (NO ₃) (mg/L)	270
Phosphate (mg/L)	0.22
Oil and Grease (mg/L)	22.7

4.2 Results of the Jar Test:

The results of the jar test at various Moringa Oleifera and Ocimum Sanctum (Tulsi) concentrations are (10 mL, 15 mL, 20 mL, 25 mL, 30 mL, 35 mL, 40 mL, 45mL and 50 mL) and organic solvent Methylene Chloride (CH₂Cl₂) dosages were 5 mL, 10 mL, 15 mL, 20 mL, 25 mL, 30 mL, 35 mL, 40 mL and 45 mL) that was carried out to measurement of pH, COD, TSS, Phosphate, Nitrate and Oil and Grease according to the standards methods for water and wastewater were performed for 9 samples of water at the laboratory of petro chemical company are presented in

Table 2,3,4,5,6,7,8,9&10.

Table .2: The concentration of effluent with 10 mL coagulant and 5 mL of solvent

Sample No.	Parameter	petroleum refinery effluent
1	COD (mg/L)	272
	TSS (mg/L)	96
	PH	6.2
	Nitrate (NO ₃) (mg/L)	210.1
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	14

Table .3: The concentration of effluent with 15 mL coagulant and 10 mL of solvent

Sample No.	Parameter	petroleum refinery effluent
2	COD (mg/L)	229
	TSS (mg/L)	86
	PH	6.3
	Nitrate (NO ₃) (mg/L)	140
	Phosphate (mg/L)	0.21

	Oil and Grease (mg/L)	12
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Table .4: The concentration of effluent with 20 mL coagulant and 15 mL of solvent

Sample No.	Parameter	petroleum refinery effluent
3	COD (mg/L)	187
	TSS (mg/L)	72
	PH	6.5
	Nitrate (NO ₃) (mg/L)	56.7
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	6

Table 5: The concentration of effluent with 25 mL coagulant and 20 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
4	COD (mg/L)	158
	TSS (mg/L)	65
	PH	6.8
	Nitrate (NO ₃) (mg/L)	32
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	1

Table .6: The concentration of effluent with 30 mL coagulant and 25 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
5	COD (mg/L)	132
	TSS (mg/L)	51
	PH	8
	Nitrate (NO ₃) (mg/L)	24
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	0.9

Table 7: The concentration of effluent with 35 mL coagulant and 30 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
6	COD (mg/L)	102
	TSS (mg/L)	43
	PH	8
	Nitrate (NO ₃) (mg/L)	20.2
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	0.8

Table .8: The concentration of effluent with 40 mL coagulant and 35 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
7	COD (mg/L)	80
	TSS (mg/L)	36
	PH	8.1
	Nitrate (NO ₃) (mg/L)	17.3
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	0.7

Table 9: The concentration of effluent with 45 mL coagulant and 40 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
8	COD (mg/L)	51
	TSS (mg/L)	18
	PH	8.2
	Nitrate (NO ₃) (mg/L)	13
	Phosphate (mg/L)	0.21

	Oil and Grease (mg/L)	0.6
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Table 10: The concentration of effluent with 50 mL coagulant and 45 mL of solvent.

Sample No.	Parameter	petroleum refinery effluent
9	COD (mg/L)	26
	TSS (mg/L)	11
	PH	8.2
	Nitrate (NO ₃) (mg/L)	6.7
	Phosphate (mg/L)	0.21
	Oil and Grease (mg/L)	0.4

V. RESULTS AND DISCUSSION

In this study, the efficiency of coagulant products such as Moringa Oleifera, Ocimum Sanctum (Tulsi) together with Methylene Chloride (CH₂Cl₂) in the removal of pH, COD, TSS, Nitrate, Phosphate and Oil and Grease was examined at various coagulant dosages. The results in brief are presented in Table 11 below.

Table 11: The Removal Efficiency of pH, COD, TSS, Nitrate, Phosphate and Oil and Grease

Sample No.	coagulant dosage mL	Parameters	% COD	% pH	% TSS	% Nitrate	% Phosphate	% Oil and Grease
1	15	Removal Efficiency	36	25.3	23.2	22.2	5	38.3
2	25		46.1	24.1	31.2	48.1	5	47.1
3	35		56	21.7	42.4	79	5	73.6
4	45		62.8	18.1	48	88.1	5	95.6
5	55		68.9	3.6	59.2	91.1	5	96
6	65		76	3.6	65.6	92.5	5	96.5
7	75		81.2	2.4	71.2	93.6	5	97.4
8	85		88	1.2	84.8	95.2	5	98.2
9	95		93.9	1.2	91.2	96.3	5	99.1

Table 12: Ministerial Decision MD159/2005 “Discharge liquid effluent in marine environment” which Waste water treatment plant should comply with it before discharged in to coastal waters

Parameters	unit	Standard not greater than
COD	mg/L	150
TSS	mg/L	15
NO ₃	mg/L	50
phosphate	mg/L	0.5
pH	-	6-9
Oil and Grease	mg/L	0.5

5.1 Effect of Natural Coagulants Dosages on Treatment Process:

The objective of this study was to assess the treatment efficiency, the two coagulants, i.e., Moringa Oleifera and Ocimum Sanctum (Tulsi) with Methylene Chloride (CH₂Cl₂), that can be used to coagulate the suspension particles in the petroleum wastewater. It was found that the behavior of coagulant may change from wastewater to another according to many factors including pH, kind of coagulant and different constituents of wastewater (Roshan & George, 2016).

5.2 Effect of pH with Natural coagulants Doses:

A plot between pH removal (%) and dosage ratios of natural coagulant was shown in Figure 4.1. A gradual decrease in pH removal (%) was attained by dosage of 15 mL, 25 mL, 35 mL, 45 mL, 55 mL, 65 mL, 75 mL, 85 mL and 95 mL in untreated water and treated water and the change is from 8.3 changes to 6.2, 6.3, 6.5, 6.8, 8, 8, 8.1, 8.2 and 8.2 in untreated water and treated water respectively. The study revealed that decrease the pH in raw water by Moringa has shown coagulants and is more effective. The present studies, treatment of Moringa and Tulsi with organic solvent were given to petroleum wastewater samples in different doses. During the analysis, it was observed that

after treatment with natural coagulants, pH was increased at all doses until reached the basic range. After treatment, the range of pH was 6.2-8.2 and within the limit. The recommended acceptable range of pH for discharging water specified by the company is between 6.0 and 9.0 is shown in table 12. The effect of pH shown in fig 2.

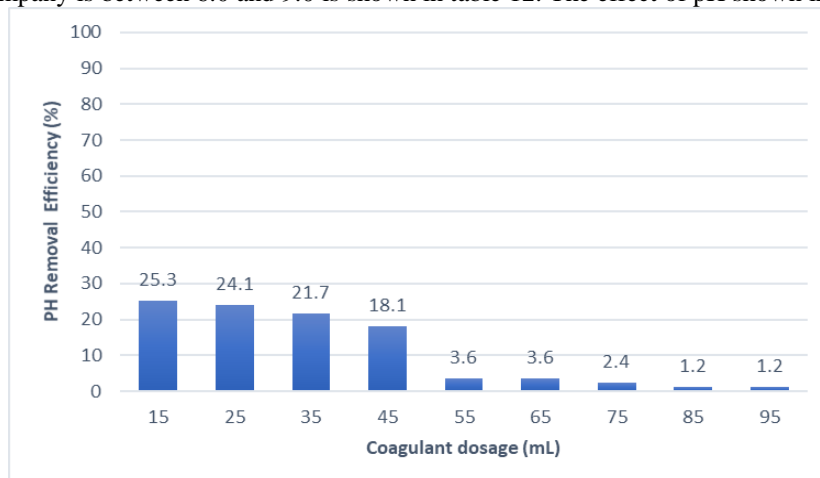


Figure 2: Effect of Natural Coagulants Dosages on the removal of pH

5.3 Effect of COD with Natural coagulants Doses:

In the initial stage, the COD of untreated water was 425mg/L. Natural coagulants dosages of 15 mL, 25 mL, 35 mL, 45 mL, 55 mL, 65 mL, 75 mL, 85 mL and 95 mL were applied the COD of nine samples of untreated water is reduced from 425 mg/L to 272 mg/L, 229 mg/L, 187 mg/L, 158 mg/L, 132 mg/L, 102mg/L, 80mg/L, 51mg/L and 26 mg/L respectively. From the above results of final concentration of COD, the samples No 5, 6, 7, 8, 9 are of 132 mg/L, 102mg/L, 80mg/L, 51mg/L and 26 mg/L respectively are less than standard range for COD before discharged to Marine, which is about not greater than 150 mg/L. The other remaining samples are not complying with the standard range, resulting cannot discharge these sample to the sea. The reductive values of untreated water value from initial value are shown in (Table 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9 & 4.10). The concentrations of COD in all the sampling point were gradually decreased. The removal efficiency of COD by adding natural coagulants is shown in Figure .3.

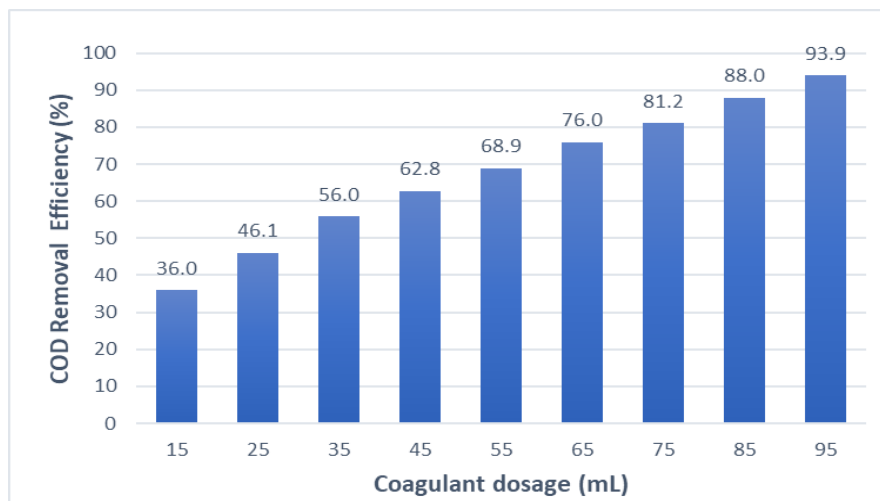


Figure 3: The removal efficiency of COD with respect to coagulant dosage.

5.4 Effect of TSS with Natural coagulants Doses:

The other parameters significant study is total suspended solid (TSS). Total suspended solids (TSS) include all particles suspended in water which will not pass through a filter. Literature classified wastewater TSS as follows: TSS less than 100 mg/L as weak, TSS greater than 100 mg/L but less than 220 mg/L as medium and TSS greater than 220 mg/L as strong wastewater. Results of the study show that TSS of wastewater from the petroleum

wastewater can be classified as medium and should be treated before discharged into stream (125 mg/L). The total suspended solids (TSS) concentrations of petroleum removed by natural coagulants are shown in Figure 4.. As shown, by using *Moringa Oleifera*, *Ocimum Sanctum* (Tulsi) together with Methylene Chloride (CH_2Cl_2) as coagulant would be improved the removal of TSS of all nine samples, were the removal efficiency as 23.2 %, 31.2 %, 42.4 %, 48 %, 59.2 %, 65.6 %, 71.2 %, 84.8 % and 91.2 % respectively. Also, from the final concentration of TSS after treatment which are about 96 mg/L, 86 mg/L, 72 mg/L, 65 mg/L, 51 mg/L, 43 mg/L, 36 mg/L, 18 mg/L & 11 mg/L respectively of nine samples, depending on this results, only sample 9 about 11 mg/L is within the acceptable range of TSS which is less than 15 mg/L.

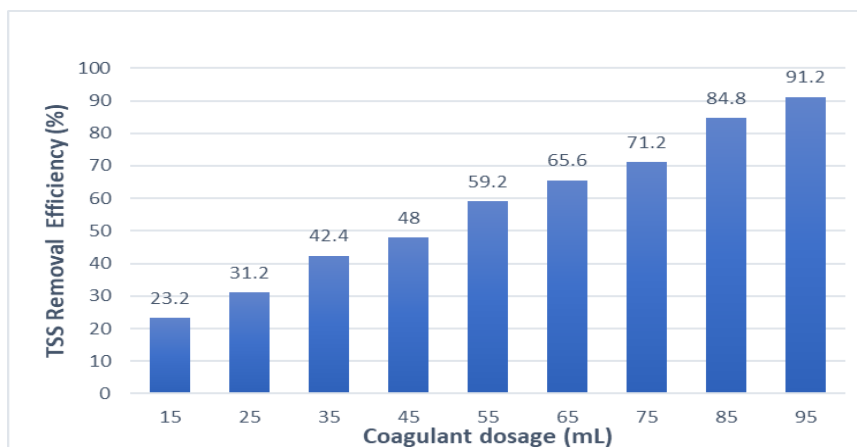


Figure 4.: The removal efficiency of TSS

5.5 Effect of Oil and Grease with Natural coagulants Doses:

The removal efficiency for Oil and grease is increase by increasing the coagulants concentration from 15 to 95 mg/L of both natural coagulants dosages with organic solvent shown in fig 5.. However, an increase in the removal efficiency of pollutants was noted in term of oil and grease when the concentration of added coagulant reaching the maximum dosage was 95 mg/L. This increase in the removal efficiency of pollutants caused by the flotation drops of solvent Methylene Chloride to the surface of produced water was about 45 mL, resulting in lesser oil and grease concentrations. The results of the oil and grease parameter after treatment were shown in tables Table 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9 & 4.10. The recorded results shown that, sample 9 about 0.4 mg/L is only complying with standard range of Oil and Grease that the discharge liquid effluent should be not more than 0.5 mg/L.

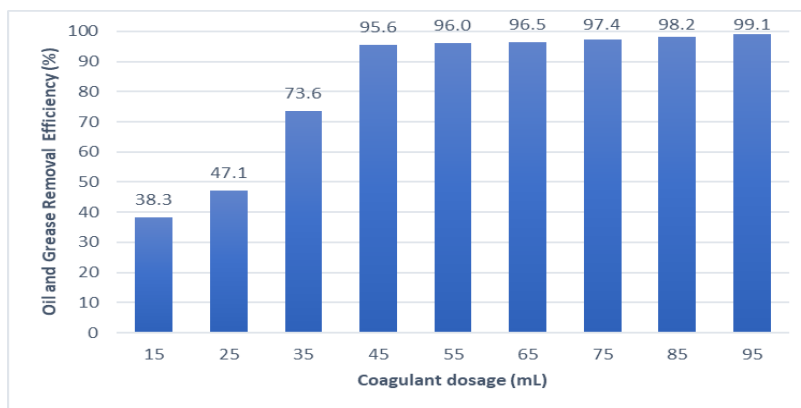


Figure 5: The removal efficiency of Oil and Grease

5.6 Effect of Nitrate and Phosphate with Natural coagulants Doses:

The nitrate/nitrite and the phosphate levels in the water samples collected from the API unit in the company relatively high and greater than the permitted levels in the company for the discharge and thus pose threat to the petroleum water tested. It must be mentioned here that the nitrate/nitrite of the water samples tested showed a significant decrease in the levels of these nutrients compared to the levels in the original water samples. Regarding

to the phosphate levels, there are constant change about 0.21 mg/L from the initial concentration which is about 0.22 mg/L, resulting less removal efficiency. The sample 4-9 in term of Nitrate are within the standard range, all the recorded results of these sample are less than 50 mg/L. Also, all the nine samples in term of phosphate are within the acceptable standard range, all the sample which 0.22 mg/L less than 0.5 mg/L. The removal efficiency of the nitrate and phosphate were gradually increased were shown in figures 6& 7.

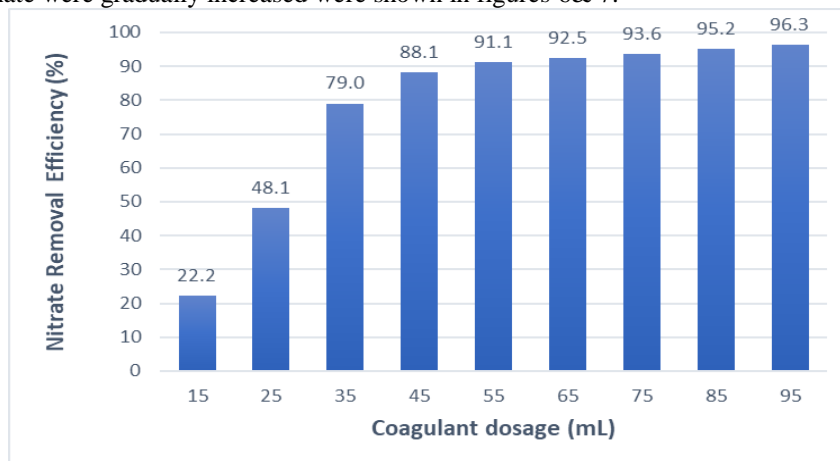


Figure 6: The removal efficiency of nitrate.

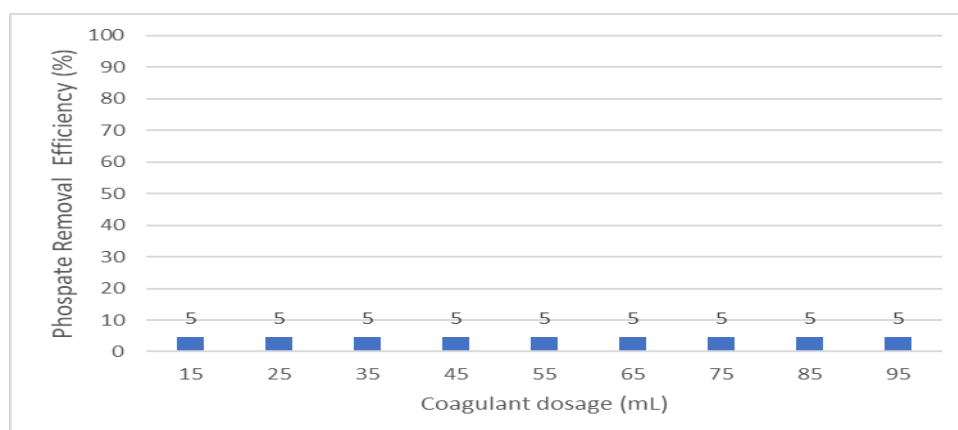


Figure 7: The removal efficiency of phosphate

VI. RESULTS AND DISCUSSIONS:

The present study reveals that the Moringa seed powder is an efficient and economically viable coagulant to purify the produced water at the petroleum refining industry. By adding Moringa seed coagulant, the pH of the water becomes basic with increasing coagulant concentration. Studies confirmed that the action of *Moringa oleifera* as a coagulant lies in the presence of water soluble cationic proteins in the seeds. This suggests that in water, the basic amino acids present in the protein of *Moringa* would accept a proton from water resulting in the release of a hydroxyl group making the solution basic. Similarly, the TSS, nitrate, phosphate, oil and grease were considerably decreasing with increasing concentration. Chemically the seeds consist of low molecular weight proteins that are water soluble. In the water, these proteins have an overall positive charge. Pollutants of turbid waters have an overall negative charge, such as sand, silt, clay and bacteria. *Moringa oleifera* seed powder solution has the capability of reducing low and high turbidity values in water. The finding from this study showed that the active agents in the *Moringa oleifera* seeds and *Ocimum Sanctum* (Tulsi) with Methylene Chloride (CH_2Cl_2) solution are water soluble. However, coagulants solution at optimum dosage was 95 mL of 50 mL of natural coagulants and 45 mL of solvent showed 91.2% TSS removal, 96.3 % Nitrate removal, 99.1% oil and Grease removal. The removal efficiency of COD by adding natural coagulants is 93.9%. According to the present study, orthophosphate levels in the water, during treatment increase with the natural coagulant dosage and also significant decrease were noted in the total nitrates and nitrites after treatment. This due to the usage of the purified proteins extracted from the *Moringa* seeds with *Ocimum Sanctum* (Tulsi) leaves. These proteins have indeed been shown to be effective in the removal of

phosphates and nitrates in the water samples tested. Instead of the treatment of water samples by the direct addition of seeds and leaves powders would inevitably add the seeds natural phosphate and nitrate/nitrite to the water samples as leachates. (Muralimohan & Palanisamy, 2014) The result indicates that all the six parameters are given better result with the treatment with petroleum industrial wastewater. The economic and environmental aspects of use of the natural coagulants extracts must be considered. Therefore, replacing alum and other chemicals coagulants the material used in the wastewater treatment unit at the refinery in Petro chemical company with a natural biopolymer such as *M. oleifera*, *Ocimum Sanctum* (Tulsi) and organic solvent or utilizing this four together, can be useful and effective. Moreover, the use of natural coagulants can substantially save money on the cost of chemical materials and reduce to a minimum the sludge produced and, hence, require less disposal. Some natural compounds have coagulant properties and learning about these properties and understanding their exact mechanisms of action can lead to their correct application.

VII. CONCLUSIONS

It was observed that *Moringa Oleifera* seed powder is an effective coagulant to remove nitrate, COD, and Oil & Grease. The maximum percentage of reduction at 50 mL dosage of natural coagulant and 45 mL dosage of organic solvent was nitrate is 96.3%, Oil and Grease is 99.1 % and of COD was 93.9 %. However the phosphate removal efficiency was 5 %,

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