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Comparative Study of Removal of Cod and Ammoniacal-Nitrogen from Industrial Wastewater using Reverse Osmosis and Adsorption

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Abstract This work is aimed at the mitigation of water resource pollution by recommending the best, efficient and relatively cheap method of treating industrial wastewater containing (2) toxic pollutants. Wastewater from Dangote Fertilizer Limited (DFL) was treated using reverse osmosis and adsorption methods of treatment putting into consideration the factors that impedes the efficiency of both methods which are (pH, temperature and contact time). Plantain husk, which is an agricultural waste, was used as adsorbent in the adsorption process while reverse osmosis study was successfully carried out using polyvinyl pyrrolidone grafted on polyvinylidiene fluoride type of membrane with pore size 0.02mm. Response Surface Methodology (RSM) was used to determine the experimental number of runs after which it gives the optimum conditions of those parameters considered. This study revealed that adsorption process is the best for the removal of COD and Ammoniacal nitrogen from industrial wastewater as it gave the highest removal rate and it is cheaper than using reverse osmosis method. It gave a higher percentage removal of 98% for Ammoniacal nitrogen and 90% for COD.

Keywords Ammoniacal nitrogen, Chemical oxygen demand (COD), adsorption, reverse osmosis

1. Introduction

Wastewater released into the environment by petroleum and petrochemical industries continued to raise serious concerns and causing a number of environmental hazards due to the several pollutants therein, including the biggest polluters of water sources in the environment: ammonia, hydrogen sulfide, mercury, arsenic, etc. It is therefore very important to find a way of reducing water use and treating wastewater to make it reusable and less hazardous [1]. The characteristics wastewaters are frequently defined in terms of Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD) and BOD_5, Total Organic Carbon (TOC), and Total Suspended Solids (TSS). However, phosphorus and nitrogen, as well as their compounds, are found waste water particularly from nitrogenous fertilizer industries; these contains ammoniacal nitrogen in extremely high concentrations which can cause serious issues like eutrophication, the loss of dissolved oxygen, and toxicity in aquatic life in lakes, rivers, and other bodies of water [2]. Reducing ammoniacal nitrogen (NH_3-N), a measurement for the amount of ammonia in wastewaters, is a distinctive difficulty for many chemical businesses, particularly the fertilizer industry [3]. Ammoniacal nitrogen is a very hazardous contaminant that, at high quantities, can kill fish and accumulate toxins in aquatic animals' internal organs and blood. It immediately burns the nose, throat, and respiratory tract in people.

Saad and Omar [4] conducted a comparative study on the use of reverse osmosis and adsorption process for heavy metals (Co, As, Cd, Cr) removal from sewage wastewater. The percentage removal of COD and ammoniacal nitrogen from six distinct industrial wastewaters using adsorption on several commercial adsorbents and the percent removal of COD and ammoniacal nitrogen by hydrodynamic cavitation employing vortex diode were compared by [3]. The outcome of this study showed that the hydrodynamic cavitation approach is the best since it achieved the highest COD and ammoniacal nitrogen removal rates. The above research is limited by the fact that the author neglected to take into consideration the number of variables that might have had an impact on the adsorption process. Second, only the hydrodynamic cavitation approach was compared; however, as was already said, there are better physico-chemical techniques, such as reverse osmosis, which is particularly effective in removing COD and ammoniacal nitrogen from industrial wastewaters. In this study, the use of reverse osmosis and adsorption process for removal COD ammoniacal nitrogen from wastewater from fertilizer plant were investigated and compared. In the reverse osmosis process, water travels through a semi-permeable membrane that filters out organic matter including ammoniacal nitrogen, COD, and BOD as well as inorganic minerals like calcium, magnesium, potassium, fluoride, salt, and phosphorus. Additionally, some organic chemicals, such as some pesticides are also removed [5]. Work by [6] shown that the polyvinyl pyrrolidone (PVP)-grafted polyvinylidene fluoride (PVDF) RO membrane treatment of generated water was superior to that utilizing polyvinyl pyrrolidone utilized by [7] in his studies on water purification using reverse osmosis. RO technique works in a wide pH range of 3–11 and pressure range of 4.5–15. [8]. Before RO, the microfiltration process is typically performed to increase the membrane's lifespan, zero fouling, and lower operating expenses. Four distinct forms of activated carbon can be distinguished based on the production process: powder-activated carbon, granular-activated carbon, and activated carbon fiber. Each has a particular [9]. The effectiveness of charcoals made from bamboo, plantain peel, cocoa pod, and maize cob in removing heavy metals from wastewaters was investigated by [10]. It was determined that plantain peel charcoal had the best performance. This study investigated and compare the removal of COD and ammoniacal nitrogen from wastewater generated by fertilizer plant using adsorption with plantain adsorbent and reverse osmosis.

2. Materials and Methods

2.1. Materials

The wastewater used for the study was obtained from Dangote Fertilizer Limited, Lekki Free trade zone while the plantain husk (waste from plantain) was collected from Eleko market, Lagos State of Nigeria. Some of the major pieces of equipment used in the study are Spectrophotometer, DR3900 (Hach); Muffle furnace, Gullen-Kamp muffle furnace size 2, GH2000; Mesh Sieve (Tyler series); weighing balance, Adventure OHAUS Corporation; Orbital shaker (Stuart-SSLIS Rotating at 250rpm; Oven (Gullen-KampDHG-9023A); pH Meter (HQ411dPh/mV); Conductivity meter (Cond 3310); Turbidity meter (TL2300(Hach)); Calibrated pipette (Pyrex BS604 made in England).

2.2 Methods

2.2.1 Characterization of the Wastewater

The wastewater was characterized using the methods described by [11] to obtain the description of the distinctive nature or content of the wastewater, which includes pH, COD content, TDS, TSS, TS, and Turbidity etc. The parameters used to characterize the wastewater are pH, Turbidity of the wastewater, total dissolved solids (TDS) in the wastewater total suspended solids (TSS) in the wastewater, Total organic carbon (TOC) in the wastewater, Chemical oxygen demand (COD) of the wastewater, Biological oxygen demand (BOD) of the wastewater, Total phosphorus, Phosphates, Nitrates, Ammoniacal nitrogen (AN). Standard methods were used for the determination of these parameters for the wastewater samples collected.

For the COD determination, Open Reflux Method was used as described by [11]. A blank containing the reagents and a volume of distilled water equal to that of sample measured was refluxed and titrated.

COD in sample was calculated using equation (1).

$$COD\ (mg\ O_2/l) = (P–Q) \times M \times 8000\ ml\ sample \quad (1)$$

where

P = volume of FAS used for blank (ml)

Q = volume of FAS used for sample (ml)

M = molarity of FAS

The whole procedure was also repeated for water samples collected after adsorption process.

Similarly, the method described by [11] was used to determine the total solid in the wastewater samples. 10% of all samples were analyzed in duplicate and these duplicate determinations were checked if it corresponded with 5% of their average weight. Total solid was calculated using equation (2)

Total solids (mg) = (P - Q) x 1000 Sample volume, ml (2)

Where:

P = weight of dried residue + dish (mg)

Q = weight of dish (mg)

2.2.2 Determination of Total Dissolved Solids

The total dissolved solids in the samples were determined using the method described by [11]. 10% of all samples were analyzed in duplicate and these duplicate determinations were checked if it corresponded with 5% of their average weight. Total solid was calculated using equation (3).

Total dissolved solids mg/L = (P - Q) x 1000Samplevolume, ml (3)

Where:

P = weight of dried residue + dish, mg Q = weight of dish

2.2.3. Determination of Total Suspended Solids

The total suspended solids in the samples were determined using the method described by [11]. 10% of all samples were analyzed in duplicate and these duplicate determinations were checked if it corresponded with 5% of their average weight.

Calculation:

mg total solids/l= (A - B) x 1000 Sample volume (ml) (4)

Where:

A = filter weight + dried residue (mg) B= weight of filter

2.2.4 Measurement of Turbidity and Conductivity

Nephelometric method described [11] was used to determine the turbidity of the samples while the conductivity was determined using the described method.

2.2.5 Determination of Ammoniacal Nitrogen Content

Nesslerization method described by [11] was used to determine the ammoniacal nitrogen content of the wastewater samples. Total ammoniacal nitrogen content was determined by calculating using the equation (5).

Ammoniacal Nitrogen, mg/l (51ml of final volume) =$A \times \frac{B}{V} \times C$ (5)

Where:

A= pg of ammoniacal nitrogen (51ml of final volume);

B = Total volume of distillate collected, in ml, including acid absorbent; C = volume distillate taken for nesslerization, in ml;

V= volume of sample taken.

The whole procedure was also repeated for water samples collected after adsorption and reverse osmosis processes.

2.2.6 Experimental Design for the Adsorption and Reverse Osmosis

RSM is a group of mathematical and statistical methods used to optimize the operating conditions for the greatest lead metal ion elimination. The optimal adsorption and reverse osmosis process variables for the removal of COD and ammoniacal nitrogen will next be determined using a three-level, three-factor Box-Behnken factorial design (BBD) (Design Expert Software, Trial Version7.1.6, Stat-ease Inc., Minneapolis, MN, USA).Optimizing a response and establishing a connection between a response (output variable) and the interactive effects of independent factors (input variables) are the basic goals of experimental design. Understanding and evaluating the impact of various parameters and how they combine to produce the answer is the main benefit of employing

RSM (s). Consequently, it is regarded as a suitable strategy to optimize a process with one or more result. The variable input parameters are pH values in the range of 5.5-7.5, contact time of 30-100 seconds and temperature of 25°C to 35°C. The three independent variables were designated as A- temperature, B- contact time and C- pH for the statistical analysis. The low, centre and high levels of each variable are designated as -1, 0, and +1, respectively as illustrated in Table 1.

Table 1: Independent variables and their levels used for Box-Behnken Design.

Factors	Variables	Low Level	Center Level	High Level
Temperature (°C)	A	25	30	35
Contact time, (seconds)	B	30	65	100
pH	C	5.5	6.5	7.5

The experimental levels for each variable were selected based on results from preliminary experiments and coding of the variables was done according to the following equation:

$$xi = \frac{Xi - Xo}{\Delta Xi} \quad (6)$$

Where:

xi is the dimensionless value of an independent variable Xi is the real value of an independent variable

Xo is the real value of an independent variable at the centre point

ΔXi is the step change of the real value of the variable, i, corresponding to the variation of a unit dimensionless value of variable, i.

The number of experiments (N) needed for the development of Box-Behnken matrix is defined as;

$$N = 2K(K-1) + r \quad (7)$$

Where:

K is the factor number is the replicate number of the central point

A total of 17 trials were run in order to optimize the parameter at which the maximum removal was obtained. Executing a statistically designed experiment, estimating the coefficient, analyzing the response, and ensuring the model is appropriate are the three fundamental processes in the optimization process. Selecting the optimal model for expressing the relationship between the response and other influencing independent variables involves conducting investigations using a variety of tests, such as sequential model sum of squares, lack of fit tests, and model summary statistics. To examine the outcome, regression analysis and analysis of variance responses were both used. The most popular second order polynomial equation can be expressed as equation shown below to fit the obtained experimental data and to identify the pertinent model terms;

$$Y = \beta_0 + \sum\beta iXi + \sum\beta iiXii^2 + \sum\beta ijXiXj + \epsilon \quad (8)$$

Where:

Y is the predicted response (the percentage removal of COD and ammoniacal nitrogen),

β_0 is the constant coefficient,

β_i is the linear coefficient of the input factor X_i

β_{ii} is the i^{th} quadratic coefficient of the input factor X_i

β_{ij} is the different interaction coefficient between the input factor X_i and X_j.

ϵ is the error of the model [12], assumed to have a zero mean.

For this study, the independent variables were coded as A, B and C. Thus, the equation can be represented as equation (9).

$$Y = \beta 0 + \beta_i A + \beta_i C + \beta_i C + \beta_{ii} A^2 + \beta_{ii} B^2 + \beta_{ii} C^2 + \beta_{ij} AB + \beta_{ij} AC + \beta_{ij} BC \quad (9)$$

Desirability Function

The desirability function approach is a technique for the simultaneous determination of optimum settings of input variables that can determine optimum performance levels for one or more responses. The desirability procedure involves two steps:

- Finding the levels of the independent variables that simultaneously produce the most

- Maximize the overall desirability with respect to the controllable factors.

The desirability function approach was originally introduced by Harrington, 1965. Then another version was developed by Derringer and Suich (1980). The general approach is to first convert each response (Yi) into an individual desirability function (di) varying over the range

$$0 \leq d_i \leq 1$$

Where if response Yi is at its goal or target, then di=1, if the response is outside an acceptable region, di=0. Then the design variables are chosen to maximize the overall desirability.

$$D = (d_1 \times d_2 \times \ldots \times d_n)^{1/n} \quad (10)$$

Where n is the number of responses in the mixture.

2.2.7 Adsorption Experiment

The main type of adsorbent material used in this study was plantain husk which was gotten from one of the markets in Ibeju-Lekki City called Eleko market. The plantain husk was used to produce the activated carbon using the procedure described by [11] and was further characterized to determine its using standard methods to obtain the moisture content, bulk density and surface area The adsorption experiment was performed using the plantain husk as described by [11]. The pH of the solution will be adjusted using hydrochloric acid and sodium hydroxide. The concentrations of COD and ammoniacal nitrogen in the filtrate was analyzed using the spectrophotometer. Batch adsorption experiments were carried out at the above temperature range by shaking series of beakers containing the desired pH in a known concentration wastewater. Samples of the wastewater were withdrawn at different intervals, filtered and the filtrate analyzed for the trace of COD and ammoniacal nitrogen content. The design of the experiment was done using Box-Behnken Design method of response surface analysis which specifically made each effect to require only three (3) levels given in Table 1. Then the concentrations of the samples were determined by using a calibrated curve. The removal efficiency of COD and ammoniacal nitrogen was defined as:

$$Re\ (\%) = \frac{Ci - Cf}{Ci} \times 100 \quad (11)$$

Where;

Re (%) was the ratio of difference in COD/ammoniacal nitrogen concentration before and after adsorption.

C_i was the concentration of COD/ammoniacal nitrogen before adsorption (mg/l)

C_f as the concentration of COD/ammoniacal nitrogen after adsorption(mg/l)

The amount of COD/ammoniacal nitrogen adsorbed at time t, (q_t) was calculated using the formula above.

$$q_t = \frac{Ci - Cf}{Ci} \times V \quad (12)$$

Where;

V was the volume (L) of the wastewater used for adsorption. M was the mass (g) of the adsorbent used.

2.2.8 Reverse Osmosis Experiment

Reverse osmosis (RO) experiments were carried out in batch on the removal of COD and ammoniacal nitrogen from industrial wastewater to study the effect of some specific process parameter using a single reverse osmosis skid as described by [13]. The parametric effects of temperature, contact time, and pH were investigated for the removal of COD and ammoniacal nitrogen by passing the wastewater through a single reverse osmosis skid that contains separation membranes. Wastewater of 1000ml with pH range of (5.5 to 7.5) and temperature range of (25ºC to 35ºC) was transferred into a small-sized feed tank connected to the suction of a small pump with suction pressure of 1.5kg/cm^2 and discharge pressure of 4kg/cm^2. Like in the adsorption case, the pH of the solution was adjusted using hydrochloric acid and sodium hydroxide. Concentrations of COD and ammoniacal nitrogen in the treated water were analyzed using the spectrophotometer. Batch reverse osmosis experiments was carried out at the temperature range of 25ºC to 35ºC by shaking series of beakers containing the desired pH in a known concentration wastewater. Samples of the wastewater were withdrawn at different intervals, from the skid and analyzed for the trace of COD and ammoniacal nitrogen content. As in the adsorption, the design of this experiment was done using Box-Behnken Design method of response surface analysis which specifically made each effect to require only three (3) levels given in Table 1. The concentrations of the samples were determined by using a calibrated curve.

The removal efficiency of COD and ammoniacal nitrogen was defined as:

$$Re(\%) = \frac{Cf - Cp}{Cf} \times 100 \quad (12)$$

Where;

Re (%) was the ratio of difference in COD/ammoniacal nitrogen concentration before and after the reverse osmosis experiment.

C_f is the COD/ammoniacal nitrogen concentration in the wastewater before the reverse osmosis experiment (mg/L)

C_p is the concentration of COD/ammoniacal nitrogen in the permeate (wastewater) after the reverse osmosis experiment (mg/l)

To know the amount of COD/ammoniacal nitrogen removed at those time intervals, the concentration of COD and ammoniacal nitrogen in the reject or concentrate can be analyzed.

$$Re(\%) = \frac{Ci - Cf}{Ci} \times 100 \quad (13)$$

Where;

Re (%) was the ratio of difference in COD/ammoniacal nitrogen concentration before and after adsorption.

C_i was the concentration of COD/ammoniacal nitrogen before adsorption (mg/l)

C_f as the concentration of COD/ammoniacal nitrogen after adsorption (mg/l)

3. Results

The results of the characterization of the wastewater before treatment are shown in Table 2. The pH obtained was 6.0. while the turbidity was 1.4 NTU. The COD in the wastewater was 412 Mg/l and the ammoinacal nitrogen was 869 Mg/L; these needs to be reduced before ejecting such waste water into the body water in the environment.

Table 2: General Characterization of Wastewater before the Adsorption and Reverse Osmosis Experiments

Constituents	Value
Turbidity	1.4 NTU
Total suspended solids (TSS)	3.6 Mg/l
Total dissolved solids (TDS)	326 Mg/l
Total organic carbon (TOC)	0.1 Mg/l
Chemical oxygen demand (COD)	412 Mg/l
Ammoniacal nitrogen	86 Mg/l
Biological oxygen demand (BOD)	5.2 Mg/l
pH	6.0
Conductivity	501 S/m
Total phosphate	0.72 Mg/l
Nitrates	1.5 Mg/l
Iron (Fe)	0.1 Mg/l
Copper (Cu)	0.8 Mg/l
Zinc (Zn)	0.5 Mg/l

From the Figure 1, it can be shown that Ammoniacal Nitrogen percentage removal is more prominent with adsorption process than with reverse osmosis. This is because comparing contact time of 30 to 100seconds, it was noticed that for adsorption; the percentage removal of Ammoniacal Nitrogen has a maximum value of (98%) at 30seconds while for reverse osmosis; the percentage removal of Ammoniacal Nitrogen has a maximum value of (82%) at 100seconds. Also, from Figure 2, it can be shown that COD Nitrogen percentage removal is more prominent with adsorption process than with reverse osmosis. This is because comparing contact time of 30 to 100 seconds, it is noticed that for adsorption; the percentage removal of COD has a maximum value of (88.9%)

at 100seconds while for reverse osmosis; the percentage removal of COD has a maximum value of (85.4%) at 65seconds.

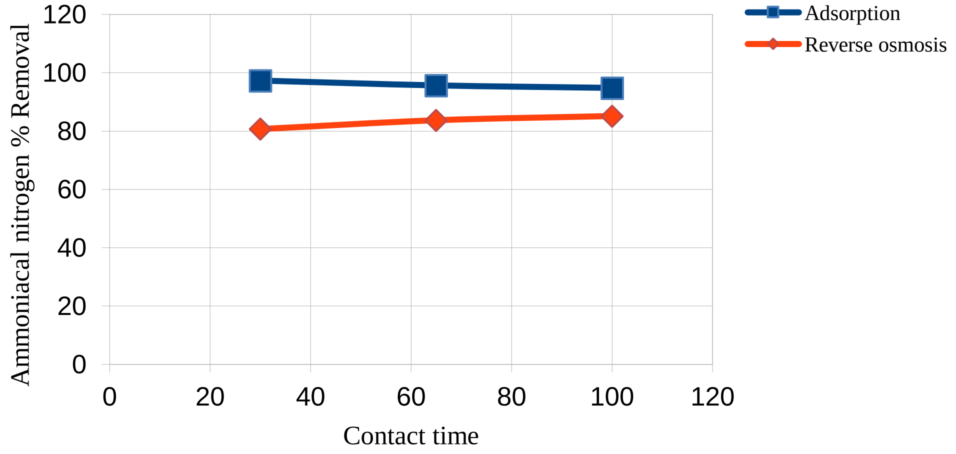

Figure 1: Graph of Ammoniacal Nitrogen percentage removal versus contact time for both Adsorption and Reverse Osmosis

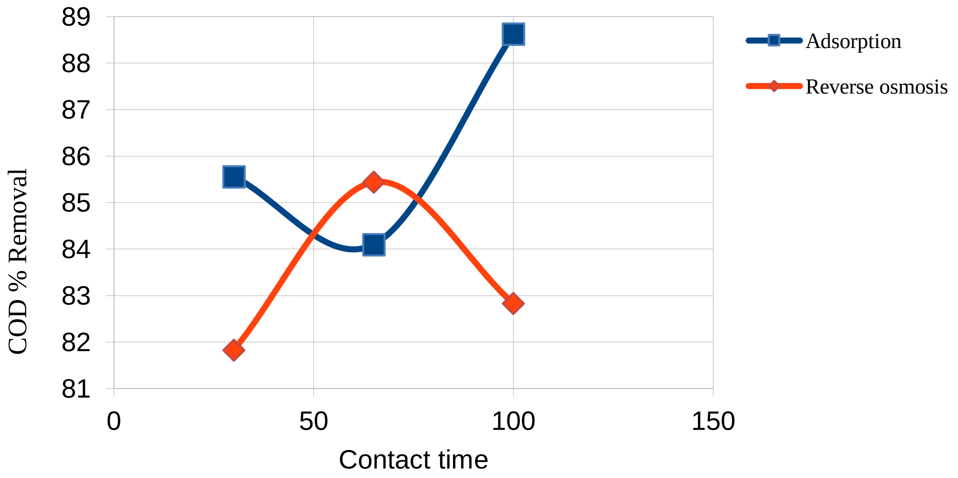

Figure 2: Graph of COD percentage removal versus contact time for both Adsorption and Reverse Osmosis

From Figure 3, it can be shown that Ammoniacal Nitrogen Nitrogen percentage removal is more prominent with adsorption process than with reverse osmosis. This was because comparing temperature of 25 to 35^{o}C, it was noticed that for adsorption; the percentage removal of Ammoniacal Nitrogen has a maximum value of (98%) at 30^{o}C while for reverse osmosis; the percentage removal of Ammoniacal Nitrogen has a maximum value of (82%) at 30^{o}C.

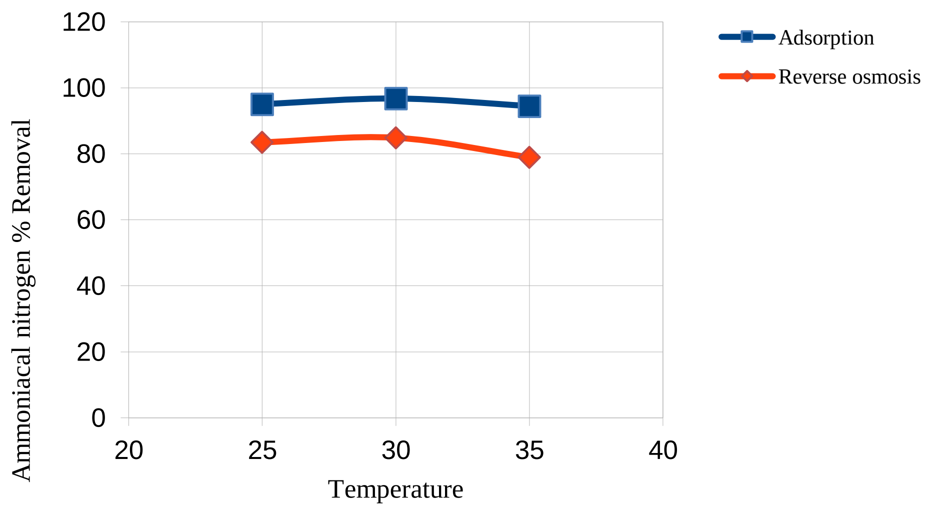

Figure 3: Graph of Ammoniacal nitrogen percentage removal versus Temperature for both Adsorption and Reverse Osmosis

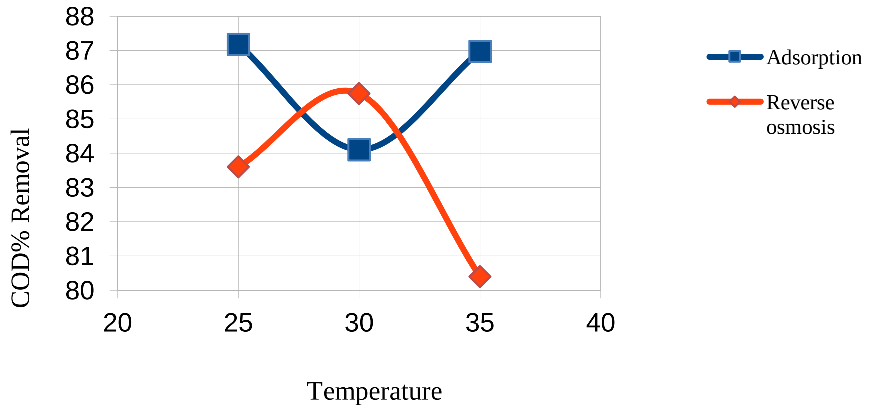

Figure 4: Graph of COD percentage removal versus Temperature for both Adsorption and Reverse Osmosis

From Figure 4, it can be shown that CODpercentage removal is more prominent with adsorption process than with reverse osmosis. This is because comparing temperature of 25 to 35ºC, it was noticed that for adsorption; the percentage removal of COD has a maximum value of (87.2%) at 25ºC while for reverse osmosis; the percentage removal of COD has a maximum value of (85.5%) at 30ºC. From Figure 5, it can be shown that Ammoniacal nitrogen percentageremoval is more prominent with adsorption process than with reverse osmosis. This is because comparing pH of 5.5 to 7.5, it is noticed that for adsorption; the percentage removal of Ammoniacal nitrogen percentagehas a maximum value of (98%) at 6.5 while for reverse osmosis; the percentage removal of Ammoniacal nitrogen percentage has a maximum value of (85%) at 6.5.

From Figure 6, it can be shown that COD percentage removal is more prominent with adsorption process than with reverse osmosis. This is because comparing pH of 5.5 to 7.5, it is noticed that for adsorption; the percentage removal of COD has a maximum value of (89.7%) at 6.5 while for reverse osmosis; the percentage removal of COD has a maximum value of (85.7%) at 6.5.

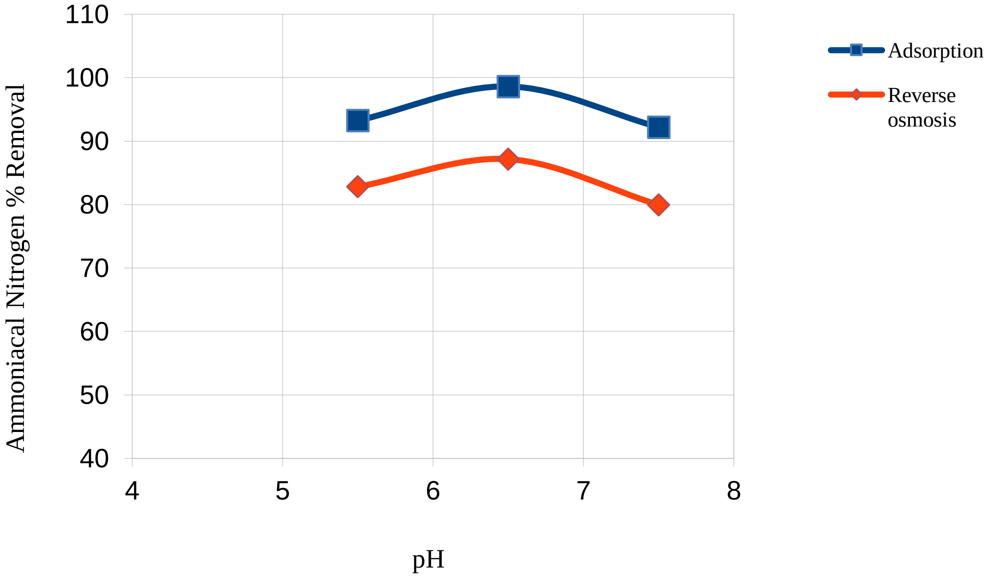

Figure 5: Graph of Ammoniacal nitrogen percentage removal versus pH for both Adsorption and Reverse Osmosis

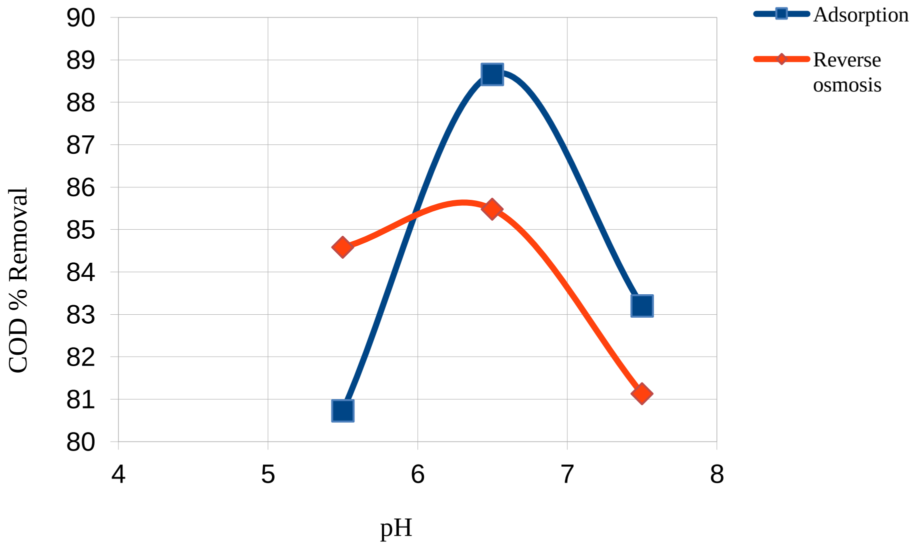

Figure 6: Graph of COD percentage removal versus pH for both Adsorption and Reverse Osmosis

4. Conclusion

In this study, the adsorption study includes the characterization of adsorbent using three parameters: moisture content, bulk density, surface area and the values ascertained are 22.5%, 0.497g/cm^3, 967m^2/g respectively and the reverse osmosis study was successfully carried out using polyvinyl pyrrolidone grafted on polyvinylidiene fluoride type of membrane with pore size 0.02mm so as to ensure that maximum pollutant/salt rejection is high. Furthermore, the statistical methodology, Box-behnken response surface methodology was demonstrated to be effective and reliable in finding the optional conditions for the high removal efficiency of COD and Ammoniacal nitrogen by adsorption and reverse osmosis and the results showed that the parameters considered have significant effects on the COD and Ammoniacal nitrogen removal using those methods. At pH = 6.5, Temperature = 30°C and contact time = 30s, Ammoniacal nitrogen has maximum percentage removal of 98% and at pH = 6.5, Temperature = 25°C and contact time = 100s, COD has a maximum percentage removal of 90%. However, at pH = 6.5, Temperature = 30°C and contact time = 100s, Ammoniacal nitrogen has maximum percentage removal of

85% and at pH = 6.5, Temperature = 25ºC and contact time = 65s, COD has a maximum percentage removal of 85%. Adsorption method is better than the reverse osmosis method for the removal of COD and Ammoniacal nitrogen from wastewater as it gave a higher percentage removal of 98% for Ammoniacal nitrogen and 90% for COD.

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