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Clay - Based Approach for Nitrate Reduction in Orogodo River Southern Nigeria.

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Abstract
Studies on Nigeria clays using X- diffractometer and Atomic Absorption Spectrometer in Reducing of Nitrates from Orogodo River was conducted. Clays were coded UD and AU and Saponite, Montmoriillorite, Chloride, illite, mixed layer, Kaolinite and Quartz was found in UD AU chloride, Illite, Kaolinite, Quartz and Hematite were present in AU. AG has Quartz mixed layer, illite, chloride, saponite, Kaoline and Hematite they were alluminosilate clays with $Si0_2$ and Al_20_3 present. The minimum efficiency removal of nitrate removal was (35% -44.90%) using batch and continuous method was (88.00%-93.41%)- other parameters were measured (p^H, Temperature)Dissolved Oxygen (DO), they were also reduced using residence time. There was significant reduction in the pollution parameters using performance efficiency of ration (1:2) which gave optimum reduction properties with the permissible limit for crop production (irrigation). The study showed that clay possesses potential for removing nitrates from rivers using local clays which is readily available and environmentally friendly.
Keywords: Clays, Environment, Nitrates, Water, Residence, Time.

1. Introduction

Most of the water on earth is not potable for irrigation or drinking purpose most are sealed in glaciers and ice caps (Adeola, et al, 2015). Fresh water and rivers have less than one-ten-thousandth of the water on earth. Water is essential for socio-economic development and maintenance of a healthy ecosystem. It is a social and political issues in developing nations (Matsouele et al, 2022). Water is essential for human, economic, health and environmental growth. It is of limited resources on earth in terms of quality and quantity (Fermandez et al, 2023. Ground water and surface water pollution have reduced fresh water availability. This is a vital issue in the Sustainability Development Goal (SDG) in the 2023 agenda adopted by United Nations Member States. (Sachs, 2012)

Major water pollutants are from anthropogenic, agricultural and industrial wastes from untreated effluents (Adesuyi, et al 2015). Nitrogen and Phosphorus are the primary pollutants in water (Abowei, et al, 2010) Nitrate ($N0_3$) is a major form of pollutant in natural and surface water. (Jagessar, et al, 2011) others are NH_3, ammonium etc. Surface water rarely contain as much as 5mg/l but when inorganic fertilizers are used, it may rise to 1000mg/l from human activities like agrochemicals, waste water for irrigation and animal waste. It is an essential element in DNA, and chemical processes in our body, plant growth and Nitrogen fixation. It can cause algee bloom in combination with phosphorus, which produces toxins to organisms, prevent boat access or swimming and deplete the amount of oxygen when the plants die, it sinks to the bottom, decomposes and depletes dissolved oxygen in water making it unfit for aquatic life and death of macroinvertebrates resulting in "eutrophication".

Nitrates is implicated in major health problems-interfering with blood ability to carry oxygen to vital tissues in infants of six months or younger called "Methmaglobinemia or blue baby syndrome", (Zohre et al 2021) pregnant women show risk of miscarriage or birth defects (Eldndge, 2012), people with insufficient stomach acid to metabolize and excrete nitrate, those who lack the enzyme methemaglobin reductase which converts affected red blood cells back to normal. Long

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term exposure can lead to diversis, starchy deposits, heammorhaging of the spleen and cancer, decrease in blood pressure and increase risk or thyroid disease (Zhai, et al, 2017). Nitrate could be converted to nitrite in the digestive system which react with nitrosotable compounds to produce N-nitroso compunds (NOC_s) which are secondary amines in food stuffs suspected to be carcinogenic (Rezvanu et al, 2021).

The world Health Organization (WHO) maximum limit is 50mg/l of nitrate in drinking water, studies have shown relevant levels of nitrates in Niger Delta Rivers. Nitrate levels greater than 10ppm exceeds state and federal standards for public drinking water supply (Tanega, et al, 2010). The Orogodo river receives run-off from farms using organic and inorganic fertilizers, pesticides, water from different abattoirs and also for irrigation. Various methods like reverse Osmosis, ion exchange that are capital intensive have been used. In this study, physicochemical analysis of water and reduction of nitrate using local kaolinite clay is used. It is cheap, abundance in the locality, easy to manipulate and environmentally friendly.

2. Material and Methods

2.1 Study Area

The study area is located between latitude 5.01 - 6.021N and longitude 6.010 - 6.261E about 50km in length fed by seepage quifer from rainforest in Mbiri Delta State. It runs through Agbor, Owa-Ofie, Ekwuoma, Abavo Oyoko and ends in River Ethiope Southern Nigeria, with friable sand, beds and intercalation of gravely units capped by laterrite soil followed by fine grained sand of different thickness between 9-58 meters (Bulouebibo et al, 2019, Iwegbue, 2012).The location is shown in the figure 1.

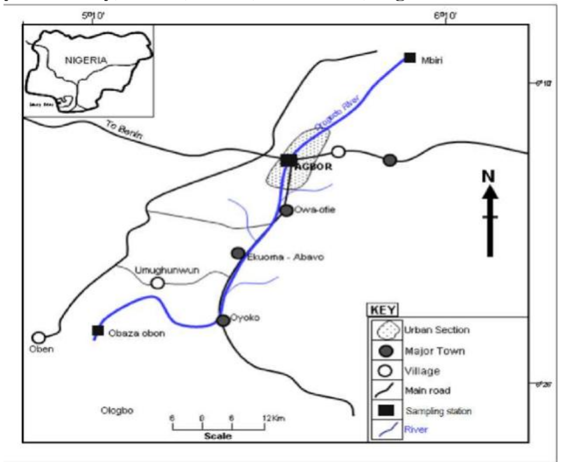

Figure 1: Map of Orogodo River, Agbor

2.2 Sample Collections

Three clay samples type were collected from Uduophori-Bomadi Local Government Area, AU, Auchi_ Edo State and Agbor Ika South L.G.A in Delta State. This was done using hand chisel and hammer, they were air dried, pulverized and sieved with 2nm sieve, stored in a clean polythene bag. Smooth stone pebbles were collected from Ovwian waterside in Udu Local Government Area of Delta State. They were carefully washed, dried and packed in cellophone bags. Suitable amount of river water was collected at different times in 50litres clean plastic containers at seperate sampling points. Plastic vats with columns of 150cm and diameter of 15cm were set in batch and continous packed with glass wool to 10cm at the base. Carefully mixed quantities of pebbles and clay in ratio 1:2 was used for optimum percentage reduction.

2.3 Method of Clay Analysis.

Mineralogical analysis was done using X-ray diffractometer PW 1800 powder of Philips. 2.5g pulverized, mixed with sodium based-coagulant and smeared in a thin layer into the X-ray diffractometer crossed match using XSPEX version 5.62. Geochemical analysis was done using Atomic Absoprtion Spectrphotometer GBS scintific after digestion (APHA, 1995, Lenore, et al, 2009).

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Precolation studies was carried out to determine the residence time of the river water for the time to collect 100ml was noted.
Cation exchange capacity was done using (Pages et al, 1995). Temperature was determined using digital thermometer, pH using HACH PH meter HQ20. Dissolved oxygen was done by alkaline - azide modificatior method of wrinkles using HACH DO Meter, nitrate using sodium saliyhlatc Colourimetric method with HACH DR 500 spectrophotometer. The mean and standard deviation, minimum, maximum and percentage reduction was done using SPSS version 25.

3. Results and Discussion

3.1 Results

Mineralogical Results of Clay.

The results obtained for percentage composition of clays is as shown in Table 1.

Table 1: Composition of clays used for this study in percentage (%)

Clay Mineral	*UD*	*AG*	*AU*
Hematite	1.20	7.30	6.70
Quartz	11.40	30.20	54.40
Mixed layer (montmorillonite)	20.10	3.10	0
Illite	8.20	10.90	2.30
Chloride	18.30	4.30	2.10
Smeetite	7.20	0	3.00
Saponite	4.70	1.20	0.50
Kaolinite	28.80	43.10	31.00

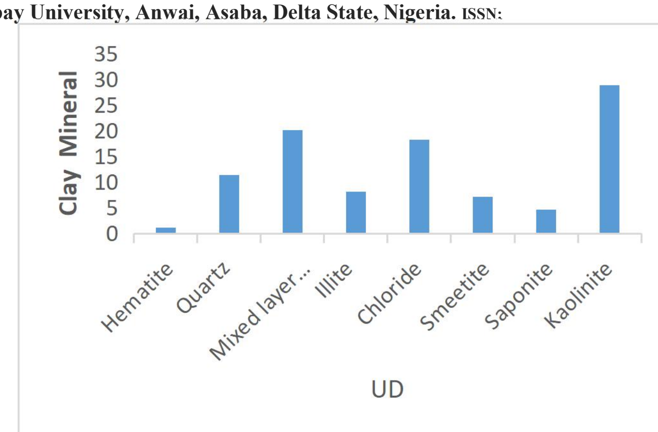

Figure 2: Composition of Clay (UD)

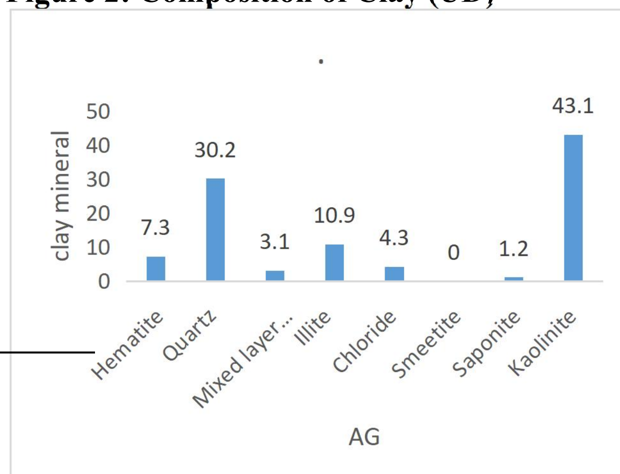

Figure 3: Composition Clay (AG)

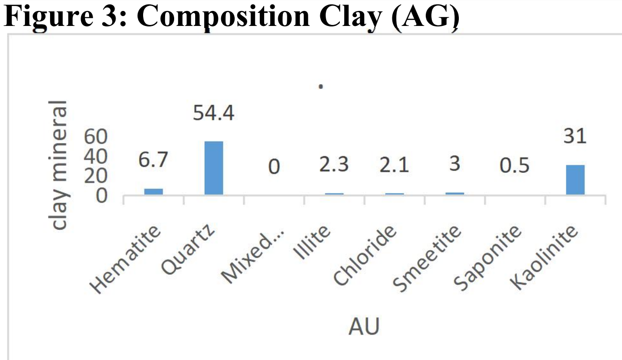

Figure 4: Composition of Clay (AU)

Table 2: Composition (%) of oxides from geochemical analysis of clay

% Oxide	UD	AG	AU
H_20	2.00	1.01	13.14

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Ca0	1.60	1.21	1.43
$Ti0_2$	0.80	0.93	0.71
K_20	1.86	1.60	3.00
Mg0	1.74	1.80	1.02
Na_20	1.00	0.98	1.60
$F_{e2}0_3$	8.89	5.89	2.90
Al_20_3	35.90	40.90	29.30
$Si0_2$	46.17	43.16	50.06

H_20 Structural water

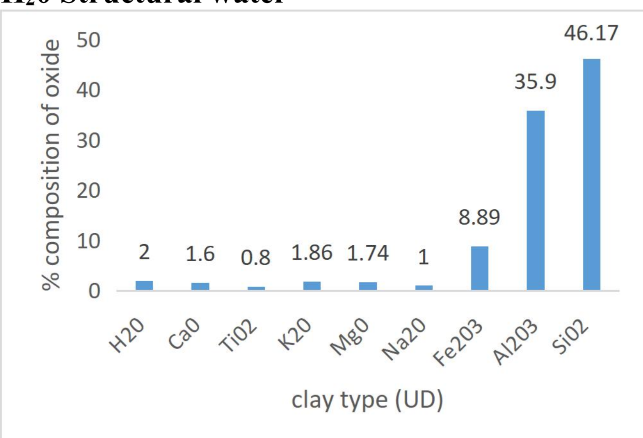

Figure 5: Percentage Composition of Oxide in Clay (UD)

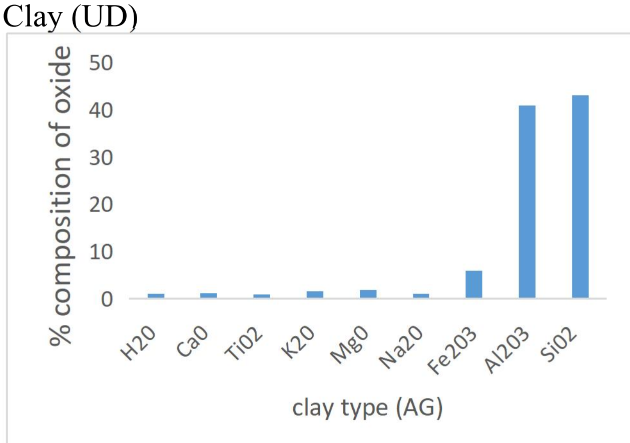

Figure 6: Percentage Composition of Oxide in Clay (AG)

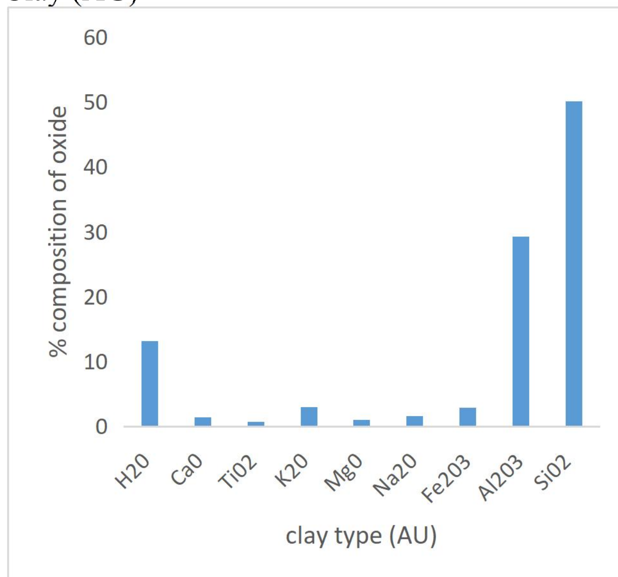

Figure7: Percentage Composition of Oxide in Clay (AU)

Residence Studies and Cation-Exchange capacity

The time to obtain the first drop from each column varied with the mineralogical compositions of the clays as shown in Table 1

Table 3: Residence Studies and Ca-Exchange Capacity

Clay Sam	***Time to ol 100ml***	***Cation-Exchange capacity***
UD	2 hours 01 minut	78
AG	4 hours 13 mir	**32**
AU	3 hours 01 mi	**10**

Table 4: Percentage Reduction for Batch Column of Nitrate Level

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			PH		Temperature ^{0}C			Do mg/l			Nitrates		
Site	Raw Values	UD	AG	AU	UM	AG	AU	UD	AU	AG	UD	AU	AG
1		5.40	5.90	5.71	26.90	25.80	26.20	4.10	4.52	4.61	42.31	15.00	48.23
2		5.38	5.92	6.00	27.70	30.50	24.20	4.31	4.83	4.20	39.00	23.21	39.21
3		5.41	6.00	6.10	24.80	25.90	25.20	4.53	4.31	4.23	36.55	25.01	30.24
4		5.41	6.10	5.80	27.50	27.90	26.20	4.70	4.50	4.62	25.20	27.40	27.45
5		5.37	6.00	5.81	26.20	25.30	24.10	4.82	4.46	4.61	28.13	36.55	25.50
6		5.40	5.82	5.90	26.50	29.70	25.20	4.84	4.47	4.70	27.31	27.45	23.20
7		5.42	5.87	6.00	28.90	29.80	26.10	4.90	4.33	4.81	36.45	31.60	15.00
8		5.41	5.88	6.00	22.50	24.90	25.00	4.91	4.53	4.62	38.36	35.21	36.60
9		5.40	5.90	6.10	24.10	25.20	24.72	4.40	4.68	4.58	25.21	38.20	27.40
10		5.41	5.89	6.00	28.20	26.00	26.20	4.42	4.70	4.54	28.10	39.00	25.50
Min		5.38	5.87	5.71	24.10	24.90	24.20	4.10	4.31	4.81	25.20	15.00	15.00
Max		5.42	6.10	6.10	28.90	29.80	26.20	4.92	4.70	4.20	42.31	39.00	48.23
Mean + SD		0.010, ±0.0097 5.40	0.082 ±5.99	0.132 5.912	1.992 ±26.50	2.085 ±27.35	0.0828 ±25.20	0.2181 ±4.40	0.163 ±4.51	0.192 -4.51	61.62 ±28.76	7.673 ±27.00	9.254 ±31.62
% Reduction											41.31%	44.90%	35.50%

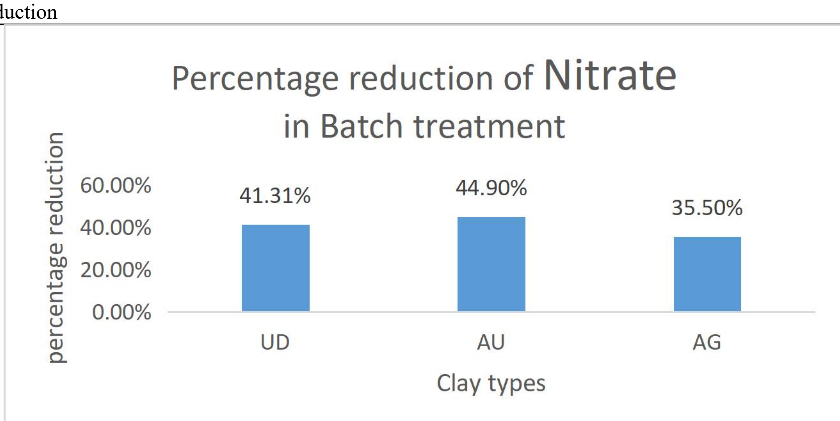

Figure 8: Reduction of Nitrate by Different Clay Types in Batch treatment

Table 5: Percentage Reduction of Nitrate Level Continuous Column

		PH		Temperature ^{0}C			Do mg/l			Nitrates		
Site	UM	AG	AU	UD	AG	AU	U	AU	AG	UD	AU	AG
1	6.81	7.10	6.70	26.20	23.60	28.50	7.81	6.30	5.31	3.60	5.37	5.56
2	6.80	7.20	7.20	25.80	22.10	27.90	6.60	6.51	5.60	4.20	5.41	5.60
3	6.80	6.90	6.90	30.00	28.90	26.70	7.74	6.00	5.41	4.31	4.82	5.50
4	6.90	6.80	6.80	29.70	30.40	28.60	7.80	5.90	5.61	3.80	4.53	6.20
5	7.01	7.00	7.00	26.50	25.80	27.60	7.60	6.70	5.74	2.14	4.60	5.80
6	7.01	7.10	7.10	27.50	27.20	26.90	7.65	6.40	6.20	4.28	4.80	5.80
7	7.30	6.62	6.62	26.50	26.70	26.50	7.60	6.20	5.75	4.30	4.70	6.00
8	6.80	7.00	7.00	27.50	25.80	30.50	6.10	7.00	6.82	4.30	4.82	6.10
9	6.75	7.10	7.10	28.70	27.70	29.60	7.10	7.20	6.80	4.32	5.30	5.75
10	6.80	6.90	6.90	28.50	27.80	27.70	6.00	6.80	7.21	4.30	5.30	5.80
Min	6.80	6.62	6.62	26.20	22.10	26.50	6.00	6.00	5.31	2.14	4.70	5.50
Max	7.30	7.20	7.20	30.00	30.40	30.50	7.74	7.20	7.21	4.32	5.37	6.20
Mean + SD	0.162 ±7.05	0.172 ±6.91	0.172 ±6.91	1.481 ±28.10	2.43 ±26.25	1.300 ±28.50	6.50 ±0.682	6.60 ±0.424	6.26 ±0.672	3.23 ±0.685	5.04 ±0.357	5.85 ±0.233
% Reduction										93.41%	89.71%	88.06%

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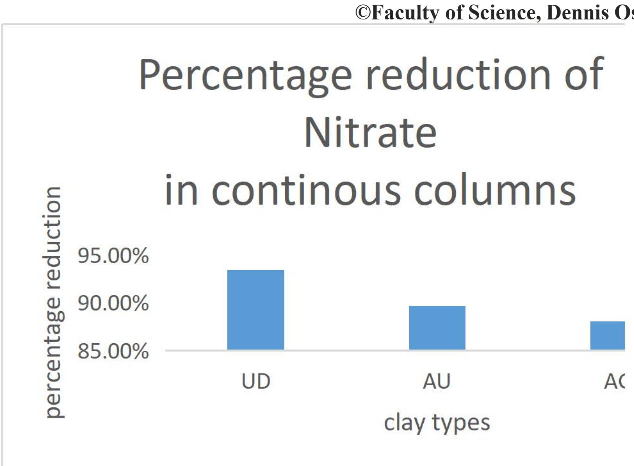

Figure 9: Reduction of Nitrates by different Clay Types in Continuous Columns

3.2 Discussion of Results

The mineralogical composition of each clay type revealed all clays contains the groups listed above in Table 1 except mixed layer (montmorillonite which was absent in clay AU. They are present in different concentrations,UD haemite 1.20%, quartq11.40% mixed layer 20.10%, illite 8.20%, chlorite18.30% smectite 7.20%, Saponite 4.70 and Kaoline 28.80% for AG Haemitite 7.30%, quartz 30.20%, mixed layer 3.10%, illite 10.90%, chloride 4.30%, smectite nil, saponite 1.20% and Kaolin 43.10 smectite%. AU Hematite 6.70%, quartz, 54.40%, mixed layer mean, illite 2.30%, chloride 2.10, smectite 3.00% saponite 0.50 and Kaoline 31.00%. Each clay mineral has its own unique mineralogical composition as shown in figures 2 ,3 and 4

From the results in Table 2 and figures 5 ,6 7, the clay samples were all alumino – silicate clays with UD having the highest level of F_20_3. Na_20 and Tio_2 impurities were relatively low. From the aluminum content of the clay mineral, they are high in Kaolinite clays (content).

The result from Table 3 shows that, the lower the residence time, the lower the percolation rate and vice versa. The higher the cation exchange capacity especially for Kaolin clays the better the rate of pollutant reduction (nitrates) (Mohammed 2020, Mansenete et al, 2022). Cation exchange capacity increases with increase in pH, as pH increases, the hydrogen held by the organic colliods and silicate clays as kaolinites, becomes ionize and is replaceable while aluminium hydroxyl ions are removed forming $Al(OH)_3$. For nitrate removal/reduction in the batch treatment method as shown in Table 4 there was reduction as Illustrated in figure 8, the higher the cation exchange capacity value, the lower the amount of nitrate. This explains why clay UD and AG reduces more nitrate than AU.

River water was passed through a percolating medium of pebble clay in ratio 1:2, the effluent collected and analyzed. The results are shown in Table 4 and 5 respectively. The result showed the pH to be alkaline. From the results, the values of nitrate in UD were more reduced in the continuous treatment method compared with the batch treatment method probably because of the mineralogical assemblage of the clay mineral. In the continuous method, three percolating media were connected in series with continuous flow of river water from the first to the third column. This is shown in Table 5 and figure 9 A general overview of the parameters showed an improvement in the quality of water.

The pH of a body of water determines water quality since it affects its solubility and metal toxicity (Aeola, et al, 2015). These were higher than those recorded by Seiyabro et al, 2013. Temperature was within (20-33^{0}c) as recommended by USEPA, EPA as for aquatic life in the tropical region which ranges between (24.10- 30.40^{0}c). The low

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temperature in section 8 could be due to high shielded plants.

The DO varies slightly for each of the treatment methods between 4.10 - 4.81mg for batch treatment and 6.00 - 7.21 for continuous treatment. It evaluates the freshness of a body of water and for aquatic organism survival. The nitrate levels were high for batch between 42.31 - 48.23 as compared to the recommended values of FEPA limits for nitrate but a little lower than public drinking water 50mg/l standard values of (50mg/l) in $N0_3$-N concentrations. High nitrate level in the river could be traced to run-off from Arbitrators, fertilizer used in farming, pesticides, irrigation on the upper side of the river, human waste in sewage, organic matter decomposition in surface water, inorganic nutrients which results in eutrophication and ecological impairment of water body (Qasemi, et al, 2018). This oxygen depletion can result in dead zones where no marine life can survive. Although the total mean concentration was a little lower than the recommended standard, monitoring of the above-mentioned activities should be done cautiously. After the continous treatment method, the concentration of nitrate was reduced to 99.411 for UD, 89.71 for AG and 88.06 for AU in continuous treatment method as shown in figure 9 whereas in the batch treatment method, UM was 41.31%, AU 44.90% and AG 35.50% depicter in figure 8. Although nitrate carcinogenicity is not proven, however nitrosammines and other carciogenic compounds are formed due to the reaction of secondary amines with the productsof nitrate reduction in the body (Huary et al, 2017). Nitrate in the body should be controlled for health protection. It can be consumed through water, food-through path ways. The best solution is to find, reduce or eliminate sources of nitrate pollution. Nitrates leads to negative impact on aquatic ecosystem and wildlife, excessive growth of algae and cyanobacteria can disrupt food chains, leading to a decline in fish population & other species, decrease or negative impact on bird populations that depends on healthy aquatic ecosystems for their survival and migration. (Matel, 2021).

4. Conclusion

A survey of the results of the treatment methods showed variation in the capacity of each clay column for the removal of nitrates traceable to the mineralogical assemblage. In the percolation rate study, water was lower in that containing more smectite residence time in UD was longer than AU than AG. The longer the residence time, in the media, the more efficient the reduction process of nitrate. There was reduction in batch treatment method but was remarkably noticeable in the continuous treatment method, as seen from the percentage reduction of nitrate and an increment in DO values. Using clay: pebble media is a good medium for nitrate removal/reduction. The more the columns employed, the better the improvements in water quality. (Raze, 2015) it is important to monitor nitrate levels in water to prevent the adverse effects & ensure the safety of both human and the environment.

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