



Removal of nitrate from contaminated water of river Rushikulya using natural adsorbent

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ABSTRACT

Nitrate contamination of surface water through different human activities is an alarming issue for human sound health survivorship. So, we have attempted to estimate and remove nitrate contamination levels in water samples, collected from the Rushikulya River in Odisha, India. A detailed study is done on the impact of nitrate contamination in various aspects and their removal is absorbed through an experimental composite adsorbent that is prepared taking rice husk dust and Bagasse waste from corn (Argo-based waste product) with different proportions of each. The nitrate absorbance was analyzed with the spectrophotometric method. The efficiency of adsorbents is identified through SEM and found that with an increase in the alkaline state of water sample up to 13 and composite proportion ratio with 20% rice husk and 80% bagasse waste from corn, removes nitrate up to 95% from contaminated water sample. A maximum of 98.0% of nitrate was removed from the water at a pH of 11. Also, by maintaining the COD range of 250 mgL⁻¹, the nitrate could be removed up to 94%. Out of all the combinations, the matrix composite of Type- 9 (CM - 9), made with the combination of RH and BW in Ratio 1:9, is found to have with good compressive strength of 0.44 MPa, tensile strength of 1.003 MPa and highest density of 279.88 kg m⁻³. These matrices are applied for the treatment of contaminated river water samples to remove nitrate.

1. Introduction

Deterioration of surface water is not a new phenomenon however, quality of surface water changes from time to time worldwide, due to both natural processes and anthropogenic activities. Due to increasing population, urbanization, and extensive agricultural practices, the surface and groundwater are contaminated. Over the last 20 years, several small and large-scale industries have been developed in the basin of the Rushikulya River. This river feeds the maximum agricultural and consumptive needs of two major districts of the state

of Odisha, India. This River is also known to be the second largest olive ridley turtle nesting site at its river mouth in India. Hence, the water quality of this river influences human and animal life living nearby directly or indirectly. This research paper attempts to determine the anionic parameter nitrate, in the river water sample, which is mostly responsible for damaging the water quality and making the water unhealthy for domestic as well as consumptive purposes. So, towards remedial measures of treatment of Nitrate, an efficient adsorbent matrix made up of agro-based waste products is prepared and subsequent treatment is experimented. The observations are estimated, and recordings are analyzed. In this research, the organic contamination of the river water is also analyzed.

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Nitrate remediation is mostly attempted by engineered processes and experimented with for the efficient removal of Nitrate. A few of them are discussed here. Nitrate contamination in the groundwater is a global issue which took so much research interest. Many natural and engineered processes have already been experimented for removing it. The reviews show that microbial autotrophic and heterotrophic mechanisms are efficient enough for nitrate removal from groundwater by the in-situ and ex-situ processes. It is found, that engineered processes can remove a much better percentage of nitrate than natural methodologies [1]. The hazardous impact of nitrate contamination in drinking water on human health is critically analyzed [2]. The effectiveness of biological denitrification against reverse Osmosis and ion exchange methodology for nitrate removal from groundwater are studied for full-scale application [3]. A detailed photocatalytic removal of nitrate from water using activated carbon loaded with bimetallic Pd-Ag nanoparticles under solar radiation is given by Soliman A. M. *et. al.* [4]. A quantities assessment of factors influencing nitrate accumulation in groundwater is studied and biological denitrification using cyanobacterial is found an effective method for removal on a large scale [5]. Nitrate removal from water using amine-grafted agricultural waste adsorbents is given by Kalaruban M. *et. al.* (2016) [6]. The removal of nitrate and fluoride using membrane and adsorption technique is given by Munoth P *et. al.* (2015) [7]. Efficient and continuous removal of nitrate from water with cationic cellulose nano paper membranes is given by Mautner A *et. Al.* (2017). The performance of these cationic nanopapers exceeds $100 \text{ L m}^{-2} \text{ h}^{-1} \text{ M Pa}^{-1}$ [8]. Electrodialysis and electro-deionization technology are applied and attempted for the removal of nitrate from groundwater. It was a fundamental in-situ remediation method and provided the foundation for further research on the removal of nitrate using ED and Ion exchange methods [9]. A pilot-scale biochar-amended woodchip bioreactor was developed by Ashoori *et al* in 2019. They remove nitrate, metals, and trace organic contaminants from urban stormwater. [10]. Critical analysis of the hazardous impact of nitrate contamination in groundwater and their sources of contamination is conducted [11]. Some research concluded that anthropogenic pollution is the main source of groundwater pollution and towards remedial

measures Ion exchange, biological denitrification, and reverse osmosis are used on full scale for their removal. Biological denitrification from drinking water found that autotrophic denitrification is relatively more complex than heterotrophic denitrification [12]. The removal of nitrate was attempted with a Sulfur-siderite autotrophic denitrification system (SSAD) to remove phosphorous and conducted with batch, column, and pilot mode. This method was found to be one of the most effective and promising ways for both phosphate and nitrate removal. Nitrate is attempted to be removed along with arsenic and fluoride from drinkable water using integrated combined techniques taking Ion exchange resin and activated alumina. Studies on the stratification of reactivity for nitrate in groundwater are attempted and discussed. Many detailed reviews are carried out to select the best methodologies among chemical precipitation, Distillation, RO, ED, IE, and bio-denitrification along with natural zeolite attempted for groundwater with suitable surface modification [13-16]. The phytoremediation process is attempted to remove nitrate from water [17]. Nitrate removal from agricultural runoff water, by the chitosan-graphite composite adsorption method is studied using bio-composite of synthesized benign, from naturally abundant chitosan by doped with graphite and SiO_2 . From kinetic analysis, this method was found to be non-expensive, and benignly prepared chitosan composites are very effective adsorbents for the separation of specific nutrients like nitrates/phosphates from water [18-24]. Mosavi *et al.* in 2009 developed a highly selective and sensitive catalytic spectrophotometric method to determine nitrite amounts in water samples. It is based on its catalytic effect on the nuclear fast red-potassium bromates redox reaction in an acidic medium [25]. Arjomandi *et al.* in 2019 discussed on quantification of heavy metals in human bodies, soil, and water. They assessed the effect of toxicity of each heavy metal on human health [26]. Moussavi *et al.* in 2015 investigated the heavy metal pollution in waters, soils, and vegetables from farms, near an oil refinery in the south of Tehran city, Iran. They used the Geographic information systems (GIS) to estimate the levels of heavy metal concentration at unmeasured locations [27]. Also, some adsorbents such as carbon nanotubes, Nitrogen-doped porous graphene nanostructured, and bimodal mesoporous silica nanoparticles were used for removal of anion and cation in water samples

[28-30]. In the present research, an attempt has been made to establish the role of composite material made up of raw rice husk and bagasse waste, to provide a medium for nitrate removal from sample water of river Rushikulya.

2. Materials and Method

2.1. Area Study

All the samples are being collected during the April-Pre-monsoon seasons, July-Monsoon, and October-

post monsoon seasons. The samples are being collected in triplicate and the mean is considered as the database for further treatment. The samples were collected in 5 L plastic containers, and thoroughly washed with water for analysis. The COD was used by the APHA method. To carry out the study, Sample water was collected from ten different areas (Table 1) near and far from industrial sites in the river basin. The location of all ten sampling sites is presented in Figure 1.

Table 1. Study Sites for Sample Collection

Name of the Site	Code
Nandika	S-1
Baradaballi	S-2
Jagamohan	S-3
Pataliguda	S-4
Pittal	S-5
Pratappur	S-6
Karapada	S-7
Kainchapur	S-8
Patagada	S-9
Pallibandha	S-10



Fig.1. Study Sites for Sample Collection in relation to Google Map

The bio-based waste raw materials are being collected from various agricultural units present nearby. The corn tree is shown in Figure 2 and the rice husk in Figure 2. After removing the corn from the plant all parts in the form of powder form can be taken as adsorbent similarly powder from corn cobs and rice husk is used to act as adsorbent shown in Figures 3 and 4.

They are mixed in different desired proportions to prepare 10 different types of composite matrix. Contaminated water is allowed to be treated through the adsorbent matrix and the efficiency of removal of Nitrate is studied under different conditions, COD, pH, and doses of composite matrix. For that 500 mL of contaminated water is taken for each reaction. SEM analysis is conducted with scanning electron microscope model no. JSM-651OLV. Nitrate is determined by the Chromotropic acid method as per IS:3025(Part 34)-1988, spectrophotometrically using UV-Vis. A spectrophotometer (Model number: SL 218, Elico, Double beam UV-VIS Spectrophotometer) was used at 410 nm [31].

2.2. Determining Procedure

Nitrate was determined by the Chromotropic acid method using a UV-Vis Spectrophotometer. Nitrate is determined spectrophotometrically by the Chromotropic acid method as per IS:3025(Part 34)-1988, spectrophotometrically at 410nm [32]. Filter photometer with violet filter was used with maximum absorbance at 400-425 nm. Dissolve 3.8 g of ammonium chloride (CAS No.: 12125-02-9)

in water and dilute to 1000 mL. Dilute 1 mL of stock solution to 100 ml of DW. The method is nitrate reacts with chromotropic acid to form a new product. First, the sample is added to a Test 'N Tube™ vial containing sulfuric acid (Hach Company Nitrogen-Ammonia Reagent Set, Thermo Fisher Scientific, USA). This sample and sulfuric acid mixture was used for calibrating by the spectrophotometer (blank, zero-point calibration). Then, the chromotropic acid was added as a nitrate reagent. Two moles of nitrate react (2.0 M) with one mole of chromotropic acid (CA: 1.0 M) to create a yellow color reaction product with maximum absorbance at 410 nm. The absorbance of CA with nitrate was caused to determine the nitrate concentration in water samples by spectrophotometer (Fig.5).

3. Result and Discussion

Due to different urbanized acts, from time to time, the water contamination level is increasing day by day. This is a high call for more advanced and innovative products designed for Pre-treatment of the wastewater from small water bodies before being discharged to a nearby river or Ocean. The data obtained for different water samples are represented in Figure 6. The data revealed that nitrate concentration in water sources is effluent addition dependent. It's all because water samples collected from sites no. 1, 2, 3, 4, and 5 are very low in nitrate content as compared to sites no. 6, 7, 8, 9, 10, site no. 7 and 9 i.e., Karapada and Patagada show maximum nitrate content ranging between



Fig. 2. Corn tree



Fig. 3. Corn cobs



Fig. 4. Rice husk

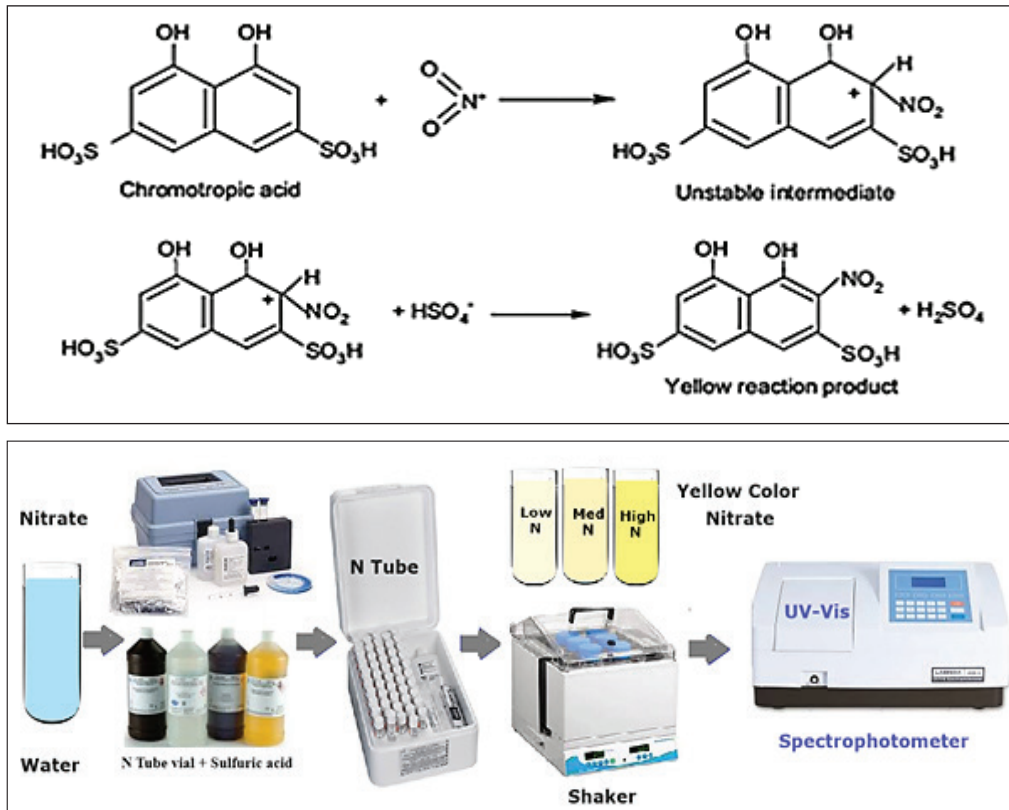


Fig. 5. Nitrate determination procedure by N Tube and UV-Vis spectrophotometer

150 mgL^{-1} to 190 mgL^{-1} which may be attributed to the fact that the addition of industrial waste from chemical industries and fertilizer industries nearby are the major sources of nitrate pollution. Also, nearby public using this water, have complaints of blue baby syndrome in children. Here, a suitable adsorbent matrix is prepared for treatment of

contaminated water samples. For the preparation of CM, rice husk and bagasse waste are chosen and are being collected from the nearby local areas. Each of these two waste materials is set for Physiochemical analysis before being added together for preparation of an efficient adsorbent matrix. The data are represented in Table 2 and Table 3.

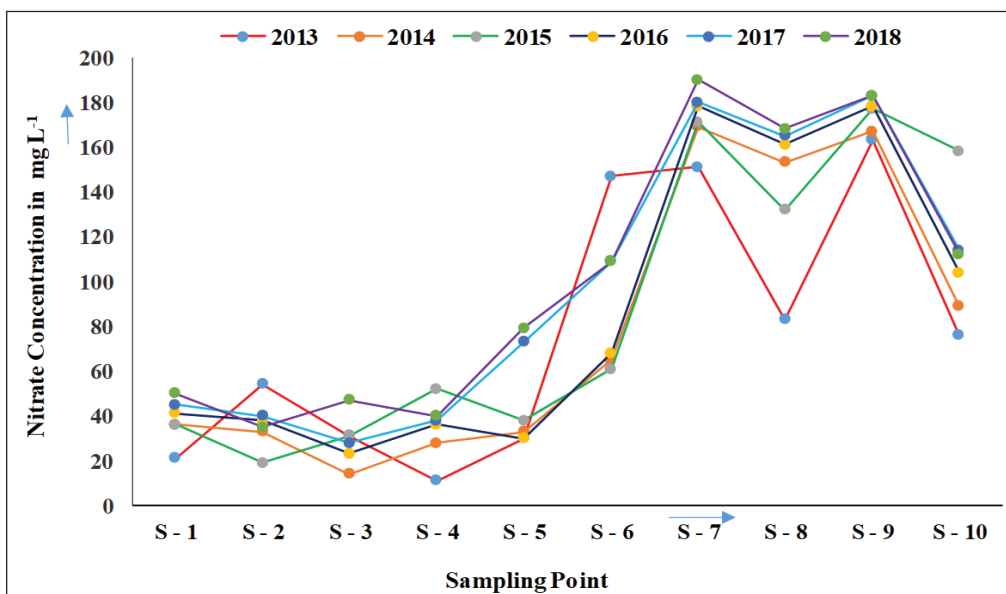


Fig. 6. Nitrate concentration profile of the sampling Sites for 2013 (mg L^{-1})

Table 2. Physical parameters of the raw material

Physical Properties	Rice Husk	Bagasse waste from Corn
Carbon: Nitrogen Ratio	65.8	92.4
pH Value	8.3	9.7
Bulk Density	118 Kg m ⁻³	310 Kg m ⁻³
Maximum water holding capacity	512 mL kg ⁻¹	588 mL kg ⁻¹

Table 3. Chemical composition of raw material (%)

Chemical Composition	Rice Husk	Bagasse waste from Corn
SiO ₂	81.08	88.2
K ₂ O	3.11	1.3
CaO	1.98	0.6
P ₂ O ₅	4.78	0.4
MgO	2.83	0.4
Al ₂ O ₃	2.01	2.3
Fe ₂ O ₃	1.59	5.1
Na ₂ O	0.53	0.1
SO ₃	0.66	< 0.1
TiO ₂	0.07	0.05
ZnO	0.02	0.08

These two bio-based waste materials are mixed in different proportion ratios, to prepare ten different adsorbent matrixes, as represented in Table 4. For the preparation of an efficient adsorbent towards nitrate removal, both the raw materials are dried and activated to attain the best absorbance efficiency, as represented in the flow sheet in Figure 7.

The activated biowaste is subsequently mixed with the addition of an organic binder and the matrix is prepared using a mechanical mold and followed by a determination of their physical properties, to

identify the most efficient matrix out of all (Table 5). It is revealed that out of all the combinations, the matrix composite of Type- 9 (CM - 9), made with the combination of RH and BW in Ratio 1:9, is found to have with good compressive strength of 0.44 MPa, tensile strength of 1.003 MPa and with highest density of 279.88 kg m⁻³. These matrices are used for nitrate removal in the contaminated river water sample. The nitrate removal is processed against a composite matrix and different conditions for efficient removal of Nitrate are studied.

Table 4. Represents the ratio for the addition of Rice Husk to Bagasse waste

Sl. No.	Rice Husk (g)	Bagasse waste (g)	Ratio (RH:BW)
S - 1	90	10	9:1
S - 2	80	20	8:2
S - 3	70	30	7:3
S - 4	60	40	6:4
S - 5	50	50	5:5
S - 6	40	60	4:6
S - 7	30	70	3:7
S - 8	20	80	2:8
S - 9	10	90	1:9
S-10	00	100	0:10

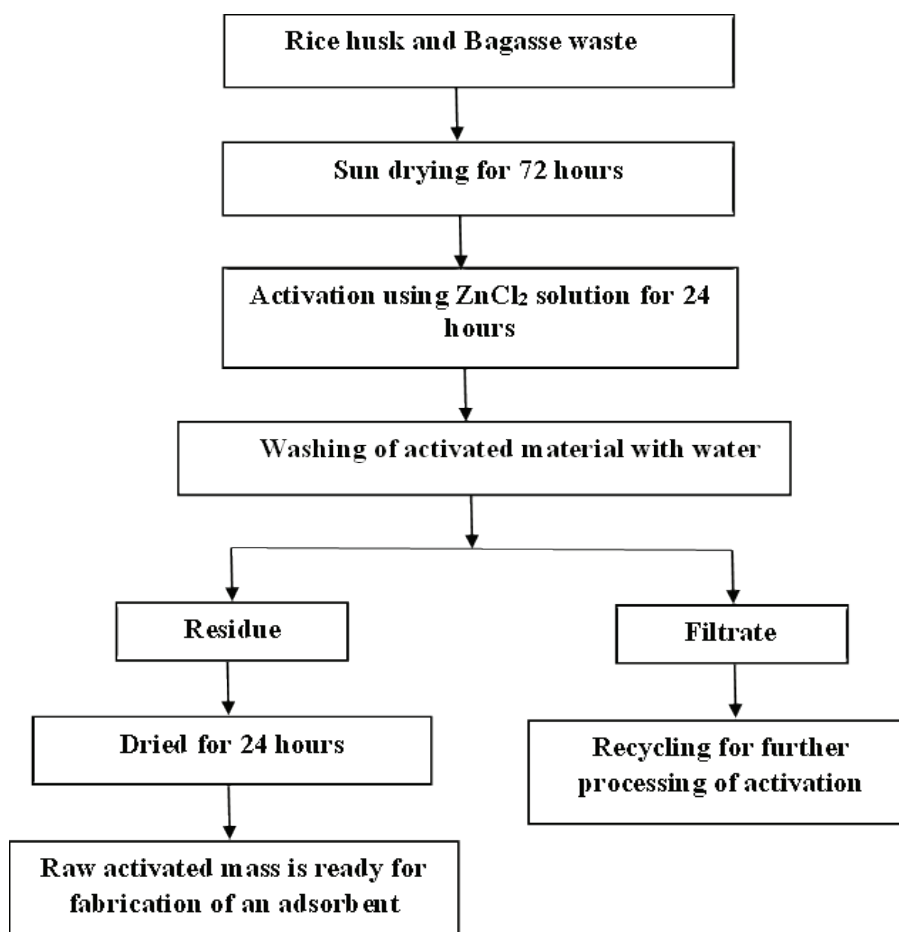
**Fig. 7.** Process flow sheet for nitrate adsorption

Table 5. Composite Matrix: Physical Properties

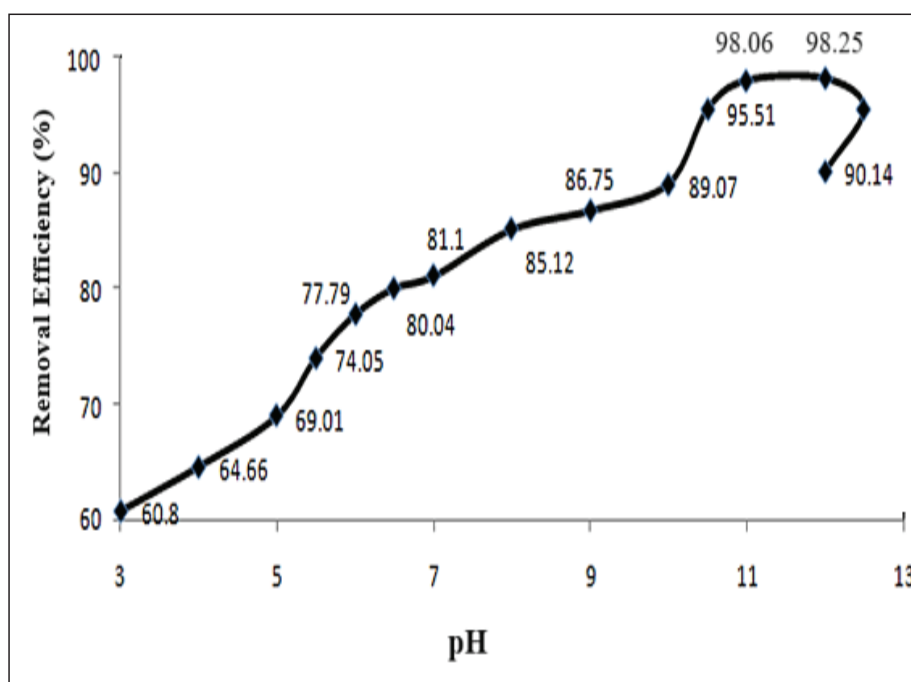
Sl. No.	Density (Kg m ⁻³)	Compressive Strength (MPa)	Tensile Strength (MPa)
CM - 1	307.22	0.29	0.884
CM - 2	259.14	0.21	0.901
CM - 3	291.45	0.37	0.893
CM - 4	314.08	0.34	1.207
CM - 5	284.31	0.39	1.021
CM - 6	333.17	0.37	1.078
CM - 7	320.11	0.34	0.889
CM - 8	301.43	0.41	0.997
CM - 9	279.88	0.44	1.003
CM-10	342.12	0.38	1.102

3.1. Role of pH on Nitrate Removal

It is observed that the removal of nitrate under alkaline conditions is more favorable and is increasing to 11-12 pH and after that removal is slowed down, as presented in Table 6 and Figure 8. An acidic medium was found very weak for efficient nitrate removal from the water sample.

In acidic conditions, with increase in pH, there is a gradual increase in nitrate removal from

contaminated water bodies. Similarly, when the pH is maintained in an acidic range, the recovery slowly increases from 65% to 80% with a pH variation of 4 to 6.5. This data revealed that acidic pH does not impact much on nitrate removal shown in Figure 9a. Nitrate removal is partially pH-dependent. When the composite matrix is added to the water sources, due to the alkalinity of the material there is a gradual increase in pH of the medium.

**Fig. 8.** Impact of acidic and alkaline pH on nitrate removal

The result obtained showed that at a pH range of 8 - 12, there is a removal of 85% (or) above the amount of nitrate. The optimum pH is found to be 11, where a maximum of 98.0% of nitrate could be removed from the water source. Again, with the further attempt of increase in pH, there is gradual decrease in removal of nitrate. The decrease in removal of nitrate with the increase in pH may be attributed to the fact that, the presence of Iron in the form of ferric Fe^{3+} state in the Baggase waste at a suitable pH \approx of 12.5 may lead to formations of ferric nitrate, which might get coagulated in the water medium leading decrease in nitrate removal. For maximum removal of nitrate in the contaminated water was obtained at pH 11.

3.2. Role of Composite Matrix doses on nitrate Removal

The impact of Nitrate removal from contaminated water samples with variable doses of composite matrix is studied. Due to Figure 9b, It has been revealed that the removal efficiency of nitrate is directly affected by the dose of the composite matrix. Out of all experimented doses, 70 mgL^{-1} addition of composite matrix dose provided the best efficiency for nitrate removal. The gradual rise in the removal of nitrate concerning the rise in the concentration of doses of the composite matrix can be attributed to the fact that with the addition of more quantity of activated composite matrix, there may be the availability of a more porous surface on the composite matrix, which would have helped the colloidal suspension to penetrate.

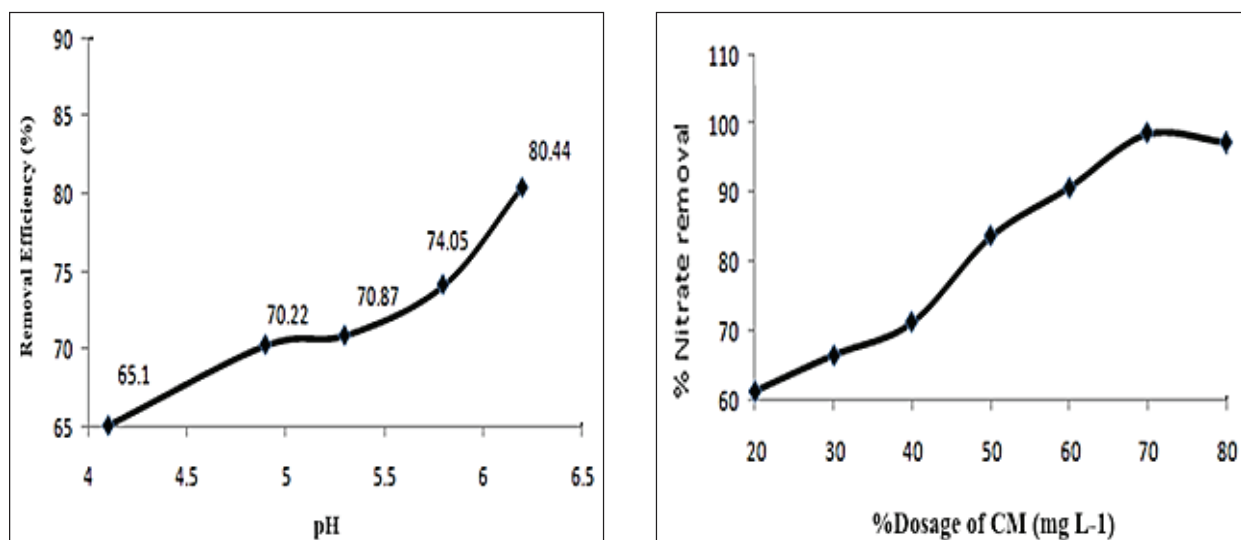


Fig. 9. a) Impact of acidic pH on nitrate removal (left)
b) Role of Composite Matrix doses on nitrate adsorption (right)

Table 6. Nitrate removal depends on doses of composite matrix

Sl. No.	Residual nitrate (mgL^{-1})	Dosages of CM-9, (mgL^{-1})	Nitrate Absorbed
1	38.8	20	61.2
2	12.6	30	66.4
3	8.4	40	71.1
4	2.1	50	83.5
5	4.8	60	90.7
6	8.9	70	98.4
7	10.6	80	91.4

Conditions: pH = 11.2, Nitrate Content = 100 mg L^{-1} , Room Temperature (30°C)

The percentage of removal of Nitrate is attempted with a most suitable adsorbent matrix and at standard pH 11.2, is studied, as shown in Table 6. It shows that with a sufficient dose concentration of composite matrix, up to 98% Nitrate removal is possible from the contaminated water sample.

3.3. Impact of COD of water sample on nitrate removal

The COD level in water samples usually restricts the determination of the organic matter present in any water medium. The impact of different COD levels of the contaminated water sample is studied for removal efficiency of nitrate which is shown in Table 7.

Due to Figure 10, nitrate could be removed from a range of 250 mgL⁻¹, up to 94%. Beyond 250 mgL⁻¹, the efficiency of removal is decreased. As in the sample water the dissolved oxygen level is much lower than the prescribed limit by WHO, the optimum COD should be limited to 250 mgL⁻¹ for effective removal of Nitrate. The rise in COD influences directly more removal of nitrate from sample water. It may be because of the influence of COD on the pH level of water and the positive influence of the rise in nitrate removal percentage. But beyond the range, the adsorption decreases up to 18% at 550 mgL⁻¹. So, the research suggests adequate measures to be adopted to maintain the COD level for efficient nitrate removal.

Table 7. Nitrate Removal efficiency against variation of COD level

Sl. No.	COD (mgL ⁻¹)	% Residue (N)	% Removal
1	500	81	19
2	460	65	35
3	380	43	57
4	320	20	80
5	250	06	94
6	180	14	86
7	110	28	72
8	80	39	61
9	50	57	43
10	30	82	18

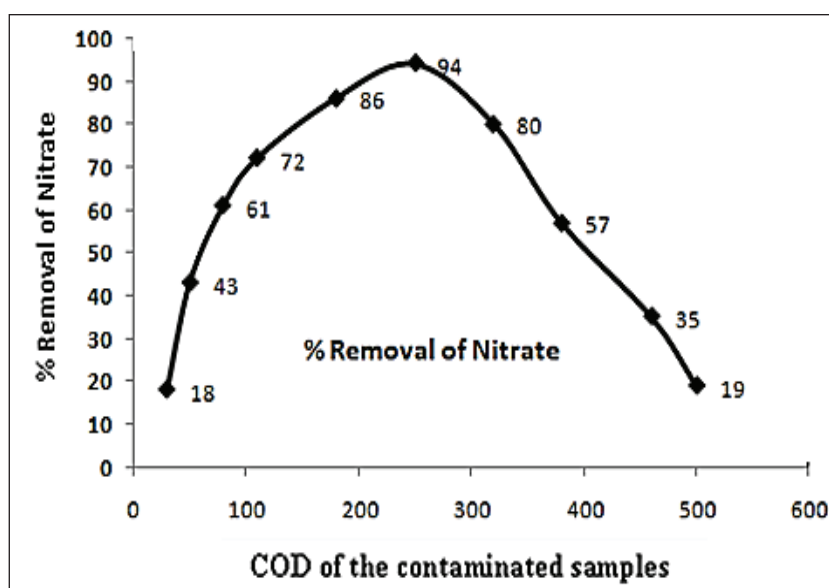


Fig. 10. Impact of COD of the sample on Nitrate adsorption

3.4. Scanning Electron Microscopic Studies on the Composite Matrix:

Scanning electronic microscopic studies are conducted to investigate the effect of pH conditions on nitrate removal. In this context, the SEM of a composite matrix was considered and the texture is found to be quite uniform throughout. The composite matrix exhibits a consistent blend of bagasse waste and rice husk dust particles, creating a porous structure for nitrate absorption, as depicted in Figure 11a. To study the effect of various pH-containing wastewater on the textural changes of the matrix, acidic, neutral, and alkaline mediums of wastewater are added to

the composite medium to study the absorption rate and textural variations. When an alkaline medium passes through the composite material, it is observed that there is maximum textural variation attributed to alkaline water sources. After passing the alkaline water, there is a complete variation in the material texture. Figure 11b represents the SEM for the treated material, which is approximately 75% distorted. When an acidic medium passes through the composite material, there is minimal textural variation caused by acidic water sources. After passing the acidic water, there is minimal variation in the material texture.

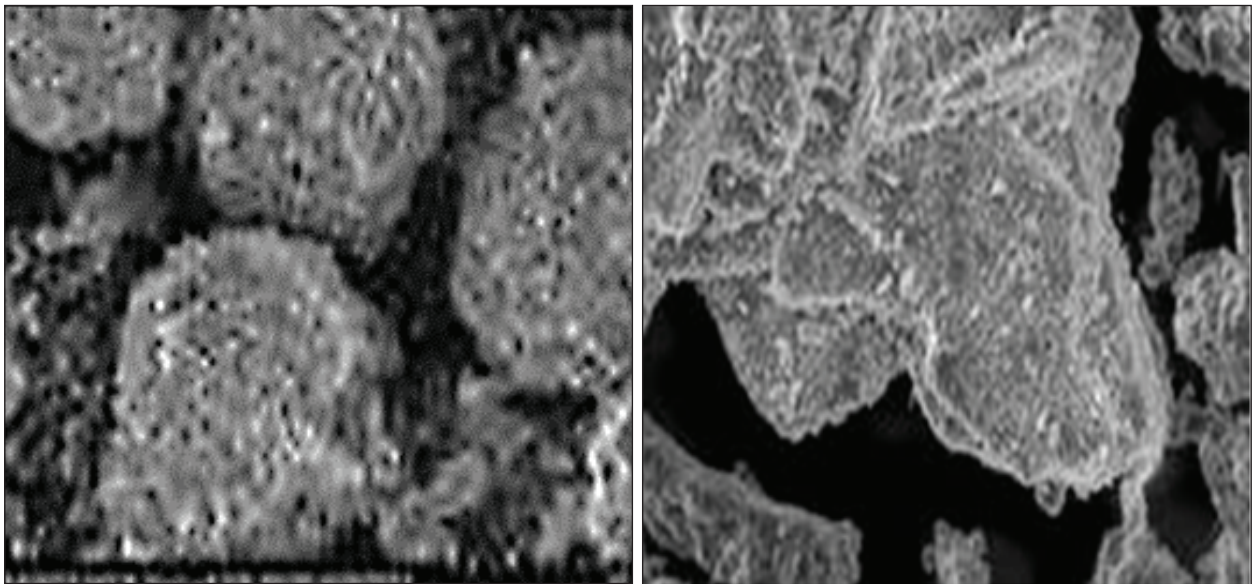


Fig. 11. a) SEM of the composite before treatment b) treatment of an alkaline wastewater

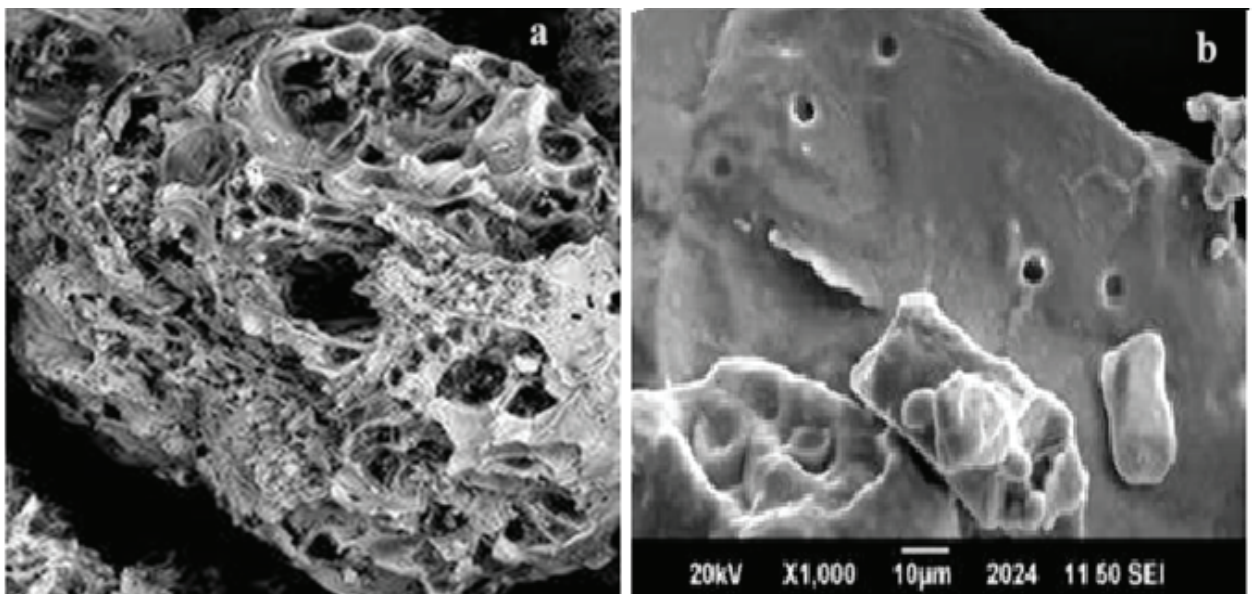


Fig. 12. a) SEM of the composite matrix after treatment with acidic wastewater b) after treatment of neutral

Figure 12a represents the SEM for the treated material, which is ~ 15% disturbed. When a neutral medium is passed through the composite material, it has been observed that there is variation in texture attributed to natural water sources. After passing the neutral water, there is a significant variation in the texture of the material. Figure 12b represents the SEM for the treated material, which is approximately 80% distorted. Thus, it is concluded that a neutral medium with an alkaline pH of 11 is the best for maintaining 96% nitrate removal from contaminated water sources.

4. Conclusion

The study on de-nitrification conditions for efficient removal of nitrate concluded that the removal is majorly dependent on the pH of the contaminated water, COD of the wastewater, and doses of composite matrices. Keeping a pH of 11, COD maintained with 250 mgL⁻¹, and a fixed dose of the composite matrix, about 96% of nitrate could be removed. The bio-based composite matrix is highly efficient for removing nitrates from contaminated water.

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