



## Continuous Adsorption of As(III) from Wastewater by Red Mud in a Fluidized bed

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(Accredited by NBA, NAAC with 'A' Grade & ISO 9001:2015 Certified Institution)

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### CERTIFICATE

This is to certify that the report entitled [Continuous Adsorption of As\(III\) from Wastewater by Red Mud in a Fluidized bed](#) submitted by Mr. K. Uday Teja, Mr. K. Hari Krishna, Mr. M. Nikhil Nirmal Kumar, Mr. S. Srikanth, Mr. S. Surya Narayana Sanjay, bearing Regd. No. 20341A0819, 20341A0821, 20341A0824, 20341A0828, 20341A0829 have been carried out in partial fulfillment of the requirement for the award of degree of Bachelor of Technology in Chemical Engineering of JNTUGV, Vizianagram is a record of Bonafide work carried out by them under my guidance & supervision of Dr. Ch. Ramya. The results embodied in this report have not been submitted to any other University or Institute.

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#### ABSTRACT :

Water pollution is growing rapidly due to the presence of heavy metals. One of them is As(III) so it should be removed from the water. Various batch adsorption of As(III) was conducted by using various activated carbons, biochar's, and biosorbents, thus limiting its scale-up. The disadvantages include that it takes a long time to removal of metals. The Continues adsorption can be achieved by a Packed bed as well as a fluidized bed. As compared to the packed bed, the fluidized bed will get more efficient because the adsorbent is fluidized so that it will be exposed to wastewater more in the fluidized bed. Adsorption time will decrease due to an increase in adsorption capacity. And there are practically no temperature gradients in bed even with quite exothermic or endothermic reactions.

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Keywords: Wastewater, Adsorption, Fluidized bed, Adsorbent, Red Mud

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## CHAPTER-1

### INTRODUCTION :

#### Introduction

Wastewater runoff into the oceans and seas is causing water contamination to increase quickly. The sources of this wastewater include mines and quarries. Fig. 1 below illustrates the origins of wastewater.

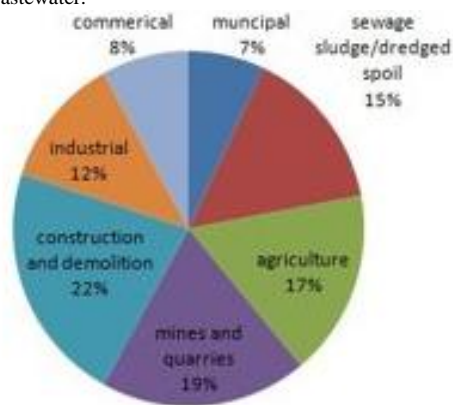


Fig. 1. Source of Wastewater

From the industries, 12% of wastewater is discharged. This wastewater consists of heavy metals like Zn, Cu, Pb, Ni, Cd, Hg, etc. Depending on their toxicity, substances can cause a variety of environmental issues. But among all these metals Arsenic is one of the metals which is more toxic and dangerous.[1]

Arsenic is chemical element. the symbol of the element is As and the atomic number is 33. It is a metalloid; it possesses both metallic and nonmetallic characteristics. The earth's crust frequently contains arsenic, which is also widely present in the environment. Arsenic is extremely harmful to both humans and animals and can lead to a number of health issues, such as cardiovascular disease, malignancies, and skin blemishes. Arsenic exposure can happen through contact with polluted soil or industrial waste, as well as through contaminated drinking water, food, or air. Over history, arsenic has

been utilized for a number of things, such as pesticides, wood preservatives, and medications. Arsenic can also be found in tiny quantities in several foods, including seafood, poultry, and rice. Classifications are is discussed in the following 1.1.[1]

### 1.1. Classifications of Arsenic

One of the harmful metals found in wastewater is arsenic. On a periodic chart, arsenic belongs to group V and period 4, and it has five free electrons in its outer shell. Its atomic structure permits the existence of arsenic in a variety of compounds with variable amounts of oxidations. Arsenic exists in four stages of oxidation, including arsenate ( $\text{As}^{+5}$ ), arsenite ( $\text{As}^{+3}$ ), arsenic ( $\text{As}^0$ ), and arsine ( $\text{As}^{-3}$ ), according to Jiang et al. (2012) and Nicomel et al. (2016). Both organic and inorganic forms of arsenic can be found in aquatic environments, while the concentration of organic forms is negligible, has fewer negative effects than inorganic forms, and does not affect the quality of drinking water. inorganic form with valence values between +3 and +5. Arsenate, or  $\text{As}^{+5}$ , is less toxic than  $\text{As}^{+3}$ , or arsenite.[1]

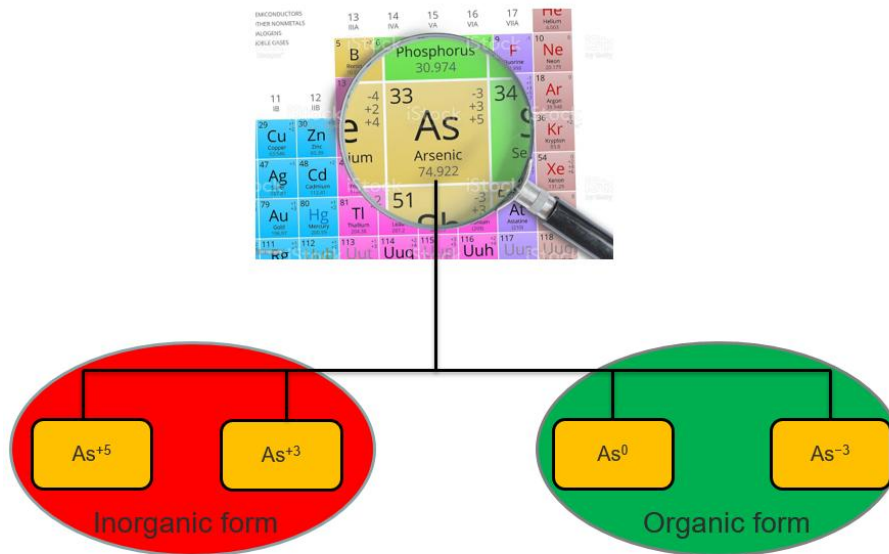


Fig. 2. Classification of arsenic

Not only the organic and inorganic it is classified into other forms like Elemental arsenic, Arsenic Salts and Arsenic Oxides but we are mainly concentrating on inorganic form only.

### 1.2. Source of Arsenic

It originates from both geogenic and anthropogenic causes. Arsenic is categorized as a group A human carcinogen by the US Environmental Protection Agency (EPA), meaning it has the ability to seriously harm human health. An adjective used to characterize human-caused alterations in the environment is anthropogenic. Geogenic groundwater refers to groundwater that has unhealthy naturally occurring high concentrations of specific components.[2]

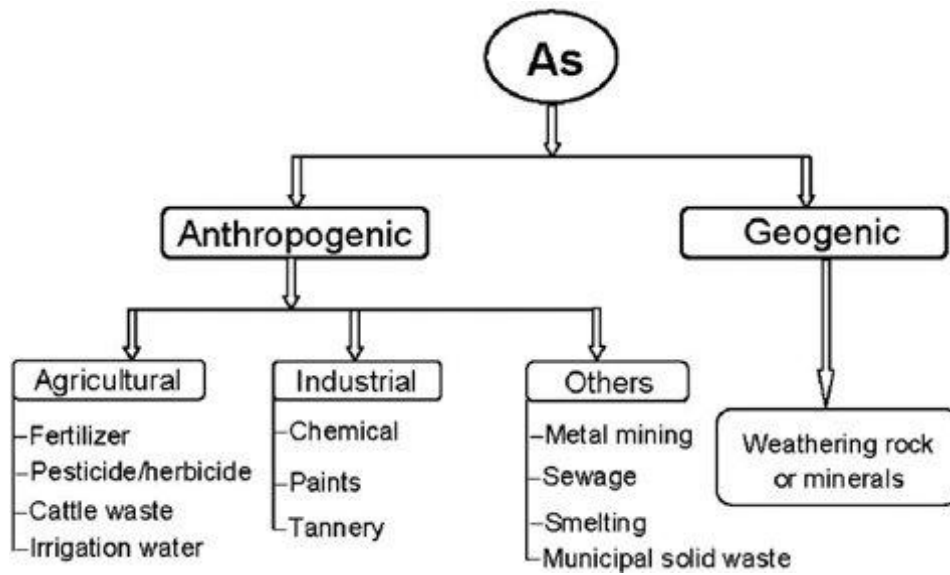


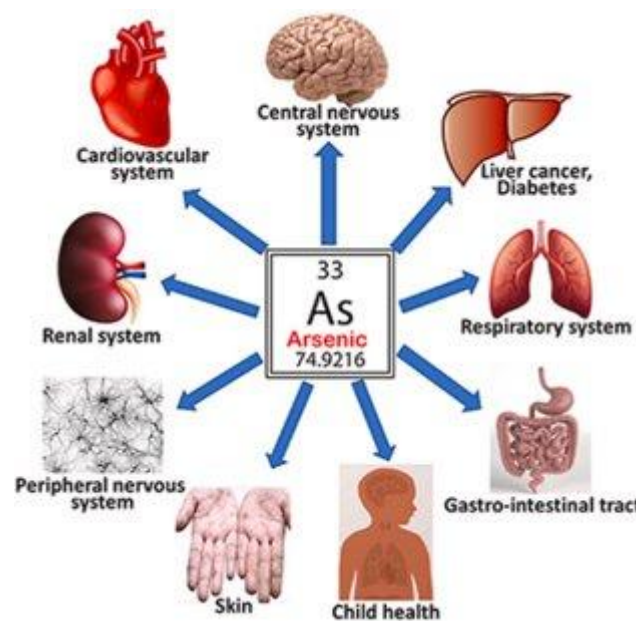
Fig. 3. Source of arsenic

Source: <https://www.researchgate.net/figure/>

In the Anthropogenic there are three different sources Agricultural, Industrial, and others. Agricultural is not much toxic then the industrial. In the industrial there are three types chemical industries, paint industries, tannery industries and others like metal mining, sewage, smelting, municipal solid waste are the main sources of the arsenic.

### 1.3. Effects caused by Arsenic

Fig 4 illustrates the serious health impacts that arsenic can have, including many different types of cardiovascular, neurological, hematological, renal, and respiratory conditions as well as malignancies of the skin, lung, bladder, liver, and kidney. About 200 million people around the world are at risk of high levels of arsenic. As a result, the WHO recommends a 10 g/L limit for arsenic in drinking water[3].



**Fig. 4. diseases caused by drinking wastewater**Source: <https://www.sciencedirect.com/>

Arsenic is a known element it causes environmental pollutant that occurs naturally in aquifers through geological processes. Most people who may be exposed to the heigh levels of arsenic come from Asia and America, and exceeds 200 million people worldwide. Arsenic exposure is linked to a number of harmful health impacts, including neuropathy, carcinogenesis, and skin disorders (such as arsenic, hyperkeratosis, pigmentation changes, etc.), carcinogenesis, and neuropathy. Arsenic has also recently been associated with the development of non-communicable diseases like diabetes and cardiovascular disease and more. The processes behind the arsenic induced diabetes include decreased cellular glucose transport, insulin resistance, impaired insulin production, and pancreatic beta-cell dysfunction and death[3].

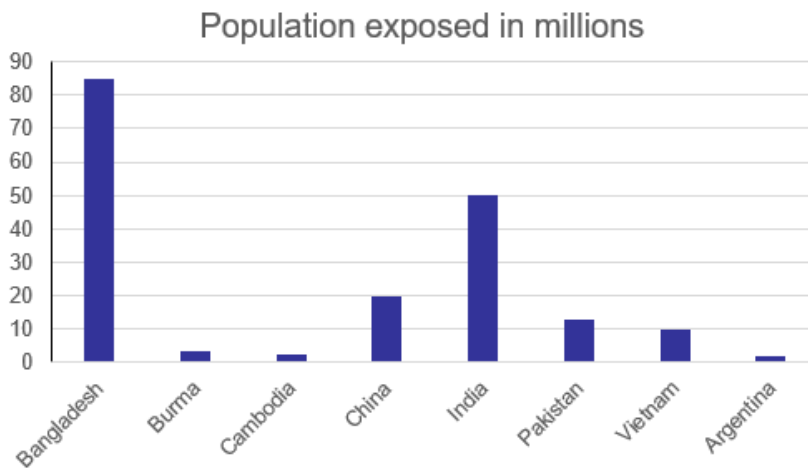
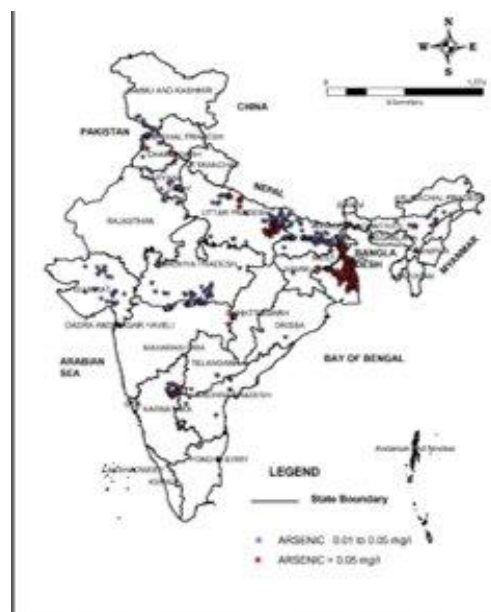
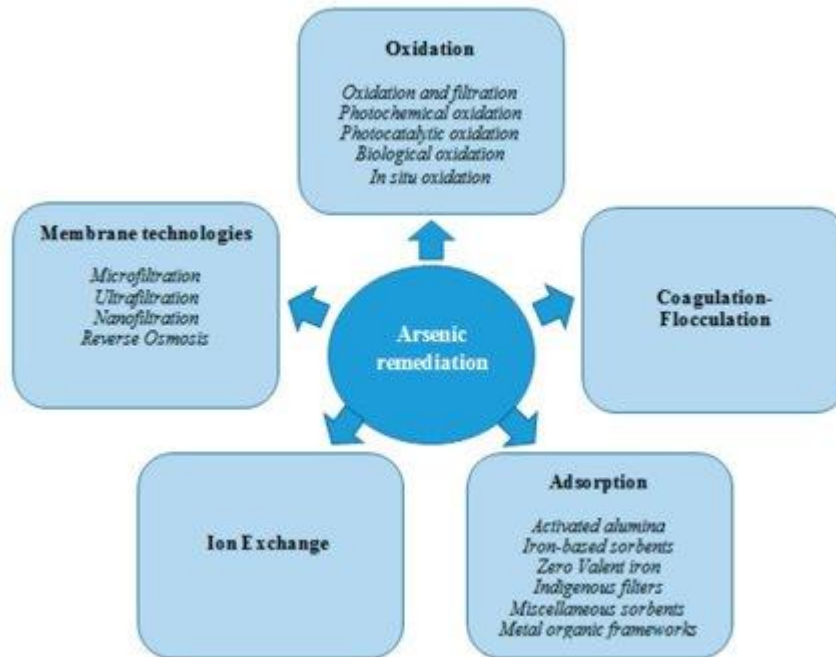


Fig. 5. Affected people throughout the world in country-wise.

**Fig. 6. Location of Arsenic in Indi**Source: <http://jalshakti-dowr.gov.in/>

#### 1.4. Methods Of Arsenic Removal

They are five methods to remove the arsenic which are Shown in the below Fig 7. Let us go through the one by one.



**Fig. 7. Methods to remove arsenic**

Source: <https://www.ncbi.nlm.nih.gov/pmc/>

##### 1.4.1. Oxidation Techniques

Soluble As(III) is converted to As(v) during oxidation processes. This method alone cannot remove arsenic from solutions. Therefore, alternative removal methods such as adsorption, coagulation, or ion exchange should be used. Oxidation is a key step in the oxidation of groundwater, as arsenite is the predominant form of arsenic at near-neutral pH levels. In addition to ambient oxygen, other chemicals and microbes have also been employed that directly oxidize arsenite in water. In developing nations, the most popular oxidants are atmospheric oxygen, hypochlorite, and permanganate. Soluble As(III) is converted to As(v) during oxidation processes. This method alone cannot remove arsenic from solutions; as a result Therefore, alternative removal methods like adsorption, coagulation, and ion exchange can be used. Oxidation is a key step in the oxidation of groundwater, as arsenite is the predominant form of arsenic at near-neutral pH levels. In addition to ambient oxygen, other chemicals and microbes have also been employed to directly oxidize As(III) in water. In developing nations, the most popular oxidants are atmospheric oxygen, hypochlorite, and permanganate. Arsenite can take hours or weeks to oxidize with oxygen, which is an extremely slow process[4]. Moreover, this requires a complicated treatment that results in an As-containing residue that is tough to discard. Consequently, it is important to choose oxidants carefully in order to effectively remove arsenic from a solution through oxidation. However, all of the cited drawbacks of oxidation alone render it a less effective approach for removing arsenic[4].

##### 1.4.2. Coagulation Flocculation

One of the most common and well-researched methods for removing arsenic from water is flocculation. During coagulation, a positively charged coagulant such as ferric chloride ( $\text{FeCl}_3$ ) or aluminum sulfate ( $\text{Al}_2(\text{SO}_4)_3$ ) reduces the negative charge of the colloid, causing the particles to collide and grow. Flocculation, on the other hand, involves the addition of anionic flocculants. This either neutralizes the charge or cross-links the larger particles produced to form flocs. The chemicals in these steps transform the dissolved arsenic into an insoluble solid that later facilitates precipitation. Alternatively, when introduced into the metal hydroxide phase, coprecipitation of soluble arsenic species is possible. In any case, the precipitate can then be removed by filtration or sedimentation [4].

The effectiveness of various coagulants to remove arsenic varies with pH. . Both  $\text{Al}_2(\text{SO}_4)_3$  and  $\text{FeCl}_3$  are suitable for removing arsenic from water

below pH 7.6. Most studies indicate that  $\text{FeCl}_3$  is a better coagulant than  $\text{Al}_2(\text{SO}_4)_3$  at pH values above 7.6 and that arsenate is more efficiently removed than arsenite between the two inorganic arsenic species. At an initial concentration of 280 g/L, an aluminum-based coagulant reduced the arsenic concentration below the maximum concentration (MCL) of 10 g/L, despite being inferior to the reported ferric chloride [4].

### 1.4.3. Membrane Technologies

Membrane filtration is a process that can be used to remove contaminants such as arsenic from water as part of drinking water production. Membranes are often made of synthetic materials with numerous pores that act as selective barriers, preventing the passage of some water components necessary propulsion and Pressure difference between feed and permeate sides. Pressure-driven membrane filtration can be divided into two general categories. In Low pressure membrane processes such as microfiltration and ultrafiltration. and high-pressure membrane processes such as reverse nanofiltration and osmosis [4].

### 1.4.4. Adsorption and Ion Exchange

Solids are used as media when removing chemicals from gas or liquid solutions. Basically, material is removed from one phase and deposited on the surface of another phase. Vanderwaals forces and electrostatic interactions between adsorbent molecules and surface atoms of the adsorbent are the key forces driving this process. Therefore, it is important to define the surface properties of the adsorbents before using them for adsorption. Surface and Polarity are the wide range of sorbents is already being investigated in several research areas. These materials include ferric hydroxide, zero valent iron, chitosan, coal, red mud, fly ash, chicken feathers, kaolinite, montmorillonite, goethite, zeolites, activated alumina, and activated carbon. The table shows that a new method to treat arsenic-contaminated water is iron-based adsorption. This can be explained by the strong attraction of inorganic arsenic species to iron. Iron can remove arsenic from water by acting as a reducing agent, coprecipitant, adsorbent, or contaminant fixative. Adsorption is said to be the most popular method for removing arsenic because of its many advantages, including relatively high arsenic removal efficiency. Ease of use, economy, no sludge [4].

## CHAPTER-2

### REVIEW OF LITERATURE :

#### 2.0. By using adsorption

Hashim et al. [5] used the High gravimetric technology, also known as HIGEE, it is a cutting-edge method that makes use of the rotating packed bed (RPB) contactor and centrifugal force. In RPB, a doughnut-shaped bed rotates to allow liquid to move rapidly through the adsorbent bed. With higher mass transfer coefficient, centrifugal force improves the adsorption process. They studied the on the based of Taguchi method was used to analyse and optimise the ideal operating conditions of an rotating packed bed (RPB) contactor for the removal of arsenic using activated carbon. They determined the initial ideal condition in an L16 orthogonal array, five parameters rotating speed, feed rate, packing density, initial concentration, and initial solution pH were taken into account. The agreement was very good and the actual removal of arsenic resulted in a predicted removal rate of 96.68% [5].

Jacukowicz et al. [6] study was carried out using a hybrid anion exchanger in which Cu(II)-Fe(III) binary oxides were deposited on the same Cu porous structure. Fe ratio 1:2, however, due to the different amount and distribution of inorganic sediments within the polymer beads, his work consisted in investigating the oxidation and adsorption of his As(III) on the surface. The best hybrid polymer's high adsorption capacity is 94.4 mg/g was validated by the equilibrium studies. He studied his As(III) oxidation and adsorption on the surface of hybrid anion exchangers. Hybrid anion exchangers differ in the amount and distribution of inorganic deposits within the polymer beads, but the same 1:2 Cu:Fe and Binary Fe oxides are deposited on its porous structure. They verified that the optimum hybrid polymer will have a high adsorption capacity: 94.4 mg/g. He got adsorbent, because of its chemical and physical makeup, demonstrated a very high maximum adsorption capacity is 94.4 mg /g. [6].

Mandal et al. [7] is conducted the experiment using a novel hybrid material. That is named as zirconium polyacrylamide (ZrPACM-43). That has been created by using an environmentally friendly sol-gel technique with aqueous solution, are zirconium oxychloride and combination of acrylamide. He used FTIR, XRD, TGA-DTA and SEM-EDS to describe the material. By adjusting solution parameters like adsorbent dose, adsorbate concentration, solution pH, and contact time. the material's capacity to remove arsenic to a certain level was examined. Arsenic(III) was removed under ideal conditions with a maximum removal efficiency of 98.22% and an adsorption equilibrium time of 120 min. At pH 10, the regeneration rates of adsorbed arsenic (III) in aqueous solution for starting concentrations of 10 mg L<sup>-1</sup>, 50 mg L<sup>-1</sup>, and 100 mg L<sup>-1</sup> were 99.78%, 94.51%, and 90.12%, respectively [7].

#### 2.1. By using biosorbent

Boddu et al. [8] has done work using biosorbents. Biosorbent were prepared by coating ceramic alumina with natural biopolymers, as studied by adsorption to biosorbents at pH 4.0 under equilibrium and dynamic conditions. Langmuir, Freundlich, and Redlich-Peterson adsorption models were fitted to equilibrium adsorption data and model parameters were evaluated. According to the Langmuir isotherms, the monolayer adsorption capacities of As(III) and As(V) adsorbents are 56.50 mg/g chitosan and 96.46 mg/g chitosan, respectively. Dip coating method, using Itosan. Removal of arsenic



(III) (As(III)) and arsenic (V) (As(V)). The monolayer adsorption capacities of As(III) and As(V) adsorbents were found to be 56.5 and 96.5 mg/g, respectively [8].

## 2.2. By using Bacterial and fungal strains

Jaiswal et al.[9] is acknowledged. The immobilized biomass of new natural strains FNBR 3, FNBR 6, FNBR 13, and FNBR 19 were tested to remove arsenic (As) from aqueous solutions. In a batch experiment, alginate beads with 0.1g biomass were employed. As adsorption (mg g<sup>-1</sup>) of 70, 68, 113 and 90 by FNBR 3, FNBR 6, FNBR 13 and FNBR 19 respectively reached biosorption equilibrium in the first two hours. The adsorption of As by biomass found to be 59.5 (FNBR 3 and FNBR 6), 74.8 (FNBR 13) and 66.3 (FNBR 19) when measured on the column and validated with the Thomas model. The highest arsenic uptake was observed with p. The immobilized biomass of these unique fungal strains appears to be most effective for industrial use in removing arsenic from contaminated water [9].

Dolphen et al.[10] has done the experiment on Arsenic contamination in rice plants lead to several physiological, and five productivities in the rice grains, according to an experiment. The health of people suffered as a result. A combination of leonhardtite has an amendment and the arsenic-resistant endophytic bacteria 7 has been researched to reduce amount of arsenic in 6 grains. 1% (w/v) leonhardtite (91.862.04%) exhibited a better efficiency than a bagasse fly ash of 16.253.97%, rice husk ash has 10.361.28%, and sawdust fly ash have 63.005.67% under, same conditions for adsorbing an initial taken concentration of 2 mg L<sup>-1</sup> of arsenic. Then results they got is that leonhardtite could reduce the amount of arsenic in rice grains. This corresponds to a decrease from 4.56 mg As kg<sup>-1</sup> to 3.12 mg As kg<sup>-1</sup> (31.58%) [10].

Bagasse(BFA), a byproduct of the sugar industry, has employed by Ali et al. [11] as an inexpensive adsorbent. To removal of arsenate and arsenite species from the waste water. The following circumstances are ideal for eliminating both kinds of arsenic: Arsenate and arsenite were removed with 95.0 and 89.5%, respectively, at pH 7.0, concentration is 50.0 g/L, contact time of 50.0 min, then adsorbent dosage is taken as 3.0 g/L, and temperature 20.0 °C. . The technology developed to remove arsenate and arsenite species is fast, affordable and can be used in the natural environment. They were applied to clean up any contaminated water sources that included arsenic species. They were applied to purify contaminated water sources containing arsenic seeds. 98.9% and 95.6% removal of both types of arsenic was achieved using column operation at a flow rate of 1.0 ml/min with 95.0% and 89.5% arsenate and arsenite removal in batch mode.

According to De et al. [12] Arsenic contamination of groundwater is a significant risk in developing countries. In this context, the use of Karanja or Pongamia pinnata seed cake, a biodiesel residue, has been proposed as a means of reducing arsenic hazards using agricultural waste. The biosorption of As(III) to heat-activated deoiled Karanja germ cake (TAKB) with a surface area of 19.8 m<sup>2</sup> g is investigated in a countercurrent multiphase fluid bed column. Analysis of variance and multiple regression analysis were used to relate the removal rate of As(III) to independent process parameters and derive a second-order polynomial. Karanja seed cake deoiled in a countercurrent fluidized bed was used in the experiments to remove up to 94.12% As(III).

An active MnO<sub>2</sub>/rice biochar composite (MBC) has been created by Cuong et al. [13] to improve the removal of As(III) for groundwater treatment. With the addition of MnO<sub>2</sub>, the porous structure of the MBC material was, resulting in a large number of sites for the surface- / interface-related activities such redox transformation and arsenic adsorption. As a result, the use of MBC can significantly improve arsenic removal. More particular, MBC had a high As(III) removal capability that was 10 times more than BC's. Using simulated groundwater and an arsenic content as low as the 10 g L<sup>-1</sup> WHO recommendation, MBC demonstrated an efficient arsenic removal efficiency of 94.6%[13].

From the literature review, it was observed the different adsorption method adopted for As(III) removal are batch process, so could not be scaled up to the industrial level. So design of a continuous process for adsorption of As(III) and its scale up have attracted many researchers. For the conversion of such batch process to a continuous one, packed column can be alternative. However, the rate of adsorption in packed column is slow. Thus the packed column can be converted into a fluidized column to increase the rate of adsorption and increase the rate of treated water. Moreover, in fluidized bed, there is no temperature gradient in the bed even with quite exothermic reaction.

Further, choice of sorbent plays a vital role in the adsorption process. From literature review, it was observed that different sorbents viz. biosorbent, Bacterial and fungal strains, activated carbon were used. However, the different activated carbon derived from plants, debris etc. requires huge plantation area, thus becomes expensive. Moreover, the process of conversion of organic matter into carbon and carbon to activated carbon is energy intensive. Simultaneously, the biosorbents exhibit better sorption capacity, however, the slow rate adsorption hinders its commercialization. Thus, in this current work, red mud, an industrial waste was chosen as an alternative sorbent. Further, the red mud was converted into red mud pallets to become compatible in fluidized bed.

Red mud is by product of Bayer process, which is the main technique for processing bauxite to produce alumina. Whereas the amounts from different plants are substantially more varied, the particular generation of RM per ton of alumina from the plants (more than 95%) that using Bayer process is between 1 to 1.5 ton globally. An estimated 140 million tons of RM are generated each year. The Fig 9 shows the global (country-by-country) creation of Red Mud for the year 2019 (India data from April 2018 to March 2019).

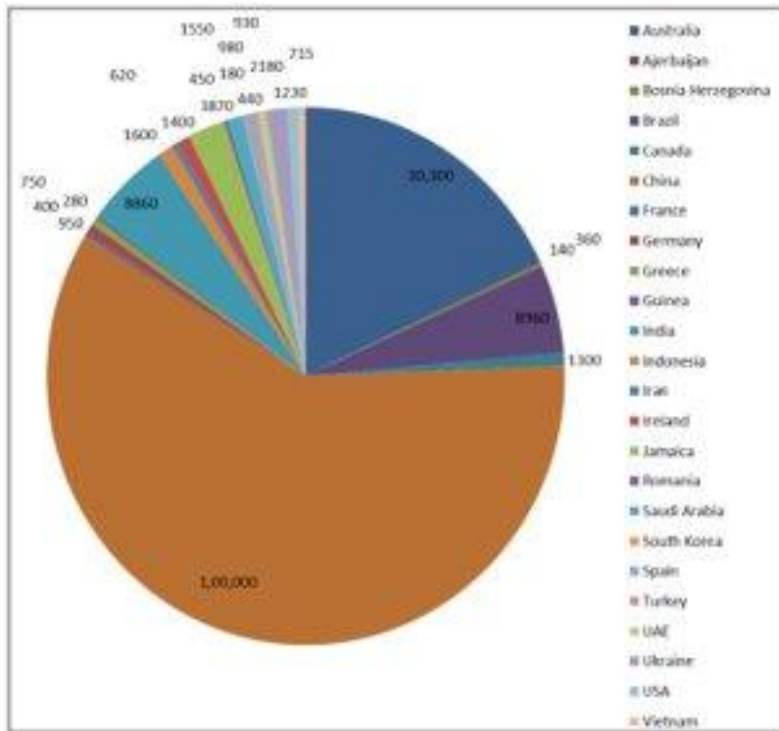


Fig. 08. Global generation of Red Mud

Source: <https://www.google.com/url>

There are seven units in India that produce red mud, and Fig.10 shows how much red mud is produced annually by each industry.

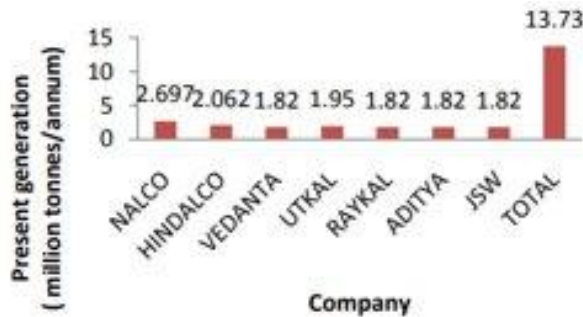


Fig. 09. Red mud generation in India

Source: <https://doi.org/10.1016/j.minpro.2012.11.001>

This quantity of RM is produced during the refining of alumina. Again, if this is dumped, it is pollution. If we dig in the sand, groundwater will get contaminated or it will be discharged into open water, such as rivers, which is particularly damaging to aquatic life. Thus, it is risky to dump the RM. Using the RM for different procedures like metal recovery is an alternative[14].



**Fig. 10. Utilization of Red Mud**  
Source: <https://www.google.com/url>

There are many Utilizations of red mud and it also used for the adsorption. So, we have selected the adsorbent is Red Mud.

### CHAPTER-3

## MATERIALS AND METHODS :

### 3.0. Materials

The red mud was collected from the Utkal Alumina Int Ltd which is in Rayagada, Orissa (see Appendix A). For the preparation of synthetic As(III) solution, As<sub>2</sub>O<sub>3</sub> was purchased from Herenba Instruments & Engineers. Sodium alginate, calcium chloride was purchased from Herenba Instruments & Engineers. Distilled water was prepared at our laboratory using double distillation unit. All the chemicals purchased are used without any further purification.

### 3.1 Experimental methods

#### 3.1.1 Preparation of red mud pallets

The red mud pellets are prepared by mixing 2.0 g of sodium alginate with distilled water(100 ml), then pouring that mixture into a 250 mL beaker and heating it to 353 K while stirring continuously as shown in Fig 11.



**Fig. 11(a). Sodium alginate in water**

Until a gel-like substance forms, this must be continued at 1000 rpm. The mixture is then added 2.0 g of RM while being constantly stirred to create a uniform mixture, and then cooled to 303 k as shown in Fig 11(b).



**Fig. 11(b). Red mud with 1000 rpm**

A 2.0% (w/w) calcium chloride solution maintained at 271 K receives the homogenous mixture as a result, which is then added as droplets. The solution is taken in burette and slowly drop by drop poured in the calcium chloride solution to form the beads of red mud pellets as shown in fig 11(c). The beads are shown in the Fig 11(d) and then filtered with distilled water to remove any excess calcium chloride, and allowed to air dry for 24 hours at room temperature pellets are shown in Fig 11(e). Thus, the prepared Red Mud Calcium Alginate Beads (ARMC) and used for the removal of As(III).

**Fig. 11(c). Formation of pellets****Fig. 11(b). pellets before drying**

Thus, the prepared Red Mud Calcium Alginate Beads (ARMC) and used for the removal of As(III).

**Fig. 11(e). pellets after drying**

### **3.1.2 preparation of As(III) solution**

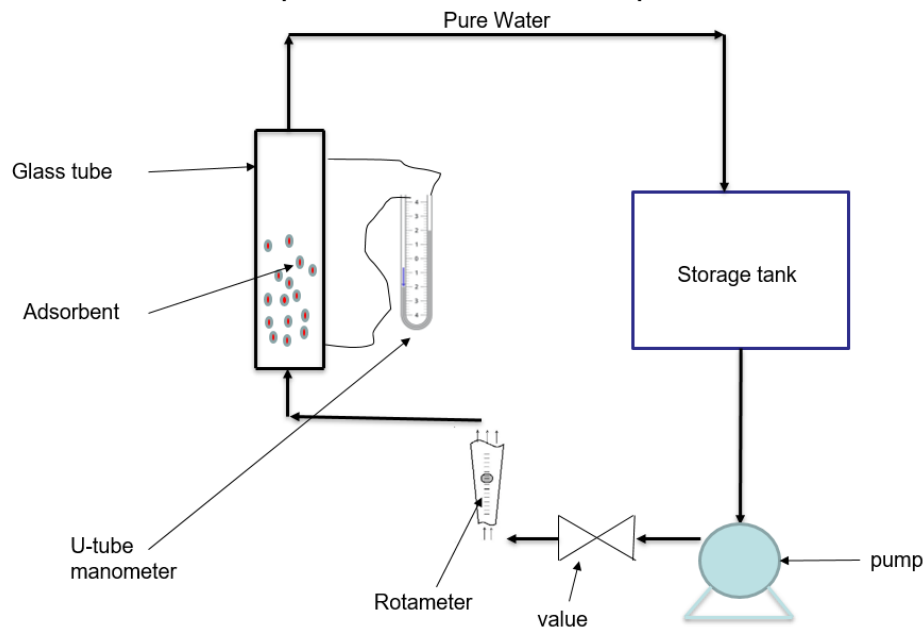
The  $As_2O_3$  is purchased from the Herenba Instruments & Engineers.

### **3.1.3 Experimental procedure**

A two-phase fluidized bed is taken with the 6cm height of the bed and the fluidized pallets are prepared by the red mud. Now fill the storage tank with the know concentrations of arsenic solution and start the setup with a certain flow rate of the solution. Note the pressure difference and pH of the solution. After desired time take the sample of the solution and find the finial concentration by using UV method.

### **3.1.4. Fabrication of a two-phase Fluidizes adsorption column**

We have taken the tube light as the column and that is cleaned with the sand and add the plastic tubes on both sides of the tube light. Now take a u-tube manometer and connect the top and bottom of the tube light. The plastic tubes are connected to the storage tank. The top tube also connected to the storage tank to recycle the fluid / solution.



**Fig. 12. Fluidized bed setup**

From the Storage tank, the water is pumped through a pump to the Fluidized column through a rotameter and valve. Rotameter is used to measure the flow rate of the water and a valve is used to control the flow rate of water. The water from the outlet of the Fluidized column is recycled into the storage tank. The flow/ schematic diagram is given in the Fig 8. And the manometer is connected at the inlet and outlet of the column to calculate the pressure difference.

### 3.1.5 Hydrodynamic study of red mud pallets in fluidized bed:

The hydrodynamic study of red mud pallets has been studied in this chapter 3.2. This study is done by using the two-phase fluidized bed that is fabricated before in 3.1.1. we have used the tap water for this studies. The experiment is carried out with 4 different bed heights they are 6cm, 8cm, 10cm, and 12cm. The density of the pallets has been calculated by use the by using the formula.

Density of the pallets = weight of the pallets / volume of the pallets

As a result, the density of the pallets is 0.9—gms/cc. With this density we have calculated the weight required for the 6cm bed height and other heights also. And the experiment has been done with different flow rates and with the different bed heights. The results of the study is given in the chapter 4. From this results we have calculated the pressure drop, superficial velocity, Expansion ratio and Fluctuation ratio by using the below formulas.

Pressure drop = manometer difference \* (density of  $\text{CCl}_4$  - density of water) \* gravity

Superficial velocity = flow rate of water / area of the bed

Expansion ratio = The highest level of the fluidized bed + The lowest level of the fluidized bed / initial height of the bed

Fluctuation Ratio = The highest level of the fluidized bed/The lowest level of the fluidized bed

## CHAPTER-4

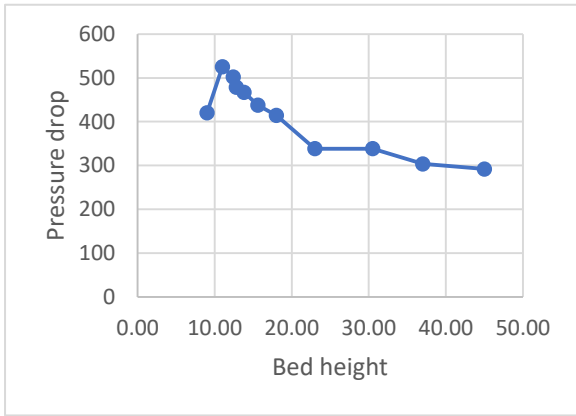
### RESULTS & DISCUSSION :

#### 4.0 Results and Discussion

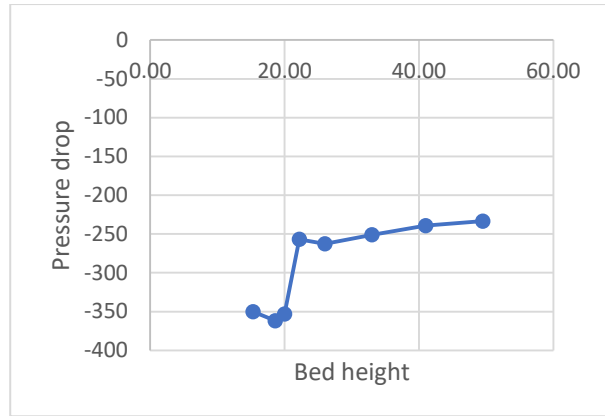
Study of the behavior of pallets in the water and how the pressure drop is changing with respective to the height of the bed has been discussed in this chapter.

##### 4.0.1 Effect bed height by Pressure drop

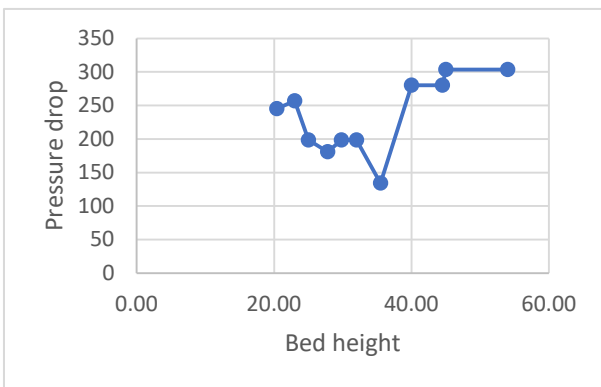
The pressure drop across the column is compared with the height of the bed in 6cm, 8cm, 10cm, 12cm initial bed heights. The pressure drop in the 6cm bed height is shown in the Fig.13(a) (see Appendix B) with increasing the bed height pressure drop is decreasing and after the bed is reaching to 20cm pressure drop becoming constant. In the 8cm bed the pressure drop is initial increases the it become constant that is shown in the Fig 13(b) (see Appendix c). similarly, the 10cm and 12cm pressure drops are shown in the Fig 13(c) and 13(d) (see Appendix d and Appendix e).



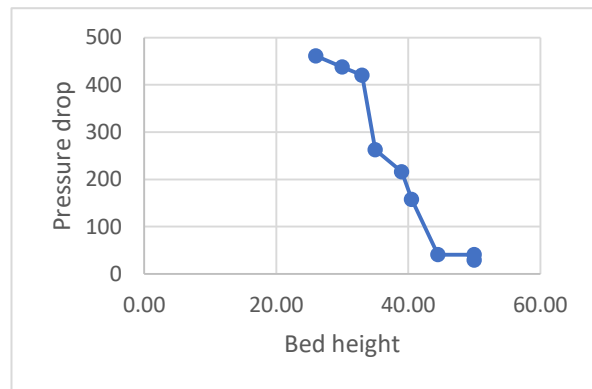
**Fig. 13(a).** Pressure drop in 6cm bed



**Fig. 13(b).** Pressure drop in 8cm bed



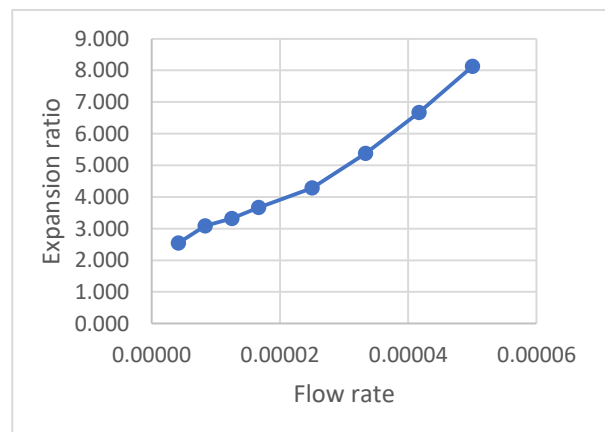
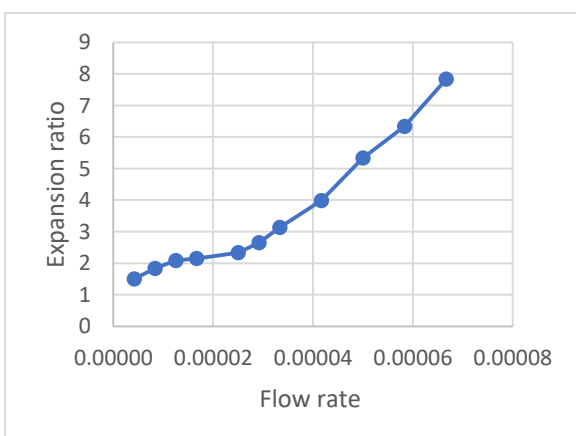
**Fig. 13(c).** Pressure drop in 6cm bed

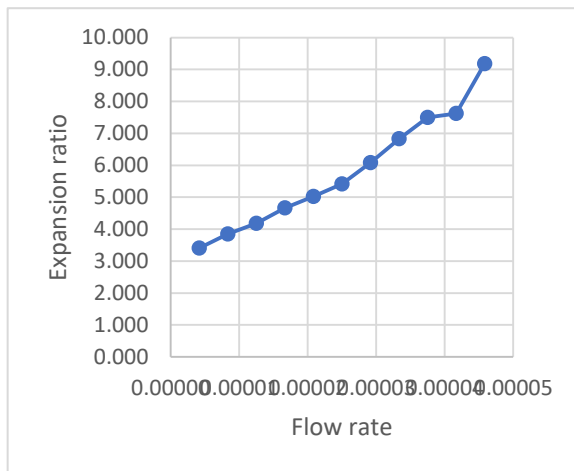
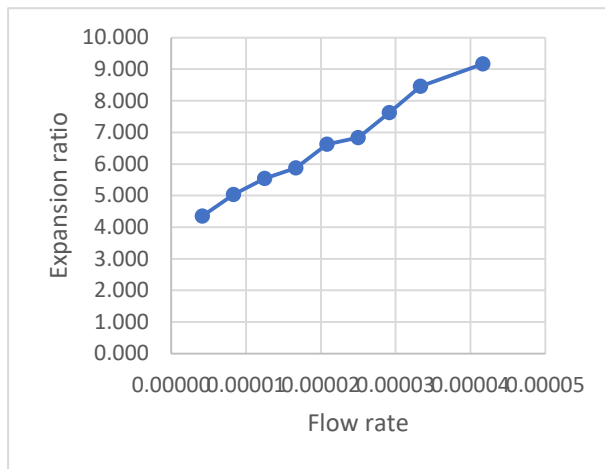


**Fig. 13(d).** Pressure drop in 8cm bed

**4.0.2 Effect on bed height by flow rate**

Bed height also effected by the flow rate of the water that has been calculated and a graph is drawn between the Expansion ratio and flow rate. In 6cm bed if the flow rate is increasing then the expansion ratio also increasing as shown in the Fig 14(a) (see Appendix B). Similarly in 8cm, 10cm, and 12cm the expansion ratio is increasing that can be seen in the below Fig 14(b), 14(c) & 14(d)(see Appendix C, Appendix D, and Appendix E).



**Fig. 14(a).** Expansion ratio in 6cm bed**Fig. 14(b).** Expansion ratio in 8cm bed**Fig. 14(c).** Expansion ratio in 10cm bed**Fig. 14(d).** Expansion ratio in 12cm bed

CHAPTER-5

**CONCLUSION :****5.0. Conclusion**

For the adsorption of As(III), red mud was chosen as an adsorbent. The red mud was formed into pallets using sodium alginate and shaped using calcium chloride solution. For continuous removal of As(III), a fluidized column was fabricated. For measuring the bed pressure drop, two tapping in the fluidization column were connected to the two limbs of U-tube manometer. Further, the hydrodynamic characteristics viz. bed pressure drop, fluctuation ratio and expansion ratio of red mud pallets were studied in the fabricated fluidized bed using water as a medium. The hydrodynamic study will further help in designing and scaling up of the fluidized bed for removal of As(III).

**5.1. Future scope of work**

- Investigation on the effect of initial concentration, superficial velocity of water, pH, and adsorption capacity in the fluidized.
- Study of adsorption kinetics.
- Comparison of adsorption capacity in a fluidized bed with the earlier batch adsorption studies.
- Development of correlation for the adsorption capacity using RSM-based and CCD
- Optimization of the operating parameters for design and scaleup.

**REFERENCES :**

- [1] W.A.H. Altowayti, N. Othman, S. Shahir, A.F. Alsharif, A.A. Al-Gheethi, F.A.H. AL-Towayti, Z.M. Saleh, S.A. Haris, Removal of arsenic from wastewater by using different technologies and adsorbents: a review, *Int. J. Environ. Sci. Technol.* 19 (2022) 9243–9266. <https://doi.org/10.1007/s13762-021-03660-0>.
- [2] P.K. Sahoo, K. Kim, A review of the arsenic concentration in paddy rice from the perspective of geoscience, *Geosci. J.* 17 (2013) 107–122. <https://doi.org/10.1007/s12303-013-0004-4>.
- [3] M.S. Rahaman, M.M. Rahman, N. Mise, M.T. Sikder, G. Ichihara, M.K. Uddin, M. Kurasaki, S. Ichihara, Environmental arsenic exposure and its contribution to human diseases, toxicity mechanism and management, *Environ. Pollut.* 289 (2021) 117940. <https://doi.org/10.1016/J.ENVPOL.2021.117940>.
- [4] N.R. Nicomel, K. Leus, K. Folens, P. Van Der Voort, G. Du Laing, Technologies for arsenic removal from water: Current status and future perspectives, *Int. J. Environ. Res. Public Health.* 13 (2015) 1–24. <https://doi.org/10.3390/ijerph13010062>.
- [5] M.A. Hashim, A. Kundu, S. Mukherjee, Y.S. Ng, S. Mukhopadhyay, G. Redzwan, B. Sen Gupta, Arsenic removal by adsorption on activated carbon in a rotating packed bed, *J. Water Process Eng.* 30 (2019). <https://doi.org/10.1016/j.jwpe.2018.03.006>.
- [6] I. Jacukowicz-Sobala, D. Ociński, P. Mazur, E. Stanisławska, E. Kociołek-Balawejder, Cu(II)-Fe(III) oxide doped anion exchangers – Multifunctional composites for arsenite removal from water via As(III) adsorption and oxidation, *J. Hazard. Mater.* 394 (2020). <https://doi.org/10.1016/j.jhazmat.2020.122527>.
- [7] S. Mandal, M.K. Sahu, R.K. Patel, Adsorption studies of arsenic(III) removal from water by zirconium polyacrylamide hybrid material (ZrPACM-43), *Water Resour. Ind.* 4 (2013) 51–67. <https://doi.org/10.1016/j.wri.2013.09.003>.
- [8] V.M. Boddu, K. Abburi, J.L. Talbott, E.D. Smith, R. Haasch, Removal of arsenic (III) and arsenic (V) from aqueous medium using

chitosan-coated biosorbent, *Water Res.* 42 (2008) 633–642. <https://doi.org/10.1016/j.watres.2007.08.014>.

[9] V. Jaiswal, S. Saxena, I. Kaur, P. Dubey, S. Nand, M. Naseem, S.B. Singh, P.K. Srivastava, S.K. Barik, Application of four novel fungal strains to remove arsenic from contaminated water in batch and column modes, *J. Hazard. Mater.* 356 (2018) 98–107. <https://doi.org/10.1016/j.jhazmat.2018.04.053>.

[10] R. Dolphen, P. Thiravetyan, Reducing arsenic in rice grains by leonardite and arsenic-resistant endophytic bacteria, *Chemosphere.* 223 (2019) 448–454. <https://doi.org/10.1016/j.chemosphere.2019.02.054>.

[11] I. Ali, Z.A. Al-Othman, A. Alwarthan, M. Asim, T.A. Khan, Removal of arsenic species from water by batch and column operations on bagasse fly ash, *Environ. Sci. Pollut. Res.* 21 (2014) 3218–3229. <https://doi.org/10.1007/s11356-013-2235-3>.

[12] D. De, V. Aniya, B. Satyavathi, Application of an agro-industrial waste for the removal of As (III) in a counter-current multiphase fluidized bed, *Int. J. Environ. Sci. Technol.* 16 (2019) 279–294. <https://doi.org/10.1007/s13762-018-1651-9>.

[13] D.V. Cuong, P.C. Wu, L.I. Chen, C.H. Hou, Active MnO<sub>2</sub>/biochar composite for efficient As(III) removal: Insight into the mechanisms of redox transformation and adsorption, *Water Res.* 188 (2021) 116495. <https://doi.org/10.1016/J.WATRES.2020.116495>.

[14] S. Samal, A.K. Ray, A. Bandopadhyay, Proposal for resources, utilization and processes of red mud in India — A review, *Int. J. Miner. Process.* 118 (2013) 43–55. <https://doi.org/10.1016/J.MINPRO.2012.11.001>.

## Appendix :

### Appendix A

Table.01: Composition of Red mud

Composition	Percentage
SiO <sub>2</sub>	5.67
LOI	9.03
Fe <sub>2</sub> O <sub>3</sub>	59.91
Al <sub>2</sub> O <sub>3</sub>	13.61
CaO	0.56
MgO	0.40
P <sub>2</sub> O <sub>5</sub>	0.13
F	0.06
Na <sub>2</sub> O	2.51
K <sub>2</sub> O	0.02
Fe	41.87
TiO <sub>2</sub>	4.97
FeO	0.28

### Appendix B

Table.02: Fluidization of 6cm bed

S.NO	WATER FLOW RATE (LPM)	MANOMETTER READINGS			BED HIGHT		PRESSURE DROP	Q(m <sup>3</sup> /s)	SUPERFICIAL VELOCITY (m/s)	R	r
		LEFT	RIGHT	Rm	UP	DOWN					
1	0.25	13.80	21.00	7.20	9.00	9.00	420.2604	0.0000041667	0.00014	1.5	1
2	0.50	12.50	21.50	9.00	11.00	11.00	525.3255	0.0000083333	0.00028	1.83	1
3	0.75	12.80	21.40	8.60	12.60	12.40	501.9777	0.0000125000	0.00042	2.08	1.0161
4	1.00	13.00	21.20	8.20	13.00	12.80	478.6299	0.0000166667	0.00056	2.15	1.0156
5	1.50	13.10	21.10	8.00	14.20	13.80	466.956	0.0000250000	0.00084	2.33	1.0290
6	1.75	13.30	20.80	7.50	16.20	15.60	437.7713	0.0000291667	0.00098	2.65	1.0385
7	2.00	13.50	20.60	7.10	19.60	18.00	414.4235	0.0000333333	0.00112	3.13	1.0889
8	2.50	14.20	20.00	5.80	24.80	23.00	338.5431	0.0000416667	0.00140	3.98	1.0783
9	3.00	14.20	20.00	5.80	33.50	30.50	338.5431	0.0000500000	0.00168	5.33	1.0984
10	3.50	14.50	19.70	5.20	39.00	37.00	303.5214	0.0000583333	0.00196	6.33	1.0541
11	4.00	14.50	19.50	5.00	49.00	45.00	291.8475	0.0000666667	0.00223	7.83	1.0889



## Appendix C

Table.03: Fluidization of 8cm bed

S.NO	WATER FLOW RATE (LPM)	MANOMETTER READINGS			BED HIGHT		PRESSURE DROP	Q(m <sup>3</sup> /s)	SUPERFICIAL VELOCITY (m/s)	R	r
		LEFT	RIGHT	Rm	UP	DOWN					
1	0.25	20.00	14.00	-6.00	15.20	15.30	-350.217	0.0000041667	0.00013968	2.542	0.993
2	0.50	20.10	13.90	-6.20	18.40	18.60	-361.891	0.0000083333	0.00027936	3.083	0.989
3	0.75	20.15	14.10	-6.05	19.80	20.00	-353.135	0.0000125000	0.00041904	3.317	0.990
4	1.00	19.00	14.60	-4.40	21.80	22.20	-256.826	0.0000166667	0.00055872	3.667	0.982
5	1.50	19.00	14.50	-4.50	25.40	26.00	-262.663	0.0000250000	0.00083808	4.283	0.977
6	2.00	19.00	14.70	-4.30	31.50	33.00	-250.989	0.0000333333	0.00111744	5.375	0.955
7	2.50	19.00	14.90	-4.10	39.00	41.00	-239.315	0.0000416667	0.0013968	6.667	0.951
8	3.00	19.10	15.10	-4.00	48.00	49.50	-233.478	0.0000500000	0.00167616	8.125	0.970

## Appendix D

Table.04: Fluidization of 10cm bed

S.NO	WATER FLOW RATE (LPM)	MANOMETTER READINGS			BED HIGHT		PRESSURE DROP	Q(m <sup>3</sup> /s)	SUPERFICIAL VELOCITY (m/s)	R	r
		LEFT	RIGHT	Rm	UP	DOWN					
1	0.25	15.00	19.20	4.20	20.50	20.40	245.1519	0.0000041667	0.00013968	3.408	0.995
2	0.50	15.00	19.40	4.40	23.20	23.00	256.8258	0.0000083333	0.00027936	3.850	0.991
3	0.75	15.40	18.80	3.40	25.20	25.00	198.4563	0.0000125000	0.00041904	4.183	0.992
4	1.00	15.50	18.60	3.10	28.20	27.80	180.9455	0.0000166667	0.00055872	4.667	0.986
5	1.25	15.40	18.80	3.40	30.50	29.80	198.4563	0.0000208333	0.0006984	5.025	0.977
6	1.50	15.50	18.90	3.40	33.00	32.00	198.4563	0.0000250000	0.00083808	5.417	0.970
7	1.75	15.50	17.80	2.30	37.50	35.50	134.2499	0.0000291667	0.00097776	6.083	0.947
8	2.00	14.60	19.40	4.80	42.00	40.00	280.1736	0.0000333333	0.00111744	6.833	0.952
9	2.25	14.60	19.40	4.80	45.50	44.50	280.1736	0.0000375000	0.00125712	7.500	0.978
10	2.50	14.50	19.70	5.20	46.50	45.00	303.5214	0.0000416667	0.0013968	7.625	0.968
11	2.75	14.50	19.70	5.20	56.20	54.00	303.5214	0.0000458333	0.00153648	9.183	0.961

## Appendix E

Table.05: Fluidization of 13cm bed

S.NO	WATER FLOW RATE (LPM)	MANOMETTER READINGS			BED HIGHT		PRESSURE DROP	Q(m <sup>3</sup> /s)	SUPERFICIAL VELOCITY (m/s)	R	r
		LEFT	RIGHT	Rm	UP	DOWN					
1	0.25	21.20	13.30	7.90	26.20	26.00	461.11905	0.0000041667	0.00013968	4.350	0.992
2	0.50	21.00	13.50	7.50	30.40	30.00	437.77125	0.0000083333	0.00027936	5.033	0.987
3	0.75	20.80	13.60	7.20	33.50	33.00	420.2604	0.0000125000	0.00041904	5.542	0.985

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4	1.00	19.50	15.00	4.50	35.50	35.00	262.66275	0.0000166667	0.00055872	5.875	0.986
5	1.25	19.00	15.30	3.70	40.50	39.00	215.96715	0.0000208333	0.0006984	6.625	0.963
6	1.50	18.50	15.80	2.70	41.50	40.50	157.59765	0.0000250000	0.00083808	6.833	0.976
7	1.75	17.50	16.80	0.70	47.00	44.50	40.85865	0.0000291667	0.00097776	7.625	0.947
8	2.00	17.50	16.80	0.70	51.50	50.00	40.85865	0.0000333333	0.00111744	8.458	0.971
9	2.50	17.40	16.90	0.50	60.00	50.00	29.18475	0.0000416667	0.0013968	9.167	0.833