

An Ecofriendly Approach of Rice Husk in Phosphate Removal and it's Synthesis in Silica Extract from Waste Residues

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Abstract -Water plays an important role in human development and for sustaining various forms of life on Earth. The physio-chemical characteristic of water helps us in determining the water quality. The present study was undertaken to carry out the analysis of phosphate present in the river water from various locations of Pune city such as Mula Mutha (Bund Garden), Alandi (Indrayani), Katraj Lake, Ambegaon (Jambhulwadi Lake). Parameters like DO and COD were also tested for the accurate data. It is found that concentration is higher than the permissible limit and it can be possible that the increase in the concentration can be a reason of Eutrophication. Adsorption with Activated Rice husk ash (ARAH) is one of the most effective, convenient and cheapest method for removal of phosphate from River water. The study revealed that the optimum conditions for the removal of phosphate compounds were achieved with percentage of 70%-80% by activating rice husk with HCL. The efficiency of locally available agro-waste rice husk was examined in batch mode for the removal of phosphate using river wastewater. Rice husk is a fuel with major constituent (>90 of ash) being silica. Characterization of adsorbent was done with the help of Electron Microscope which indicates the crystalline silica nature and presence of Si-O-Si group. Rice Husk Ash is the best source of silica and can be easily extracted by forming Xerogels with using basic solution such as NaOH. The effect of various parameters such as time of contact, adsorbent concentration, pH and temperature were also studied. Up to 89% phosphate removal was achieved at pH 6 using 2g/L dose in 120 min of contact time.

Key Words- Rice Husk Activated Carbon (RHAC), Phosphate, COD, DO, River Water Sample[Jambhulwadi-Lake, Katraj-Lake, Indrayani- River{Alandi}, Mula-Mutha River{Bund-Garden}], Silica gels, HCl, NaOH, Spectrophotometer, Muffle Furnace etc.

1. INTRODUCTION

Rapid increase in population, urbanization and industrialization results in tremendous waste production. These wastes may sometimes be disposed into the water bodies without adequate treatment which in turn may have a huge negative impact on the surrounding environment, aquatic life systems and on human health. The generated waste water comprises of various contaminants such as nitrates, phosphate, heavy metals, suspended solid, sulphate etc.

The sources of dissolved Oxygen in water through aquatic plants which as a result of photosynthesis evolve oxygen and air from where Oxygen is dissolved in water depending on salinity, temperature and water movement. In eutrophic water reservoir like rivers the organic nutrients accumulate abundantly which in turn are subjected to microbial decomposition. More growth of micro-organisms, plants etc. in them lead to depletion of oxygen. This depletion increases with increase in water depth, high altitude and warm water. Dissolved Oxygen is measured by titrimetric method. The principle behind this mechanism is that the dissolved oxygen combines with Manganese Hydroxide $[Mn(OH)_2]$ which in turn liberates iodine after acidification with Sulphuric Acid (H_2SO_4). The Iodine (I₂) can be titrated with Sodium Thiosulphate ($Na_2S_2O_3$) solution by using Starch indicator. DO is determined by Winkler's method. Potassium Iodide (KI) is produced and it cannot be oxidized by Sodium Thiosulphate ($Na_2S_2O_3$). Hence, Manganese Hydroxide $[Mn(OH)_2]$ is used as an Oxygen carrier to bring reaction between KI and O₂. Chemical Oxygen Demand (COD) is used to measure the total quantity of Oxygen –consuming substances in the complete chemical breakdown of organic substances in water. COD determines the quantity of Oxygen required oxidizing the organic matter in water sample. The organic material that are present in the river water sample gets completely oxidized by Potassium Dichromate ($K_2Cr_2O_7$) in the presence of Sulphuric Acid (H_2SO_4), Silver Sulphate ($AgSO_4$) and Mercuric Sulphate ($HgSO_4$) for the production of CO₂ and H₂O. The sample is back flowed with a known amount of Potassium Dichromate ($K_2Cr_2O_7$) in Sulphuric Acid medium and excess $K_2Cr_2O_7$ is determined by titrating against Ferrous Ammonium Sulphate using Ferrous ion indicator. The amount of O₂ required to oxidize the organic matter is directly equivalent to the source of dichromate consumed by the sample.

Sources of water bodies in Pune such as Indrayani River (Alandi), Katraj River (Katraj), Mula Mutha River (Bund garden), and Jambhulwadi Lake (Katraj) were the site of experimentation. Several small scale industries near these rivers discharge their

waste effluents to the water bodies creating a problem and adding pollution. Human wastes such as plastics and other toxic wastes also add up to the contamination. These problems must be addressed and should be given a proper attention to come up with a solution for an urgent efficient treatment.

Phosphate

Phosphorus is mainly used in agriculture sector as fertilizer and in households as detergent, resulting in release of phosphate into the environment (Rout et al., 2014). The other major sources of phosphate include weathering of rocks as well as industrial activities (Baghel et al., 2006). Phosphate is an important nutrient required for the growth and development of plants and aquatic life. However if the phosphate level exceeds its limit, high concentration of phosphate may lead to algal blooms. Eutrophication mainly occurs when the phosphate concentration is higher than the 0.02 mg/l in water bodies (Kilpimaa et al., 2014). Kidney damage and osteoporosis have been reported due to the consumption of high concentration of phosphate (Oliveira et al., 2012). The continuous discharge of phosphate level in water system stimulates the growth of toxins in the water bodies (Zhang et al., 2011).

Estimation of Phosphate is a colorimetric method which is based on Beer Lambert Law. Phosphate will readily react with Ammonium Molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}]$ to form Molybdophosphoric Acid $[\text{H}_3\text{PMo}_{12}\text{O}_{40}]$ or Phosphomolybdic acid, also known as Dodeca Molybdophosphoric acid or PMA, is a yellow-green chemical compound that is freely soluble in water and is a polar organic solvents such as ethanol. In presence of suitable reducing agents to form a blue colored complex, the intensity of light is directly proportional to the concentration of Phosphate in the solution. [8] ANALYSIS AND REMOVAL OF PHOSPHATE FROM WASTEWATER BY USING RICE HUSK - Prof. S. M. Gawande, Ms. Prachi K. Shelke², Ms. Neha A. Dhoke³, Ms. Madhura D. Lengre⁴, Ms. Diksha A. Dere et.al.

The Phosphate content of an unknown concentration can be obtained first by plotting series of standard solutions against the corresponding concentrations, by using a standard calibration curve. The concentration of Phosphate in the unknown sample can then be determined from the graph. Normal range varies from 0.05- 0.3 mg/dm³ or 2.5- 4.5 mg/dL.

Recently more attention has been given to the natural material such as rice husk for the removal of phosphate from the water.

Rice husk.

Rice husk is an agricultural waste obtained from the rice mill. The annual generation of rice husk in India is in the range of 18–22 million tons (Srivastava et al., 2006). Rice husk removal during rice refining creates disposal problem due to its low commercial value. The handling and transportation of rice husk is problematic due to its low density. Much of the rice husk produced from the processing of rice is either burnt or dumped as waste. Burning of rice husk in open fields cause environmental and health problems in the surrounding areas especially in developing countries. Therefore, it is very important to fully utilize the rice husk ash.

The present study deals with the utilization of agro-waste rice husk ash for the removal of phosphate from wastewater. Batch mode adsorption studies were performed to study the influences of various experimental parameters like pH, adsorbent dosage, contact time and the effect of temperature using activated rice husk ash (ARHA). Various kinetic and isotherm models were also applied to investigate the adsorption capacity of ARHA for phosphate removal from waste water.

Why we choose rice husk for this experiment?

- Activated charcoal acts as adsorbent which helps in removal of phosphate from wastewater.
- Utilizing agro-waste residue and getting the best from it and also to Minimize agro-waste residue from the environment.
- Extraction of silica from the rice husk for various applications in different industrial sector as silica being the major constituent of rice husk ash.
- Rice husk ash also aid importance to agricultural sector in micro-green plantation. The ash increases the soils

pH. There by increasing the availability of phosphorus improving the aeration in the crop root zone and also increase the water holding capacity and level of exchangeable potassium and magnesium (AICOAF2001).

- Abundantly available in cheap estate.
- Environmental friendly.
- Efficient way of treating wastewater.

Silica(extraction of silica by using rice husk ash)

Chemically, silica is an oxide of silicon and is generally colorless to white and insoluble in water. When associated with metals or minerals the family of silicates is formed. Silica has widespread industrial applications including use as a food additive, i.e., anti-caking agent, as a means to clarify beverages, control viscosity, as an anti-foaming agent, dough modifier, and as an excipient in drugs and vitamins.

The composition of Silica found in RHA is mainly in the form of active silica [69, 70]. The active silica thus possesses high specific surface area and porosity to the minimum extent [17]. Figure 2 shows the SEM micrograph of RHA after combustion at 750 °C for 6 h.

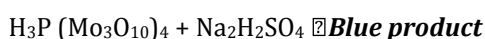
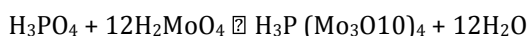
A porous structure which would give high specific surface area could clearly be seen in the micrograph. According to the study reported by Liou (2004), nitrogen adsorption carried out on RHA reveals an arrow and bi-modal pore size distribution based on the Barrett-Joyner-Halenda (BJH) method (desorption) [71],with mode pore size of approximately 2.5and 4.0nm. Then arrow pore size distribution of RHA that falls in the mesoporous range could be utilized for various applications, particularly for applications involving adsorption.

The application of siliceous RHA in cement as a pozzolanic material has been widely studied due to the high percentage of silica content and greater porosity nature which is best suited for environment and causes less pollution on the agricultural ground. RHA could be further converted into bioactive Silica Xerogel further. Initially RHA is converted into precursor sodium silicate solution through a hydrothermal process. Further conversion of sodium silicate into silica Xerogel is as given in Figure 2. Subsequently, the purified RHA is converted into sodium silicate through heating and stirring of the RHA with sodium hydroxide and distilled water. The resulting mixture is centrifuged to recover the solution, sodium silicate. Silica is dissolved in NaOH to extract sodium silicate solution. The pH of the extracted sodium silicate solution is then adjusted to pH 6 with the addition of HCl solution. The obtained gel is dried at 100°C for 12 hours, resulting in silica Xerogel.

Materials and method.

3.1. C. To determine Phosphate in River water sample without RHA:-.

☒ *Chemical reaction:-*



☒ *Materials Requirement:-*

Glassware: - test tubes, cuvette, pipette, and beaker *Instruments:* - spectrophotometer, test tube rack *others:* - distilled water tissue paper marker etc.

☒ *Reagents:-*

1. Ammonium Molybdate solution(50ml)

[Solution a☒Dissolve 25 gram of Ammonium Molybdate in 1.73mL distilled water.

Solution b [Add 280ml of H_2SO_4 to 400mL of distilled water and cool. Mix the solution "a" and "b" and dilute it to make 1 L.]

2. Stannous Chloride solution

[Dissolve 2.5 gram of Stannous Chloride in 100 ml of Glycerol by heating on a water bath for rapid dissolution.]

3. Standard Phosphate solution

[Dissolve 4.385 gram of dried anhydrous Potassium Hydrogen Phosphate in distilled water and make up the volume to 1 liter and further dilute the solution 100 times.]

☐ Preparation of standard curve:-

- i. Make a various dilutions at the interval of 0.1 mg/ml from the Standard Phosphate solution according to the given table.
- ii. Take 50ML of each dilution and add 2ml of Ammonium Molybdate solution and add 5 drops of $SnCl_2$
- iii. Take readings at 690 nanometer and plot a graph between absorbance and concentration.

Protocol. Take 50ML of filtered clear sample in a cleanflask.

1. Add 2ml of Ammonium Molybdate followed by 5 drops of $SnCl_2$ to solution.
2. Blue color solution will appear and take the reading at 690 nm in a spectrophotometer using distilled water as blank.
3. Take the readings after 5 minutes but before 12 minutes of addition of last reagent.

Find out the concentration with the help of standard curve.

Objective2. Isolation of Rice Husk Activated Carbon from Rice Husk and it's analysis of Phosphate removal efficiency in River water sample.

Preparation of adsorbent.

The Rice Husk Ash (RHA) which is a raw material for the experiment was arranged from a Rice Mill from Dadachi Wasti Lohegaon, Pune. It passed through various sieving process before the actual pre-treatment of RHA. The Rice Husk Ash was washed thoroughly using double distilled water and heated at a very higher temperature around $500^\circ C$ for 2 hours in muffle furnaces.

The Rice Husk Ash was then activated by using 1N Hcl to enhance the surface molecules to get charged up by the application of acid. The mixture of Hcl and Rice Husk Ash was maintained in the ratio of 1:1 for 24 hours at room temperature to prepare the ARHA. Later, ARHA was washed with double distilled water until neutral pH was obtained and later dried at $110^\circ C$ in oven for 24 hours to achieve constant weight and sieved again to ensure uniform particle size.

Batch adsorption Experiments:-.

Adsorption experiments were carried out in batch mode by using different amount of ARHA with 25 ml of River Wastewater Sample in 100 ml of conical flask and shaken on to a rotary shaker at 150 rpm at $25^\circ C$ temperature. Phosphate stock solution (1000ppm) was prepared by dissolving 4.39g potassium Dihydrogen phosphate (KH_2PO_4) in 1L of distilled water. Further serial dilutions were made to have Sample wastewater of desired phosphate concentration (10 ppm).

Adsorption studies were performed ranging pH from 1-7 with dose varying from 0.4g/L to 2g/L and having contact time of 60-240 min. After adsorption, the solution was filtered through the Whatman filter paper (42 No: 125mm) and phosphate

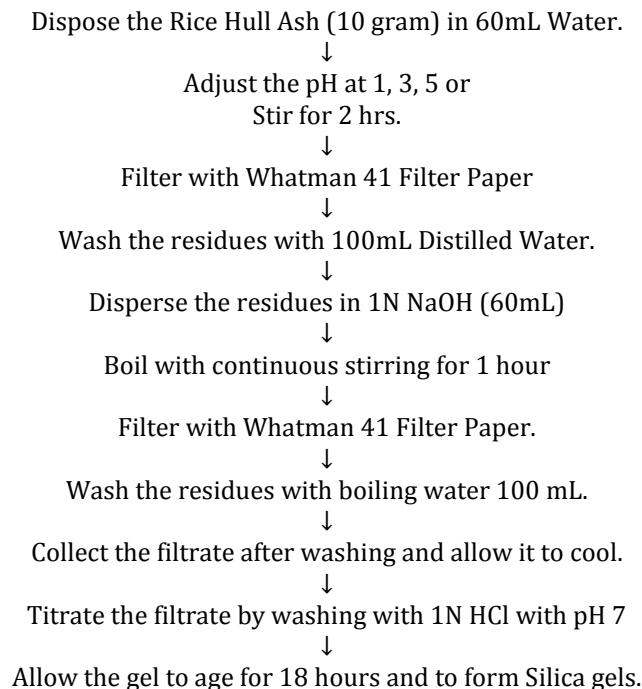
concentration from treated solution was analyzed spectrophotometrically (Make: Shimadzu-1800) at adsorption range of 690 nm using stannous chloride method. The percentage removal of phosphate was calculated by using the formula given below.

$$100 \% R_f = \frac{C_o - C_e}{C_o} \times 100 \dots (\text{Efficiency Removal Formula}).$$

Where C_o is the initial concentration (mg/l) and C_e is the final concentration of phosphate (mg/L). And R_f = Percentage removal efficiency}.

Objective3. Synthesis of Silica gel from the residual waste of Rice Husk Ash from the experiment of Phosphate removed in River water sample.

Flowchart.



Acid washing

An acid washing step is used to remove the small quantities of contaminants such as minerals before silica extraction from RHA. 10 grams of RHA samples were mixed in 60 ml of distilled water, and the pH was adjusted to 1, 3, 5 or 7 by simply using 1 N HCl. These solutions were then stirred for 2 hours on a magnetic stirrer, and were later filtered through Whatman No. 41 Filter paper and then the RHA residues were washed with 100 ml of Boiling Distilled Water. The residues were used for Silica extraction. The Filtrate and washings at each pH were collected and dried in an evaporating petri-dish. These dried Filtrates were then further used for Silica extraction in a proper defined protocol.

Silica extraction

Silica was extracted from RHA by applying the method of Kamath and Proctor (1998). 60 mL portions of 1N NaOH were added to the washed RHA residual samples and boiled in a covered 250 mL Erlenmeyer Flasks for 1 hour with constant stirring to dissolve the Silica and producing a Sodium Silicate Solution. The solutions were then Filtered through Whatman No. 41 Filter paper, and the carbon residues were washed with 100 mL of boiling Distilled Water. The Filtrates and washings were allowed to cool at room temperature and were titrated with 1N HCl with constant stirring to pH 7. Silica gels started to precipitate when the pH decreased to <10. The silica gels formed were aged for 18 hours.

[6] A facile method for production of high-purity silica Xerogels from bagasse ash- Samsudin Affandi a, Heru Setyawan et.al. [7] A simple method for production of pure silica from rice hull ash-U. Kalapathy, A. Proctor, et.al.

Result and discussion.

To determine phosphate in River water sample without RHA.

Phosphorus normally occurs in nature as phosphate which is also called as orthophosphates. Most phosphates are found as salts in ocean sediments or in rocks. Plants absorb Phosphate directly from the soil and they further proceed up the food chain after the death of animals or plants as the decayed organic matter is directly decomposed back to the soil and produces Phosphate layers. Use of Synthetic Fertilizers disturbs the Phosphorus Cycle. Sulfuric Acid is used in conversion of Phosphate Rock into a Fertilizer product called Super Phosphate. Sometimes plants may not be able to utilize Phosphate as a Fertilizer thus it is lost off from the land via Water runoff. It may further lead to Eutrophication.



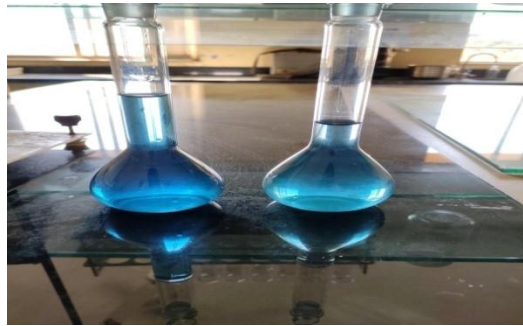
Phosphate determination using spectrophotometrically without RHA.

Objective2. Isolation of Rice Husk Activated Carbon from Rice Husk and it's analysis of Phosphate removal efficiency in River watersample.

Rice Husk Ash was washed thoroughly using double distilled water and heated at a very higher temperature around 500°C for 2 hours in muffle furnaces. Rice Husk Ash was activated using 1N Hcl. The mixture of Hcl and Rice Husk Ash was maintained in the ratio of 1:1 for 24 hours at room temperature to prepare the ARHA. Later, ARHA was washed with double distilled water until neutral pH was obtained and later dried at 110°C in oven for 24 hours to achieve constant weight and sieved again to ensure uniform particle size.

Rice husk during weighing. Rice Husk Ash after treatment with HCl





Determination of Phosphate with and without RHA respectively.

Observation.

Effect of adsorbent on Phosphate removal in River Waste Water in Batch studies.



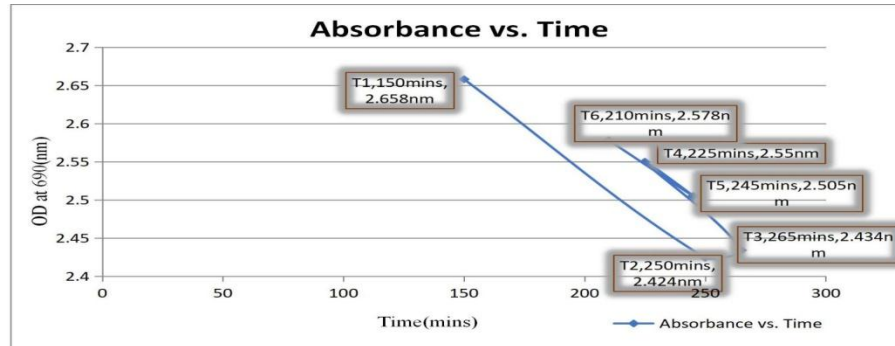
Effect of adsorbent dose and contact time

The removal of phosphate by using ARHA on River Waste Water with varying contact time (150-265 min) and adsorbent dose (0.4 g/L – 2g/L) at 25°C and 120 rpm of agitating speed. It was observed that the removal efficiency increases with increasing the contact time. Maximum 79% removal was observed using 0.8g/L dose in 265 minutes.

Observation Table -1.

<i>Sr.No.</i>	<i>Weight of Ash (grams)</i>	<i>Volume of KH₂PO₄ (mL)</i>	<i>Time (minutes)</i>	<i>pH</i>	<i>OD at 690nm</i>
T1	0.4g	1.5Ml	150	1	2.658
T2	0.6g	1.2mL	250	3	2.424
T3	0.8g	1.5mL	265	5	2.434
T4	1g	1mL	225	7	2.55
T5	1.2g	1.5mL	245	3	2.505
T6	2g	1.5mL	210	5	2.578

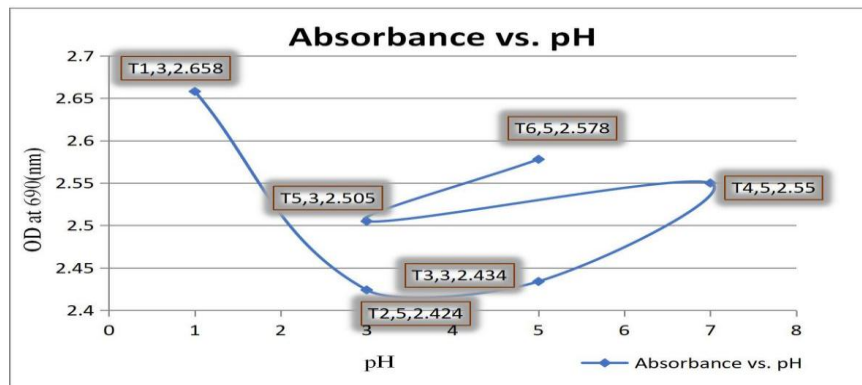
A graph shows effects of RHA adsorbent dose w.r.t Time



The result indicates that the adsorption of phosphate ions increases with the increase in the contact time and after achieving maximum removal desorption process starts.

Effect of pH

The degree of ionization and speciation of adsorbate is mainly affected by pH of solution (Chaudhary et al., 2013, Liu et al., 2013). Hence the removal of phosphate was studied using River Wastewater containing 10 ppm of phosphate and ranging pH from 1.0 to 7.0. The results show that the percentage removal decreases with increases in pH of the solution.



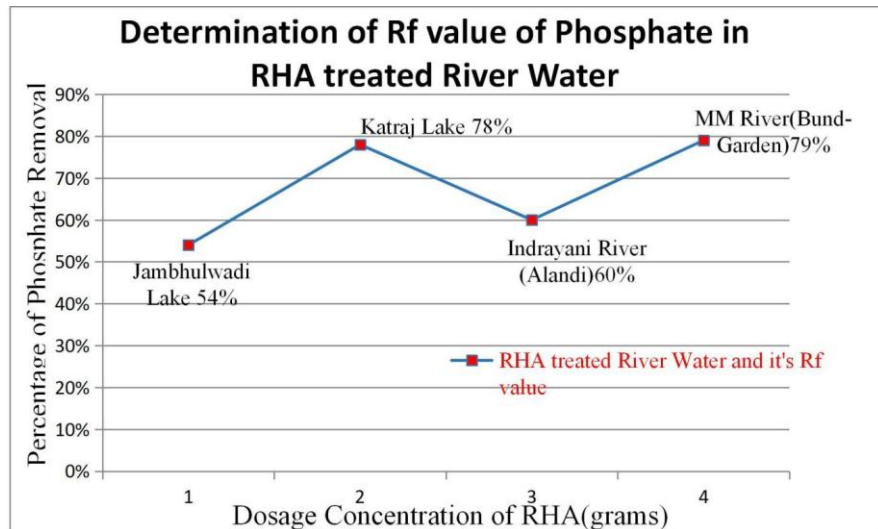
A graph shows effect of RHA adsorbent dose to pH.

Maximum removal of up to 79% was observed at pH 3.0 indicating that the phosphate removal is more efficient under acidic condition and it declines with increase in pH of solution.

Percentage Removal Efficiency: -

Observation-Table 4.2.1a.3.1:-

Sr.No.	Co-Ce/Co	Rf Value(Percentage removal efficiency)
Jambhulwadi Lake	0.54	54%
Katraj Lake	0.78	78%
Indrayani River(Alandi)	0.6	60%
Mula-Mutha River(Bund-Garden)	0.79	79%



A graph shows Rf value of Phosphate in RHA treated River Water w.r.t adsorbent concentration.

In the above mentioned graph along with the observation table suggest that the Rf value of Mula-Mutha River (Bund Garden) is highest in Phosphate Removal than the other cases. It is estimated to be 79% followed by Katraj Lake (78%), Indrayani River-Alandi (60%) and lowest in Jambhulwadi Lake i.e., (54%). We analyzed that the River of Bund garden was highly polluted with Phosphate as it is situated around the residential areas. It can be possible that the sewage and Kitchen- Agro based wastes dumped in the River directly causing increase in organic matter along with the Phosphate matter. So RHA was used as potable source in decreasing the Phosphate concentration because the increase in the Phosphate level was causing Eutrophication in the Water bodies which was creating havoc in the aquatic environment. According to the experiment, RHA which is economically feasible can be used as a best and cheapest source in decreasing the higher Phosphate concentration.

Objective 3. Synthesis of Silica gel from the residual waste of Rice Husk Ash from the experiment of Phosphate removed in River water sample.



Fig-01 (On addition of Hcl) Fig-02(Found Silica gel after 18Hrs)

Conclusion & Future Aspects

There Are Various Reasons For The Acceleration Of Phosphates In River Water Sample such as Jambhulwadi Lake, Katraj Lake, Indrayani River (Alandi) and Mula-Mutha River(Bund Garden).

The presence of Phosphate in River Water can be possible of Geographical locations. The use of Fertilizers and Pesticides in agriculture sectors, Generation of household wastes accumulates in excessive increase in Phosphate concentration. The Pune city is situated on the bank of all those rivers and so the various organic sources including Phosphate are discharged into these River water bodies causing Eutrophication. Phosphate will stimulate the growth of plankton and aquatic plants which provide

food for fish. This may cause an increase in fish population and improve the overall water quality. However, if an excess of phosphate enters the water bodies, algae and aquatic plants will grow widely, increasing temperature of the river also and using large concentration of Oxygen causing Eutrophication.

Bathing and washing clothes by using chemical detergents causing increase level of Phosphate concentration.

Rice Husk which is a waste part of agro-industries can be utilized as a cheapest and economically feasible source in treatment of Phosphate Removal. It is the best source of Silica as well and can be further extracted by forming Xerogels from Rice Husk Activated Carbon by titrating with NaOH. These Silica gels can be further purified and can be used in Pharma industries, medical diagnostic kits in which Silica can be used for surface coating, sodium silicate films etc. Since Rice Husk Ash has activated charcoal present in it and when burnt at a higher temperature, it is very useful in plant growth as well as the Charcoal provides direct Carbon source for plant's growth. In future it can play a great source in pollution control and utilizing it for water purification waste water generated from sewage plants. It can also be used in increasing biomass of plants in agricultural sector as RHA based charcoal can be used in increasing water retention capacity and further decreasing soil erosion.

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