

**Studies on the Rice Husk Ash based filters  
for the treatment of  
pulp and paper industry wastewater**

**A Dissertation**

*submitted in partial fulfilment of the requirement*

*for the award of degree of*

**Masters in Technology**

in

**Environmental Science and Technology**



Submitted

By

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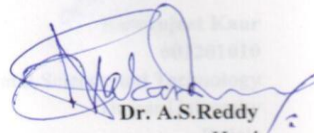
## CERTIFICATE

This is to certify that thesis entitled, "**Studies on the Rice Husk Ash based filters for the treatment of pulp and paper industry wastewater**" submitted by **Ms. Kawanjeet Kaur** in partial fulfilment of the requirements for the award of **Masters in Technology Degree in Environmental Science & Technology** at **Thapar University, Patiala** is an authentic work carried out by her under our supervision and guidance.

To the best of our knowledge, the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma.



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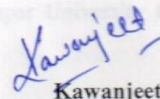


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## DECLARATION

I, the undersigned, hereby declare that the research work presented in the M.Tech project entitled “**Studies on the Rice Husk Ash based filters for the treatment of pulp and paper industry wastewater**” has been carried out by me under the supervision and guidance of *Dr. Amit Dhir, Assistant Professor, School of Energy and Environment, Thapar University, Patiala.*

Further, I declare that no part of this Dissertation has been submitted for a degree or any other qualification of any other university or examining body in India/elsewhere.



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## ACKNOWLEDGEMENT

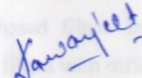
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The successful completion of any task would be incomplete without acknowledging the people who made it possible and whose constant guidance and encouragement secured the success.

With the profound sense of gratitude and heartiest regard, I express my sincere feelings of indebtedness to my guide *Dr. Amit Dhir*, Assistant Professor, School of Energy and Environment, Thapar University for his positive attitude, excellent guidance, constant encouragement, keen interest, invaluable co-operation, generous attitude and above all his blessings. He has been a source of inspiration for me.

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Last but not the least I would like to express my heartfelt thanks to my brother, my parents, and my loving friends who with their thought provoking views, veracity and whole hearted co-operation helped me in doing this work.

  
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## ABSTRACT

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The demographic growth, urbanization, industrial development and climate change have fundamentally changed water issues. Industrial wastewater constitutes major source of metals, toxic materials, chloride contents, alkalinity, acidity etc. It is thus necessary to invent new models and solution to meet the local needs of water users. Paper manufacturing is highly capital, energy and water intensive industry. The effluent from these industries contains high level of Total Dissolved Solids, pH, Biological Oxygen Demand, Chemical Oxygen Demand and Colored Organic Compounds. The treatment of pulp and paper wastewater has been carried out with activated sludge process, anaerobic digestion, aerated lagoons and biological filtration method which results in the reduction of BOD to permissible levels but Total Dissolved Solids (TDS) content persists. So there is a need for the reduction of TDS so that the treated wastewater can be used for irrigation purposes. The main agro-residue raw materials used in pulp and paper industries are wheat straw, baggasse, sarkanda grass, jute rags as well as waste papers. Rice production is an important part of our national economy. During milling of paddy about 78% of weight is received as rice, broken rice and bran and rest 22% of weight is received as husk. This husk is usually used in boilers for producing steam or electricity and after the firing process solid mass is left out which is termed as Rice Husk Ash (RHA). RHA is collected from the particulate collection equipment attached upstream to the stack of the rice husk fired boilers. In India about 20 million tonnes of RHA is produced annually. This RHA is great environment threat causing damage to the land and surrounding area in which it is dumped. So there is a need to find the alternative uses of RHA. One technique may be the preparation and application of RHA based filters in the treatment of industrial wastewater. RHA based Column and Ball filters with different composition of cement, RHA and sand were prepared for the reduction of TDS, TSS and chloride content from secondary treated wastewater sample. The effect of different parameters like contact time, size of the filters and treatment efficiency were evaluated. Obtained results indicate that ball filters are better than column filters and optimum composition of ball filter was found to be 100 g cement, 50 g sand and 50 g RHA for wastewater treatment. The results showed that 71% reduction in TSS, 51% reduction in TDS and 51% reduction in concentration of chlorides was obtained after 9 h of exposure time. The experiments to prove filter repeatability were also

conducted which showed that the treatment efficiency was increased from 14 to 43% in column filters and 23 to 51% in ball filters after 5 days and thereafter, the efficiency decreased to 19 and 29% respectively after 10 repeated cycles. So, it is evident from the results that the RHA based ball filters can be employed efficiently for the treatment of wastewater at a tertiary stage for the reduction of TDS before its safe discharge. Thus, RHA based filtration can be applied in conjunction with the existing biological processes for the treatment of TDS removal in wastewater from agro residue based pulp and paper mill.

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# CHAPTER 1

## INTRODUCTION

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The increase in world's population results in the increase scarcity of water resources. The world's population is increasing at a rate of around 80 million people per year, which is leading to an increase in the demand for fresh water of around 64 billion cubic meters per year<sup>3</sup>. This demographic growth, urbanization, industrial development and even climate change have fundamentally changed water issues. It is thus necessary to invent new models and solution to meet the local needs of water users. Wastewater is any water that has been adversely affected in quality by anthropogenic influence. Classifications of wastewater:

- 1) **Domestic wastewater:** Such wastewater is produced by the mere acts of living.
- 2) **Industrial wastewater:** Wastewater produced by industrial processes is termed as Industrial wastewater. The principle physical characteristics of industrial wastewater are its solids content, color, odor and temperature and chemical characteristics include biological oxygen demand (BOD), chemical oxygen demand (COD), alkalinity, acidity, chlorides etc.

### 1.1 Pulp and Paper Industry wastewater

Paper manufacturing is highly capital, energy and water intensive industry. There are currently around 715 pulp and paper mills operating [IEP, 2013]. Among the seventeen most polluting industries identified by Ministry of Environment and Forests (MoEF), pulp and paper industry is one amongst them. The current water consumption in India is about 75 m<sup>3</sup>/tonne of product [MoEF, 2014]. In India, around 905.8 million m<sup>3</sup> of water is consumed and around 695.7 million m<sup>3</sup> of wastewater is discharged annually by this sector [CPCB, India]. The production in 2005-2006 was 5.9 million tonnes paper, against an installed capacity of 7.6 million tones [MoCL, 2007]. However, with rise in education and income level, and overall economic growth, it is estimated that demand would be over 14 million tonnes in 2020 [Jaakko Poyry Management Consulting, 2002]. The major categories for which norms have been set up are wood based, agriculture residue based and recycled fiber and market pulp based mills. The main agro-residue raw materials used in pulp and paper

industries are wheat straw, baggasse, sarkanda grass, jute rags as well as waste papers. The effluent from these industries contains high level of Total Dissolved Solids (TDS), pH, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Colored Organic Compounds. During the washing of wheat straw and rice straw the effluent contains high amount of BOD and chloride content. The treatment of wet washing effluent with 2-stage Activated Sludge process, anaerobic digestion and biological filtration method results in the reduced BOD but TDS including chloride content still persist. So there is a need for the reduction of TDS so that the treated wastewater may be used for irrigation purposes. Although the wastewater from paper and pulp industry is treated using different treatment methods like preliminary treatment, primary treatment, and secondary treatment but even then the TDS level is very high above 2000 ppm so this large figure serves to underline the importance of effluent treatment by using economic and most convenient method [ CPCB, 2011].

**Table 1.1:** Discharge standards of Finally Treated Effluent for Pulp and Paper Mills [CPCB, 2011]

<b>Parameter</b>	<b>Existing norms</b>	<b>Proposed norms</b>
Effluent volume m <sup>3</sup> /t	50	20
Ph	6.5-8.5	6.5-8.5
Suspended solids, mg/L	100	50
Total Dissolved Solids, mg/L	2100	1200
BOD, mg/L	30	30
COD, mg/L	450	225

## **1.2 Rice husk ash production and associated problem**

Rice production is an important part of our national economy. Rice husk is an abundantly available waste material in rice producing industries. During milling of paddy about 78% of weight is received as rice, broken rice and bran. Rest 22% of weight is received as husk giving an annual total production of 120 million tones accounting about one-fifth of the annual gross rice production throughout the world. It is insoluble in water, woody and silica cellulose structural arrangement. The husk

contains about 75% organic volatile matter and the balance 25% of weight of this husk is converted into ash during the firing process, is known as Rice Husk Ash (RHA). The rice husk ash (RHA) is collected from the particulate collection equipment attached upstream to the stack of the rice husk fired boilers. Burning the husk under controlled temperature under 800°C can produce ash with silica mainly in amorphous form [Chandrasekhar, 2003]. RHA in turn contains 90% amorphous silica and small proportion of alumina, potassium oxide, manganese oxide, iron oxide, calcium oxide etc [Zemke et al., 2009]. Type of ash varies according to the burning technique depending on the temperature. During the combustion at 550 to 800 °C, amorphous silica tends to be formed and at greater temperature crystalline silica is formed [Bui, 2005]. Whereas, controlled burning leads to the production of pure silica. So for every 1000 kgs of paddy milled, about 220 kgs (22%) of husk is produced, and this husk is burnt in boilers, to generate about 55 kgs (25%) of RHA. Rice husk is used as fuels in boilers for power generation. With the growing emphasis on environment friendly industries the concept of generating energy from rice husk has found great importance. It is estimated that about 70 million tons of RHA is produced annually worldwide. In India about 20 million tonnes of RHA is produced annually. This RHA is great environment threat causing damage to the land and surrounding area in which it is dumped. Handling and transportation of RHA is great problem due to its low density. Rice husk has a very low nutritional value and is not appropriate for composting. Therefore 100 million tons of rice husk ash produced globally begins to impact the environment if not disposed off properly. Huge amount of RHA is produced in Punjab region and there is a problem of disposal of RHA so there is a need to look for the alternative use of Rice Husk Ash for environment-friendly purpose. During the past few years the use of RHA has widened serving as a source of insulating materials for reinforcing the tensile strength, as fertilizers, as microbial nutrients for single-cell protein production, concrete etc.

### **Properties of RHA**

The rice husk ash (RHA) has more than 92% by wt of silica with high porosity and large surface area, because it retains a cellular structure skeleton. The physical characterization of rice husk and RHA has pointed out some properties such as the presence of functional groups that make adsorption process possible. The chemical composition of rice husk ash is given in Table 1.2

**Table 1.2:** Typical husk analysis [Bronzeoak, 2003]

Property	
Bulk Density(kg/m <sup>3</sup> )	96-160
Length of husks(mm)	2.5-5
Hardness	5.5-6.5
Ash	22.4
Carbon (%)	35.77
Hydrogen (%)	5.06
Oxygen (%)	36.59
Nitrogen (%)	.32
Sulphur (%)	.082
Moisture (%)	8.05

**Table 1.3:** Properties of RHA [Basha et al, 2005]

Component	%
Loss on ignition at 800°C	4.00
Moisture content	4.98
Silica (SiO <sub>2</sub> )	92.1
Alumina (Al <sub>2</sub> O <sub>3</sub> )	0.51
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	0.40
Calcium oxide (CaO)	0.55
Magnesium oxide (MgO)	nd
Sodium oxide (Na <sub>2</sub> O)	nd
Potassium oxide (K <sub>2</sub> O)	1.53
Titanium dioxide (TiO <sub>2</sub> )	0.02
Manganese oxide(MnO)	0.08
Phosphorus pentaoxide (P <sub>2</sub> O <sub>5</sub> )	0.36
Sulphur trioxide (SO <sub>3</sub> )	0.12

### Use of RHA in treatment

Adsorption is one of the effective ways to remove color, odor, organic and inorganic pollutants from wastewater. The adsorption process provides an alternative treatment and provides promising technology because of its simplicity. RHA has good adsorptive properties due to the presence of silica and carbon. Silica can provide

aesthetic color to composite and utilization of rice husk reduces environmental pollution and green house effect. RHA has been used for the removal of various dyes, heavy metals. Due to its refractory properties RHA is used in steel industries, ceramic industries and for the manufacture of refractory bricks. RHA also helps in improving residual soil properties. High porosity of RHA is essential to absorb oil. RHA also has extensive uses in the field of civil engineering.

### **1.3 Treatment technologies in Pulp and Paper industry wastewater treatment**

The paper and pulp industry uses large amount of water and subsequently has to treat large amount of wastewater. To prevent any health hazards caused by discharging wastewater into lakes, streams, the wastewater must be treated before discharge. Therefore there is a need for effective wastewater management plan. There are different methods for the treatment of wastewater generated from paper and pulp industry:

- 1.3.1 Preliminary treatment:** Pre-treatment includes the removal of materials that can be easily collected before they clog the pumps mainly solids and hydrocarbons. The influent passes through bar screen to remove the large objects. It includes grit removal and oil and grease removal. Acidic wastewater is neutralized using ammonia, lime or soda ash.
- 1.3.2 Primary treatment:** This is the second step in wastewater treatment process which involves the physical separation of suspended solids. This separation reduces suspended solids and BOD levels. Within the primary clarifiers suspended solids are allowed to settle down which results in 60% removal of TSS and 30% BOD removal. It prepares the wastewater for biological treatment and adjusts the pH. It involves screening, grit removal floatation, sedimentation, coagulation, filtration and flocculation.
- 1.3.3 Secondary treatment:** This process reduces the contaminants that are left in wastewater after primary treatment process. It can remove up to 90% of organic matter by biological treatment process. Different micro-organisms like bacteria and fungi decompose the organic matter and convert it into simpler

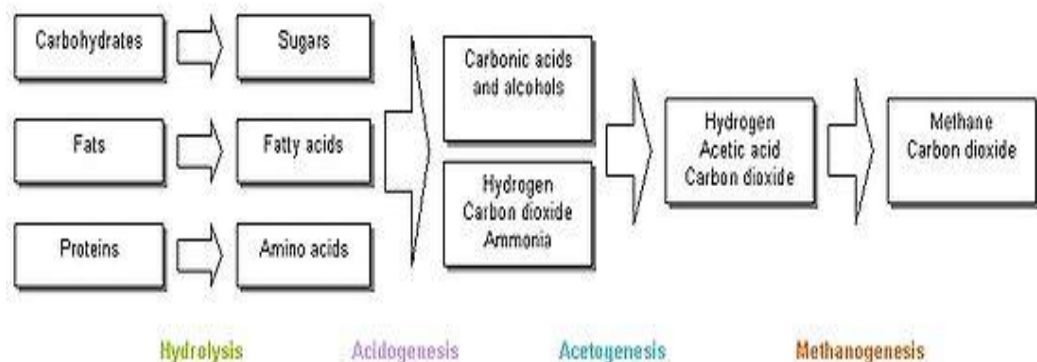
form. Biological treatment can be classified into Aerobic treatment and Anaerobic treatment.

**Aerobic treatment:** In this complex organic matter breaks down to simple organic matter with the help of micro-organisms in the presence of oxygen.

**Anaerobic treatment:** This treatment is also done with the help of microbes in the absence of oxygen and if the oxygen is needed it is supplied by the oxygenated compound like  $\text{SO}_2$ .

It consists of 4 stages:

- Hydrolysis
- Acidogenesis
- Acetogenesis
- Methanogenesis



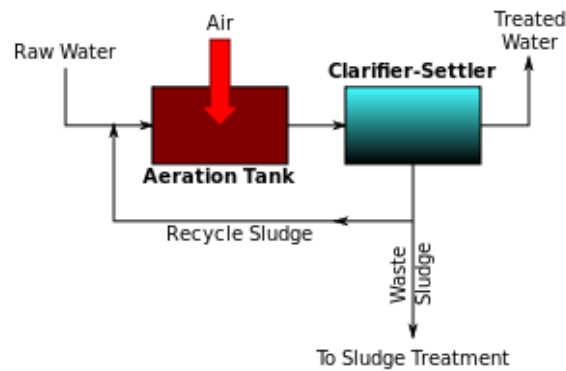
**Fig 1.1:** Anaerobic digestion

Different approaches used for secondary treatments are Suspended film systems, Fixed film systems and Lagoon systems.

**Suspended film systems:** in this organic matter and nutrients are absorbed from wastewater. For e.g.: Activated Sludge Process

**Activated sludge process:** It is most commonly used aerobic biological treatment process. It is mainly designed for removal of biodegradable organic matter and also used for removal of nutrients, TSS, pathogens and heavy metal removal. It contains of an aeration tank and settling tank. In Aeration tank, oxygen is injected and thoroughly mixed into wastewater and favorable conditions for biological treatments are maintained. In Settling tank, waste sludge is allowed to settle down and remove

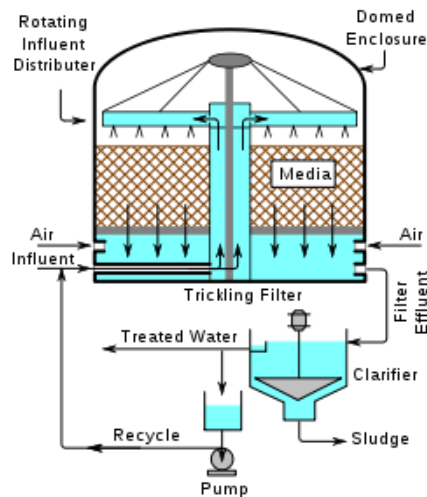
biological flocs from mixed liquor and allowed clarified secondary effluent out. It also has returned activated sludge system in this part of waste sludge is recycled to the aeration tank and remaining sludge is removed for further treatment. The normal drawbacks in the system are foaming in the aeration tank and high maintenance cost due to the use of diffuser for air supply.



**Fig 1.2:** Activated sludge process

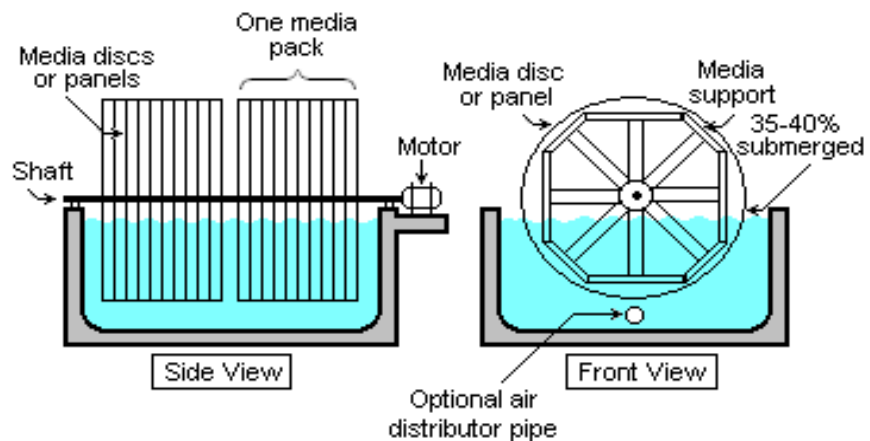
**Fixed film systems:** In this technique wastewater is spread over the substrate and nutrients and organic matter are absorbed by micro-organisms. Fixed film systems are trickling filters, rotating biological contractors and sand filters.

**Trickling filters:** In this clarified wastewater is sprayed over the packing by rotating spray arm to trickle through as a thin film over the bio-film alternatively exposing to wastewater and air and then the treated effluent is passed through secondary clarifier and treated as secondary effluent. It requires less energy and withstands toxic loads. It has some disadvantages that it is sensitive to low temperatures and it has odor and flies problem and nitrogen removal is very difficult.



**Fig 1.3:** Trickling filters

**Rotating biological contractor:** This is used to remove BOD and brings about nitrification. In this system, clarified wastewater is fed to RBC and the unit which is partially submerged is used and rotated in RBC tank and usually provide with an enclosure to prevent the algal growth, protects the discs from sunlight and to prevent heat loss. It is sensitive to temperature and when oxygen demands exceed supply then it causes order problems.



**Fig 1.4:** Rotating biological contractor

**Lagoon system:**

**Aerated lagoon system:** It consists of a large surface pond which is equipped with mechanical aerators to maintain an aerobic environment to prevent settling of suspended biomass. It treats high volume of sewage but requires high maintenance cost.

**Anaerobic lagoons:** It is used to treat high strength wastewater which stabilizes organic matter and converts it into carbon dioxide and methane. Inside these solids in wastewater separates and settle in layers. If sulfur is present hydrogen sulfide is produced which causes odor problems.

**1.3.4 Tertiary treatment:** The main tertiary treatment using membrane processes like ultra filtration, membrane bio reactors, reverse osmosis etc.

**Ultra filtration:** Treatment efficiencies using ultra filtration are high as high molecular weight compounds which are difficult to biodegrade are easy to remove. The main limitations include fouling of membranes, membranes have to be replaced periodically and smaller soluble such as chloride and metal ions are not removed.

**Reverse osmosis:** It is similar to ultra filtration but membrane pore size is much smaller so it removes chloride ions. The major limitations include pre treatment is important, it takes long amount to treat the water, not appropriate for treating water contaminated with coliform bacteria.

Tertiary treatment is expensive and has many drawbacks like membranes have to be replaced periodically, fouling of membranes, etc. So, there is a need to find an alternative way which is inexpensive. So, RHA can be used for the treatment of industrial wastewater by coupling it with biological treatment in place of tertiary treatment.

## CHAPTER-2

### REVIEW OF LITERATURE

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RHA poses great environment threat causing damage to the land and the surrounding area in which it is dumped. So there is a need to find alternative ways to use RHA so that its disposal problem can be reduced. This chapter deals with literature review listed the uses of RHA in different fields like use of RHA in concrete, production of silica from RHA and its decontamination applications.

#### 2.1 Use of RHA in concrete

Zhang et al., (1996) studied the effects of incorporation of rice husk ash in cement paste and concrete and the compressive strength was determined and the results showed that incorporation of rice husk ash in concrete reduces the porosity. Ahmad et al., (2012) studied the use of RHA in partial replacement of concrete. Concrete and mortar containing 25% RHA as a replacement of cement was used and then different analysis were carried out like X-ray diffraction, Scanning electron microscopic examination, resistance to aggressive chemicals and cost analysis were carried out. The results were better compared to conventional concrete and it leads to substantial cost savings. Ayswarya et al., (2012) studied the use of RHA for property modification of high density polyethylene (HDPE). Characterization of RHA was done with X-ray diffraction and SEM. RHA was blended with HDPE in the presence of compatibilizer. The results showed that compatibilized RHA-HDPE has better tensile strength and have excellent thermal stability which proves that RHA is a valuable reinforcing material for HDPE and has the great environment benefits. RHA was used in combination with waste glass powder (GP) as replacement of Portland cement [Ghavidel et al., 2013]. Hybrid mixtures of RHA and GP were prepared and compressive strength and strength activity index were determined for 28 days. The results showed that both RHA and GP were used together as replacement of Portland cement without any adverse effect. Noorvand et al., (2013) made an attempt for the replacement of cement with untreated RHA. Different concentrations of nano  $\text{TiO}_2$  were added into blended cement and mechanical properties were analyzed by X-Ray diffraction and Scanning Electron Microscope techniques and the result showed that nano  $\text{TiO}_2$  blended cement improved the mechanical properties of untreated RHA. Raut et al., (2013) made an attempt to utilize recycled paper mill residue and rice husk ash to improve the properties of bricks. Varying amount of recycled paper mill

residue, rice husk ash and cement was added and homogeneous mixture was prepared. Results were analyzed by using X-ray diffraction and Scanning electron microscope technique. The results showed that the bricks prepared from rice husk ash and recycled paper mill residue are of light weight and has more compressive strength.

## **2.2 Production of Silica from Rice Husk Ash**

Della et al., (2002) used RHA for the preparation of silica from rice husk ash. The product was characterized in terms of silica content, morphology, particle size and porosity. Similar study of production of silica from rice husk ash was studied by [Mittal et al, 1997] and found that a by-product Sodium Silicate is formed during this process which can be used for making bricks. Criado et al., (1996) investigated different properties of silica and explained that primary leaching of rice husk ash with hydrochloric acid before combustion at 600°C gave pure silica with high specific surface area. If the leaching was done at white ashes before combustion at 600°C, amorphous silica was obtained. Preparation of silica powders with rice husk ash by green route was studied by [Guo et al., 2010]. Na<sub>2</sub>CO<sub>3</sub> was used as the silica extraction reagent and the raw materials, RHA and waste gas, were obtained from power plant. The results were characterized by X-ray diffraction and infrared spectra (IR). The results showed that the synthetic procedure was inexpensive, environment friendly and was suitable for large scale production. Liu et al., (2012) studied the production of Silica and activated carbon simultaneously from rice husk ash with potassium carbonate and concluded that the entire procedure was simple, environmental-friendly and economically-effectively

## **2.3 Use of RHA in wastewater treatment**

Prasad., (2002) prepared a low cost water filter using nylon membrane along with the concrete mixture of RHA, pebbles and cement. It was found that there was 99% reduction in turbidity. Ganvir et al., (2002) also made an attempt to prepare such type of filters using perforated food grade plastic container which reduced the turbidity and bacterial count.

## **2.4 Decontamination applications**

RHA is basically inorganic in nature. The heating of rice husk at different temperature produces RHA containing different concentrations of carbon and silica. RHA particles

contain a large number of functional groups on which adsorbate molecule interact [Namasivayam et al., 2002]. Rice husk ash was used to prepare two sorbent materials which have the potential to remove six heavy metals Fe, Mn, Zn, Cu, Cd and Pb present in agriculture and sewage wastewater [Daifullah et al., 2003]. The efficiency of both sorbents in the removal of heavy metal was 100%. Adsorption capacity of rice husk ash is due to the presence of both silica and carbon. Katayon et al., (2005) also prepared rice husk ash into sorbent material for heavy metal removal and dye removal. Ngah et al., (2008) had done similar study to remove heavy metals using low cost adsorbents in replacement of conventional methods. It was analyzed that chemically modified plant wastes showed better adsorption capacities.

Sumanjit et al., (2001) utilized RHA as an adsorbent for the removal of different acidic dyes and COD from wastewater. Srivastava et al., (2007) studied the adsorption of Brilliant Green (BG) on RHA. Different parameters were altered like contact time, initial pH, adsorbent dose and initial concentration. Equilibrium isotherms were analyzed for adsorption for BG on RHA. The effect of temperature on BG adsorption onto RHA has also been studied.

Chowdhury et al., (2009) studied the use of RHA for the color removal from Methylene Blue (MB) and Congo Red (CR) by altering the various parameters like initial concentration of dye, contact time and adsorbent dose and the results revealed that a very high percentage removal of color was achievable for both dyes. RHA was used as an effective adsorbent for the removal of methylene blue dye [Sharma et al., 2010]. Results showed that the adsorption of methylene blue on RHA was favorably influenced by an increase in temperature. Bharathi et al., (2013) employed RHA as an adsorbent for the removal of 2 basic dyes safranin and methylene blue and the adsorption capacity was analyzed.

Feng et al., (2004) employed RHA as an adsorbent for the adsorption of lead and mercury. Different parameters were altered like ionic strength, pH, particle size and contact time and the adsorption rate and adsorption capability were checked. It was concluded that lead ions showed better results than mercury ions and finer the rice husk ash particle used, higher the pH and better adsorption of lead and mercury ions takes place. Wongjunda et al., (2010) reported the use of RHA for the removal of chromium. Treatment of RHA with NaOH results in the formation of modified RHA (MRHA). The results showed that the adsorption capacity for chromium removal has been increased. Ghorbani et al., (2012) explained the synthesis and performance of

polypyrrole nanocomposite coated on rice husk ash for removing the heavy metals such as iron, copper, manganese and zinc from wastewater. The results concluded that RHA was an efficient media for the removal of heavy metals in continuous mode using fixed bed column. Sadon et al., (2012) studied the use of RHA and MRHA for effective adsorption and different parameters were also studied. Different parameters were altered and adsorption capacity was analyzed. Hsu et al., (2007) employed RHA for the adsorption of paraquat using methacrylic acid modified rice husk ash. Carboxyl groups were bound to the surface of rice husk ash and characterization was done. Microwave incinerated rice husk ash for the adsorption of copper was employed and concluded that the microwave incinerated rice husk ash was an effective adsorbent for the removal of copper [Johan et al., 2011].

Hameed et al., (2009) made an attempt to use RHA as a novel adsorbent. RHA serves as an ideal adsorbent in refining gold-thiourea complex, removes organic and inorganic compounds from wastewater and act as an air purifier in cleaning of atmospheric pollutants. It was analyzed that the RHA was used as a novel adsorbent which overcomes the wastewater problems at reasonable cost.

Srivastava et al., (2007) studied the removal of cadmium and zinc ions from binary systems by using rice husk ash as an adsorbent. Higher percentage of metal ion removal was possible provided that the initial adsorbate concentration in the solution was low. Imyim et al., (2010) made an attempt for the removal of humic acid from water. RHA was modified with aminopropylation reaction with 3-aminopropyltriethoxysilane and the results showed better adsorption.

Zhao et al., (2013) reported the use of rice husk ash for the sorption of copper from aqueous solution. Chemical modifications were done by treating rice husk ash with  $H_3PO_4$  which increases the sorption capacity of copper. Analysis was done by X-ray Fluorescence analysis and morphological characteristics were determined by Scanning Electron Microscope. The use of rice husk ash for the removal of Arsenic from water by altering different parameters like contact times, pH and RHA dose and the removal efficiency has been studied. It was reported that 10g/L of RHA dosage can remove Arsenic by 5-12% [Saha et al., 2002].

Faccini et al., (2012) studied the use the RHA for the purification of biodiesel from the waste frying oil by using the different concentrations of RHA 1%, 2%, 3%, 4% and 5% and then test it with two different purification methods. 4% concentration of RHA showed better results for the removal impurities from biodiesel. Kenes et al.,

(2012) used thermally treated RHA and analyzed as an adsorbent for petroleum for the remedial measures of petroleum spills. Petroleum adsorption capacity was analyzed and different parameters like petroleum density, contact time and heating temperature on petroleum sorption capacity were also analyzed by X-ray diffraction method and Scanning electron microscope. Dahlan et al., (2009) worked on the removal of SO<sub>2</sub> and NO from flue gas by combustion process in fixed bed reactor by using rice husk ash and CaO based sorbent. For this various metals were used to check the best metal impregnated over RHA/CaO sorbent. The results showed that CeO<sub>2</sub> impregnated with RHA/CaO sorbent had the highest sorption capacity.

Rayner et al., (2009) made an attempt to manufacture ceramic pot filters for water treatment. Clay and RHA were used for making filters. Clay was used as binding material instead of cement. Different parameters were also altered. The use of rice husk ash in bitumen with other aggregates was reported in manufacture of building blocks “Bitublocks” which can be used to construct a Bitublock wall [Forth et al., 2006] and this resulted in less CO<sub>2</sub> emission. Khan et al., (2011) reported the work on RHA which increases the resistance of concrete towards chlorine and sulphate and make it corrosion resistant that reduces environmental problems.

Muntohar et al., (2002) used different concentrations of RHA with Lime in soil and analysed decrease in soil swelling and improvement in strength and bearing capacity. Similar study was done by [Rao et al., 2011] who used RHA and RHA in combination with Fly Ash to upgrade soil as a construction material which reduces cost. Hossain., (2011) had done the study on the stabilization of soil using Rice Husk Ash with cement and RHA with Lime and Gypsum respectively.

Mak et al., (2014) explained the approach of using RHA in replacement of polymers which proves to be environment friendly. A cleaner production was achieved by using rice husk ash blended with polypropylene in the gas-assisted injection moulding process. Different stages of injection pressure were applied and results proved to be environment friendly.

Ding et al., (2014) used rice husk for the preparation of activated carbon and liquid compound fertilizer by a simple method which combined the process of H<sub>3</sub>PO<sub>4</sub> activation and KOH etching. The results showed that activated carbon exhibit better results including high adsorption capacity and low ash content and the wastewater produced from this route was used to produce fertilizer containing Si, K and P which proves to be environment friendly.

## **2.5 Technological gaps**

Based on the literature review, the following work has not been done in previous years:

1. The high amount of silica in RHA causes risk of contamination which has not been documented.
2. The reuse and repeatability of RHA based filters has not been cited.
3. The use of RHA based filters for the treatment of wastewater has not been studied.
4. Reduction of chloride concentration from the effluent of agro-residue based industries has not been cited.
5. Treatment for reducing TDS level from the effluent of Pulp and Paper industry has not been studied.
6. The study of RHA based filter having different geometrical shapes has not been studied.

## **2.6 Objectives**

Based on the gaps the purpose of the study includes:

1. Preparation of low-cost rice husk ash based filters for the treatment of industrial wastewater.
2. Optimize different parameters to enhance treatment efficiency for removal of TDS.

## CHAPTER 3

# MATERIALS AND METHODOLOGY

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This chapter deals with the materials and the methods followed in the preparation of RHA based filters and the purification of wastewater samples using these filters.

### 3.1 Materials

- **Ordinary Portland cement (OPC)** – OPC was collected locally. It was used as binding material for rice husk ash which provides strength to the matrix.
- **Rice Husk Ash** – RHA was procured from boiler house of a pulp and paper industry, it was used to make filter which act as an adsorbent.
- **Sand** – Sand was obtained locally and it was used to provide strength to the mixture.
- **Wastewater** – Effluent samples were collected from Pulp and Paper industry near Ludhiana which was treated with anaerobic treatment followed by 2-stage activated sludge process.
- **Chemicals:** Standard silver nitrate (0.10 N, N/10 volumetric solution), Potassium chromate indicator or solution, Sulphuric acid and sodium hydroxide were procured from Loba chemicals, India.

### 3.2 Instruments:

**3.2.1 pH meter:** pH of the solution was monitored by using pH meter and pH was adjusted with the help of 0.1 N NaOH and 0.1 N HCl. Instrument was calibrated with freshly prepared buffer solutions (of pH 4 and 9) from time throughout the study.



**Fig 3.1:** pH meter

**3.2.2 TDS meter:** The TDS of the wastewater samples was measured using TDS meter.



**Fig 3.2:** TDS meter

### **3.3 Methodology**

Following are the steps for the treatment of wastewater samples using RHA based filters.

#### **3.3.1 Preparation of filters**

Different concentration of RHA, cement and sand were taken to prepare column and ball filters. These concentrations were marked as C1, C2, C3 and C4.

- C1 – 100g cement, 50g sand and 50g RHA
- C2 – 80g cement, 80g sand and 40g RHA
- C3 – 100g cement, 75g sand and 25g RHA
- C4 – 100g cement, 30g sand and 70g RHA

#### **Ball filters**

RHA and cement were taken in different quantities mentioned above and thoroughly mixed. After that the mixture was turned over number of times with the required amount of sand until an even color and consistency was observed. 100 ml water was added slowly and the mixture was further turned manually until a mixture of a sufficient workability was achieved. The balls of uniform size were prepared

manually. Thereafter, the balls were dried for half an hour in an oven at a temperature of 97°C and air dried for 24 hours.

### **Column filters**

RHA and cement were taken in different quantities mentioned above and thoroughly mixed. After that, the mixture was turned over number of times with the required amount of sand until an even color and consistency was observed. 100 ml water was added slowly and the mixture was further turned manually until a mixture of a sufficient workability was achieved. For column filter, the mixture was filled in the mould. Thereafter, the columns were dried for half an hour in an oven at a temperature of 97°C and then air dried for 24 hours.

### **3.3.2 Curing of balls and column**

Curing process was identified as one of the important part of the preparation of ball and column filters using rice husk ash and cement because the strength gained by the column and ball depends upon the curing. Curing was done after 24 h of the drying by spraying normal water onto ball/column filter twice a day for 3 days.

### **3.3.3 RHA based filters for the tertiary treatment**

Ball and Column filters were used to treat wastewater from Pulp and Paper Industry after secondary treatment by passing 300 ml of wastewater samples through the filters of different composition. Different retention time was provided in the experiment and 50 ml aliquots were taken at different intervals for analysis. Treatment efficiency of samples at different duration was measured in terms of TSS and TDS. The estimation of chlorides was done with the treated samples. After all these experiments, the analysis on the repeatability and reuse of column and ball filters was done for a sample for 10 cycles.

### **3.3.4 Chloride estimation**

Chloride estimation was done using titrimetric method. 50 ml of the sample was taken and pH was adjusted neutral (7-8) with H<sub>2</sub>SO<sub>4</sub> and NaOH if it was not in the range (with the help of pH meter). After that, 2 drops of potassium chromate was added, while shaking it was titrated with N/35 (0.0141 N) silver nitrate. The appearance of brick red color was the end point of chloride ion.

**Formula used:**

$$\text{Chloride (mg/L)} = \frac{\text{ml sample} - \text{blank AgNO}_3 * 1000}{\text{ml sample}}$$

## CHAPTER 4

### RESULTS AND DISCUSSION

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The mixture of cement, sand and RHA was used to make ball filters and column filters which were used for wastewater purification. Different parameters were optimized to enhance the treatment efficiency for removal of TDS.

#### 4.1 Characterization of Industrial sample

Characterization of treated wastewater samples was carried out by measuring its physical characteristics like TSS, TDS, BOD, COD and chloride content.

Treated wastewater after anaerobic process followed by 2 stage activated sludge process collected from Pulp and Paper Industry near Ludhiana has high amount of TDS, TSS and chlorides.

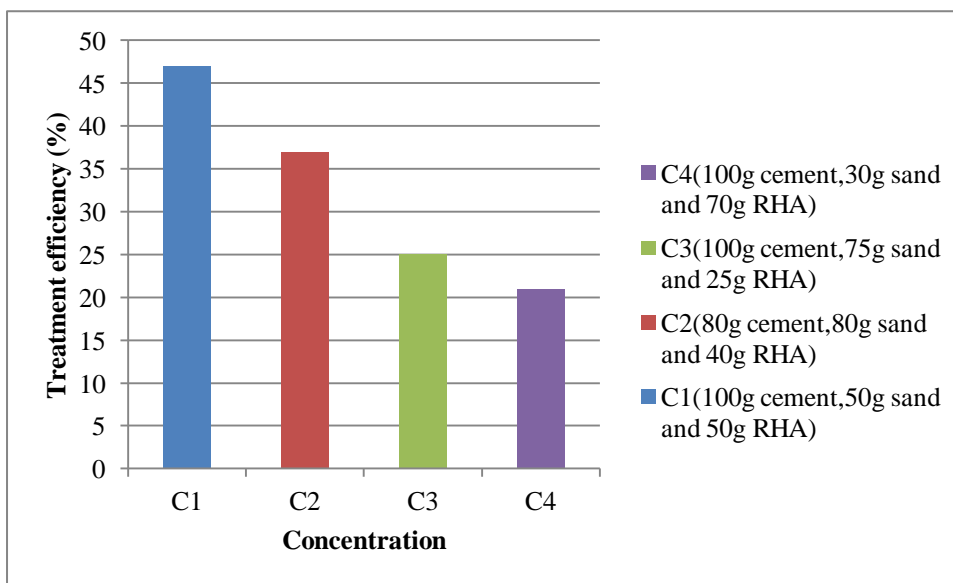
**Table 4.1:** Physical characteristics of industrial wastewater sample

S.No	Parameters	Value
1	TDS(ppm)	1820
2	TSS(mg/L)	640
3	Chloride(mg/L)	890
4	BOD(mg/L)	30
5	COD(mg/L)	225

#### 4.2 Effect of composition of RHA

The treatment efficiency in terms of TDS was calculated for different composition namely C1 (100g cement, 50g sand and 50g RHA), C2 (80g cement, 80g sand and 40g RHA), C3 (100g cement, 75g sand and 25g RHA) and C4 (100g cement, 30g sand and 70g RHA) after treatment with ball filter for 6h. Fig. 4.1 shows that C1 ball filter is efficient in TDS removal as compared to ball filters of other composition. The high concentration of silica in RHA is responsible for high capacity of adsorption in case of C1 ball filter. Further increasing the RHA concentration as in the C4 composition was not found to be suitable because of less strength and these balls lose

their shapes. So, further experiments were performed using the ball filter with C1 composition. Similar study was also done by [Ranu Pachuri., 2013] and results showed that C1 composition was more efficient as compared to others. Saha et al., (2002), performed similar experiment by varying different concentration of RHA for the removal of Arsenic from water. Faccini et al., (2012) varied different concentrations of RHA by 1%, 2%, 3%, 4% and 5% for the purification of biodiesel from waste frying oil. 4% RHA showed better results. The low amount of RHA results in lower number of active sites available for adsorption, so the treatment efficiency decreases.



**Fig 4.1:** Effect of composition of RHA

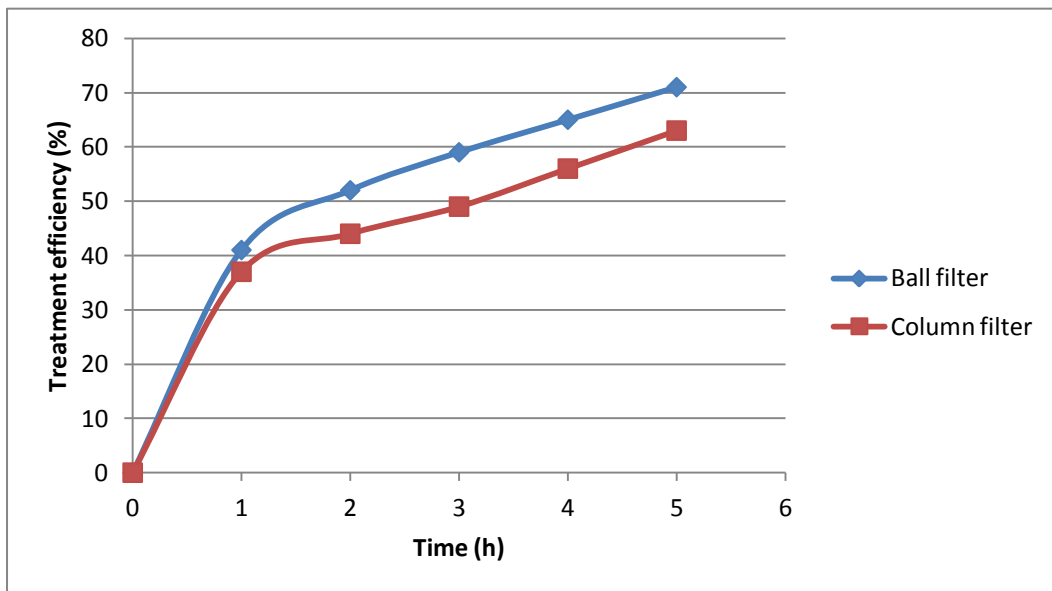
### 4.3 Assessment of the comparative treatment efficiency of the ball and column filter

The treated wastewater from the pulp and paper industry was subjected to RHA based treatment to assess the comparative treatment efficiency of the ball and column filter in terms of TSS and TDS.

#### 4.3.1 Treatment efficiency in terms of TSS

The results depicted in Fig 4.2 shows that the treatment efficiency in terms of TSS is reduced from 640 to 185.6 mg/L (71%) after 5h with ball filter and from 640 to 236.8

mg/L (63%) with column filter both having composition 100 g cement, 50 g sand and 50 g RHA. The ball filter was found to be more efficient as compared to column filter for TSS reduction. The treatment efficiency was found to be a function of time. It is evident from the results that the rate of adsorption is more in the first 1 h because of the availability of more number of active sites for adsorption and the rate of adsorption became less in the remaining time due to the decrease in the active site for adsorption. Similar study was also done for the reduction of TSS using modified RHA by the Suspension Burning Process [Agrilectric Research Company, 2009]. Treated RHA material was used for the treatment of plant scale wastewater and the results showed 99.8% reduction in TSS which was not possible without the addition of modified RHA product.

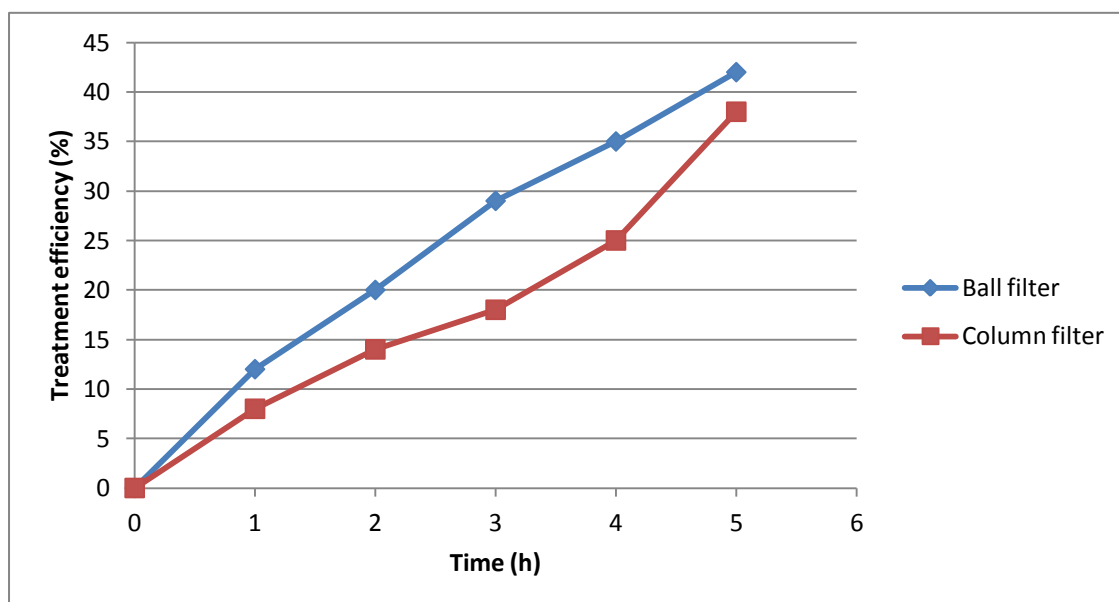


**Fig 4.2:** Comparison of treatment efficiency using column and ball filter in terms of TSS

#### 4.3.2 Treatment efficiency in terms of TDS

The comparative TDS removal of the ball and column filter was analyzed. Results presented in Fig 4.3 shows that TDS was reduced to 1050 ppm from 1820 ppm (42% reduction) for ball filter and for column filter it was reduced to 1130 ppm having composition 100 g cement, 50 g sand and 50 g RHA. It was observed from the results that at first TDS removal is more as more adsorbate is available for adsorption and with more time the active sites for adsorption goes on decreasing. It was analyzed that the ball filter is better in removing TDS as compared to column filter. Similar findings

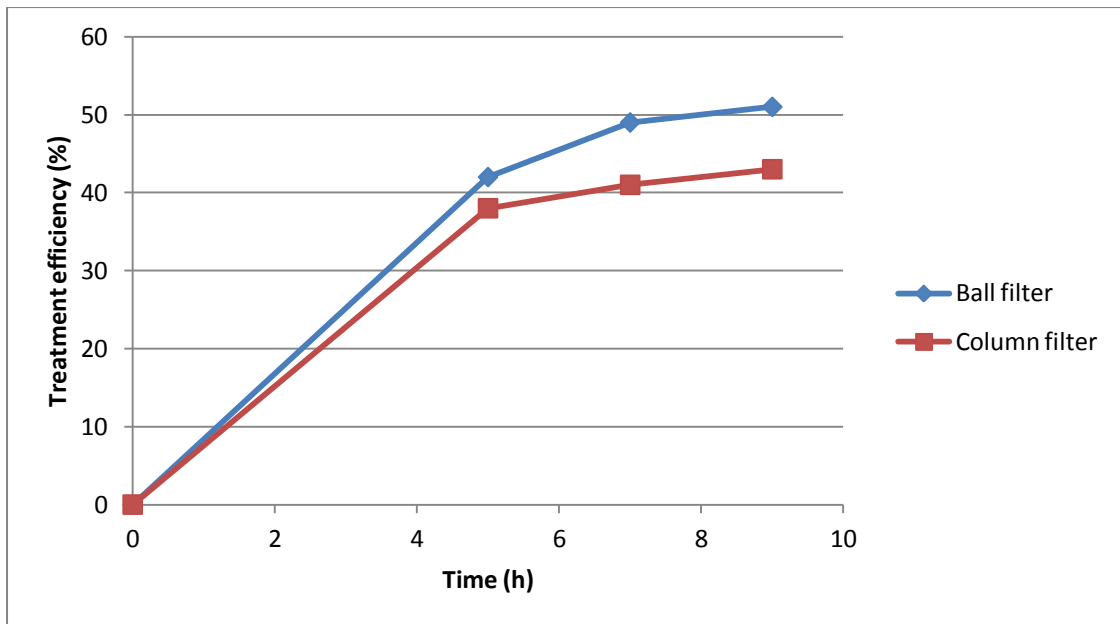
were observed for the reduction of TDS level using RHA as an adsorbent with sea water [Shiue et al., 2014]. Because of large surface area, high porosity and various functional groups RHA acts as an adsorbent. RHA was modified by phosphorylation and amination and the results analyzed shows 26.8% reduction.



**Fig 4.3:** Comparison of treatment efficiency using column and ball filter in terms of TDS

#### 4.4 Effect of contact time

In order to establish the optimum contact time required for obtaining the desired treatment efficiency in terms of TDS removal, experiments were conducted with C1 composition of ball and column filter for a period of 9 h. Fig. 4.4 depicts that the treatment efficiency in terms of TDS came out to be 51% reduced after 9 h that is from 1820 ppm to 892 ppm for ball filter and from 1820 ppm to 1040 ppm (43% reduction) in case of column filter. The results show that more reduction in TDS is during first 5 h and when the time increases the active sites for adsorption get filled up and create hindrance for the adsorbate to get adsorbed. Singha et al., (2012) reported similar experiment for the removal of lead ions from the aqueous solution by varying contact times.

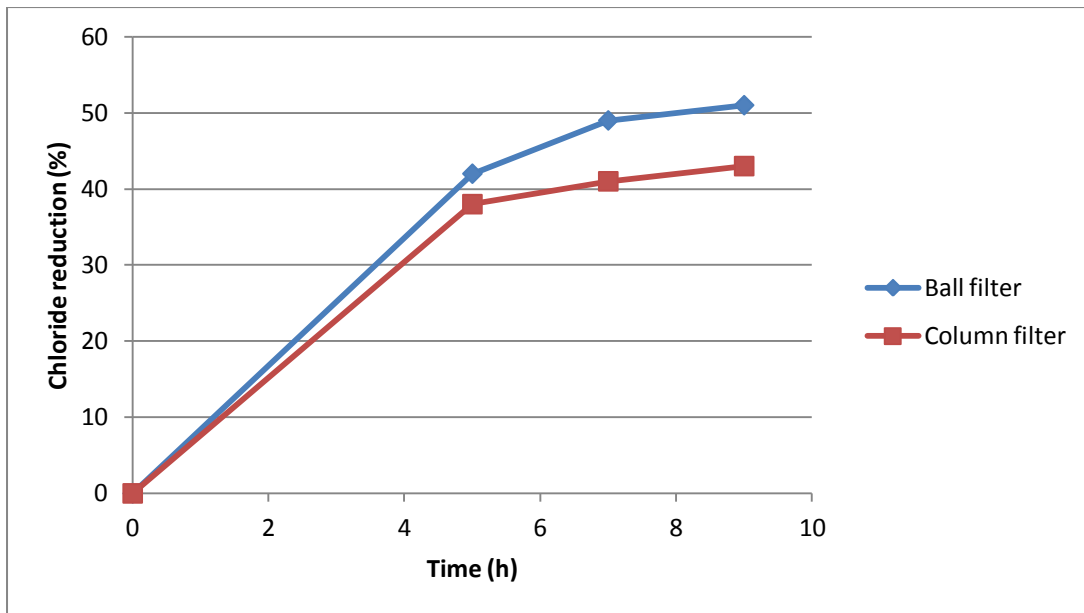


**Fig 4.4:** Effect of contact time on treatment efficiency (TDS)

The initial adsorption rate was rapid but when the adsorption reached at an equilibrium value the active sites for adsorption became saturated and rate of adsorption decreases.

#### 4.5 Estimation of chlorides

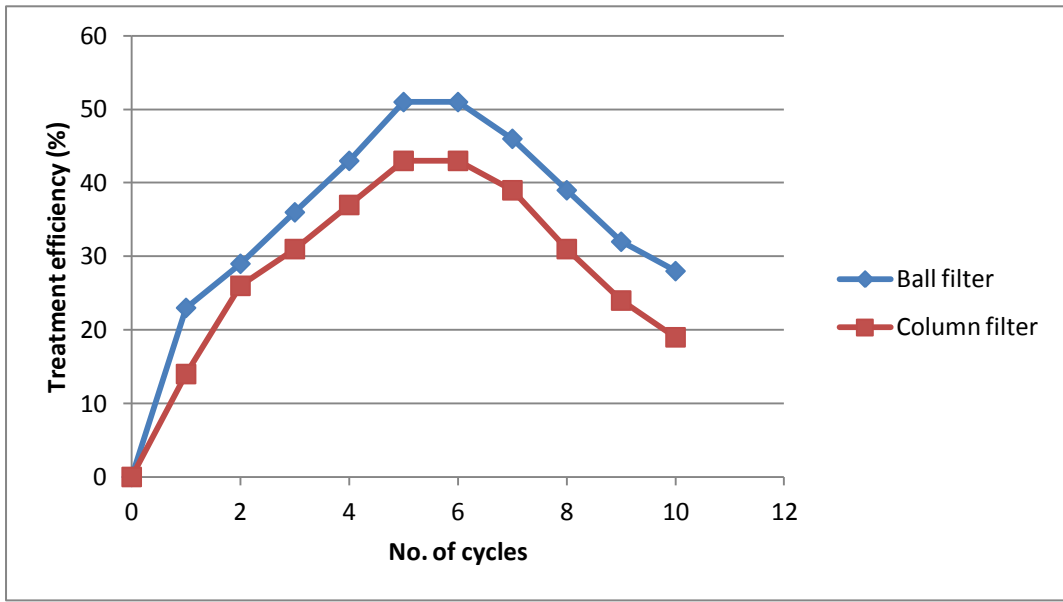
The estimation of chloride was done using ball filter and column filter having composition 100g cement, 50g cement and 50g RHA for wastewater sample. Fig. 4.5 shows considerable reduction in chloride content. The initial chloride content was 890 mg/L and after 9 h it was reduced to 436.1 mg/L (51% reduced) for ball filter and to 507.3 (43%) for column filter. The amount of chlorides present in the wastewater sample is initially higher so more chloride is available for adsorption which in turn increases its removal efficiency. Similar study for the reduction of chloride level was performed by replacing OPC with 30% RHA [Rajagopal et al., 2008].



**Fig 4.5:** Chloride estimation for wastewater sample using ball and column filter

#### 4.6 Filter reuse and repeatability

The experiment for repeatability of ball filter and column filter in the treatment of wastewater was conducted with C1 concentration. The repeatability of filter is done to avoid huge generation of solid mass. If the filters are not reused the quantity of generation of filters are more which results in high cost. As depicted from the results (Fig. 4.6) ball filters were found to be more efficient than column filters after 10 cycles of treatment because of the presence of high surface area in case of ball filters. The treatment efficiency is increased from 14 to 43% in column filters and 23 to 51% in ball filters after 5 days and thereafter, the efficiency decreased to 19 and 29% respectively after 10 repeated cycles. Similar study for the repeatability of cycles for the reduction of TDS was also done for sea water [Shiue et al., 2014]. The experiment was performed with 18 cycles and the results proved that at former adsorption was more and TDS was reduced to 26.8%.



**Fig 4.6:** The effect of repeatability of treatment on treatment efficiency

#### Conclusion

At the global level, the industrial wastewater treatment is one of the key aspects in reducing the water pollution. So, there is need for the treatment of wastewater especially from the pulp and paper industry because effluent from this industry contains high amount of BOD, COD, TSS, TDS and chloride contents. Even the treatment of effluent with anaerobic treatment followed by 2-stage Activated Sludge Process, the BOD could be reduced up to the permissible limits but TDS level still persists. Hence, there is a need to reduce TDS to avoid environmental degradation. The potential of using RHA as an adsorbent for the treatment of treated wastewater from pulp and paper industry has been explored. Ball filter and column filter was prepared using Rice Husk Ash, cement and sand. The high concentration of silica in RHA explains its high adsorption capacity. The results depicted that ball filters showed better treatment efficiency when compared to column filters. The optimization of filters was done by varying relative concentration of RHA, cement and sand and by varying contact time. The results showed that composition with 100 g cement, 50 g sand and 50 g RHA was optimum for reduction of TDS from treated wastewater. The retention time of 5 hr was found to be optimum because within this time there was significant decrease in TDS level to 42%. A considerable reduction in chloride level up to 51% was observed. The repeatability of ball and column filter was also determined for wastewater treatment by treating wastewater sample for 10 cycles and the results showed that ball filter was more efficient as compared to column filter because of the presence of large surface area for adsorption. In this way, RHA which causes major threat to environment utilized in a better way by the preparation of RHA based filters for the treatment of wastewater as a tertiary treatment step. Hence, RHA based filters can be used as a coupled treatment to existing biological treatment for industrial effluents.

### **Future prospects**

This study gives insight into the treatment of secondary stage treated industrial wastewater through RHA based filters. The used RHA based balls can be further used in construction purposes for concrete making and can be used in road surfacing. RHA based filters can be further used along with the nylon membrane for the treatment of wastewater. Additional modification can be done for the removal of heavy metals from wastewater. More study may be carried out for its use in the manufacture of fly ash and RHA mixed bricks.

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