



## Synthetic Grey Water Treatment Through FeCl<sub>3</sub>-Activated Carbon Obtained from Cotton Stalks and River Sand

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Received 23 September 2018; Accepted 03 January 2019

### Abstract

The research objective was to reclaim greywater through simple, easily available, and cost-effective methods. For this purpose, an activated charcoal was prepared from biomass (cotton stalk) through the pyrolysis process and sand collected from river Indus. Both materials were subjected to separate columns and applied as filters. Whereas, the efficiency of both materials as filter media was analyzed on the synthetic grey water (SGW). The formulation of synthetic greywater was a complicated process because the selection of ingredients and their amount should not exceed from the real grey water. So, for the presence of fecal contamination, a small amount (10 ml L<sup>-1</sup>) of settled sewage was added to the distilled water, while to mimic the organic load, several chemical products of technical grade were also added. The physicochemical and microbiological characteristics of this SGW were tested before and after treatment. The results show that both mediums (AC and river sand) were very effective in the greywater treatment. The removal efficiency for BOD and COD was up to 91.2%, and 70% respectively. Similarly, the removal measure for turbidity was 91.3%. While the pH showed that the synthetic grey water was alkaline in nature with a value of 10 because the washing detergents used during the preparation of SGW, but after passing through both filter columns, pH was observed in between 7 and 8 units. Furthermore, the removal value examined after passing SGW from both columns for total coliforms was 46.87 CFU/100 ml from 1500 CFU/100 ml.

*Keywords:* Synthetic Grey Water (SGW); Activated Carbon (AC); Cotton Stalk; Sand Filter.

## 1. Introduction

Dissolved pollutants (organic and inorganic) of water can be removed through an effective adsorption technique. Activated carbon (AC) is very familiar with all types of adsorbents due to high adsorption capacity. The adsorption frequency of activated charcoal relates to its great surface area, high pores distribution, and rapid grade of external reactivity [1]. Activated carbons are mostly extracted from raw carbon-rich resources in an oxygen-tight atmosphere through carbonization and followed by the activation process of the charcoaled material. The activation process can be carried out by means of chemical or physical activation [2]. Iron salts allow the preparation of activated carbons having a high specific surface area (965 m<sup>2</sup> g<sup>-1</sup>) and very small pores by activation at temperatures far below those used for activators generally described in the literature. Characterization studies have shown that the components present in the iron-impregnated material are completely pyrolyzed at a temperature of 280 °C [3]. Agro-industry produce millions of tons of lignocellulosic waste every year all over the world, additionally, usage of such wastes for activated carbon production will also help in reducing solid waste disposal issue [4]. Subsequently, an extensive range of agro wastes has been explored in Pakistan to produce activated carbon [5]. The cotton stalk is the fourth largest crop of Pakistan and

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 <https://dx.doi.org/10.28991/cej-2019-03091249>

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every year about 25100 million tons of cotton stalk waste produced in the country [6]. The proximate analysis of the shredded cotton stalk in terms of bulk density  $34.92 \text{ kg/m}^3$ , moisture content 13.63 %, volatile matter 74.52 %, ash content (4.95 %, fixed carbon 20.53 % and calorific value of cotton stalk biomass (3827 cal/g) respectively [7]. By utilizing cotton stalk for the preparation of activated carbon will provide an economical way for a beneficial product as well as it will also help the waste management issue. In addition, second media slow sand bed filter was also used to improve the efficiency of activated carbon treatment process because AC has some limitations on pathogen removal [8], while slow sand filters (SSF) can be easily obtained from local constituents and having an ability to remove 99% pathogens [9]. Moreover, water scarcity has developed a universal problem in current years. Now the water reclamation is the only way of preserving freshwater reservoirs. The interesting aspect is not to reclaim all household wastewaters but only grey waters (GW), this reclaimed water decreases potable water consumption by 29% to 47% [10]. Furthermore, Greywater is the whole domestic water arises from shower or bath, kitchen activities, and laundry purpose only excludes water containing excreta. It is the major point pollution source, which directly discharged from societies to the rivers and sea without any further treatment process [11]. There are several parameters such as quality and quantity of water supply, local practices, the culture of the area, and washing at the water source or home etc., influence the characteristics of greywater [12]. The most common method used for the physical treatment of greywater is recognized as filtration. While sedimentation or disinfection is a prior method applied to coarse filtration like sand bed filters. The highest rate for the removal of dissolved and suspended solids was obtained in membrane filtration but it had some limitations on organic pollutants [13]. The reason to select synthetic grey water (SGW) for the assessment of  $\text{FeCl}_3$ -Activated carbon efficiency instead of real grey water (RGW), was the variation in the characteristics of real greywater [14] and recipe followed in the current study was a part of a previous research [15]. The objectives of this research were to study the effects of the salt based activating agent (Iron Chloride) in the production of activated carbon and to analyze its efficiency on the synthetic greywater. The temperature ( $400^\circ\text{C}$ ) and the volume of activating agent (4<sup>th</sup> part of charcoal material) used in the activation process were at its minimum level, but the adsorbent ( $\text{FeCl}_3$ -AC) obtained showed maximum level of efficiency during the treatment of synthetic greywater (SGW).

## 2. Materials and Methods

### 2.1. Collection, Modification and Gradation Analysis of River Sand

The Indus Delta River is an arid climate formed by high discharges from river sediments (about 400 million metric tons of sediment per year), moderate tidal range (2.62 m), and extremely high wave energy ( $14 \times 10^7 \text{ erg/s}$ ). Coast and average wave height of 1.84 meters, strong wind monsoon in the southwestern part of summer and northeastern winter. The delta of the rather rough grain thus produced has a lobed shape, lacks dense vegetation, and is dissected by many tidal channels with mangroves in the lower delta plain. A prominent distribution in sedimentary basins, and a system dominated by waves, with little distribution along the coast unless it is characterized by sediments and sand dunes [16]. The sand used in this study was obtained from the lower delta plain, near Hyderabad, Sindh, Pakistan to fabricate the sand filter column. The obtained sand was washed thoroughly after collection in order to remove dirt and other impurities. Furthermore, after washing process, the residual quantity of sand was dried through an oven for 1 day at temperature of  $110^\circ\text{C}$ . The gradation analysis by wet sieving method used to analyze the grain size of sand [17].

### 2.2. Preparation and Characterization of $\text{FeCl}_3$ -Activated Carbon

The  $\text{FeCl}_3$  Activated charcoal was obtained from biomass (cotton stalk) through the pyrolysis process. The first stage was carbonization of biomass at  $550^\circ\text{C}$  for 1 hour. After carbonization charcoal was active with the help of an activating agent (iron III chloride or  $\text{FeCl}_3$ ) at  $400^\circ\text{C}$ . The mass ratio between the activating agent and charcoal was 1:4 respectively. Alert considerations regarding particle size distribution can provide substantial operational assistance. This allows the fixed contaminants to adsorb to the surface of the adsorbent. Representative particle size values for CA samples were achieved during the test. Use ASTM D-2862-97 (reapproved in 2004) [18].

### 2.3. Preparation of Synthetic Grey Water

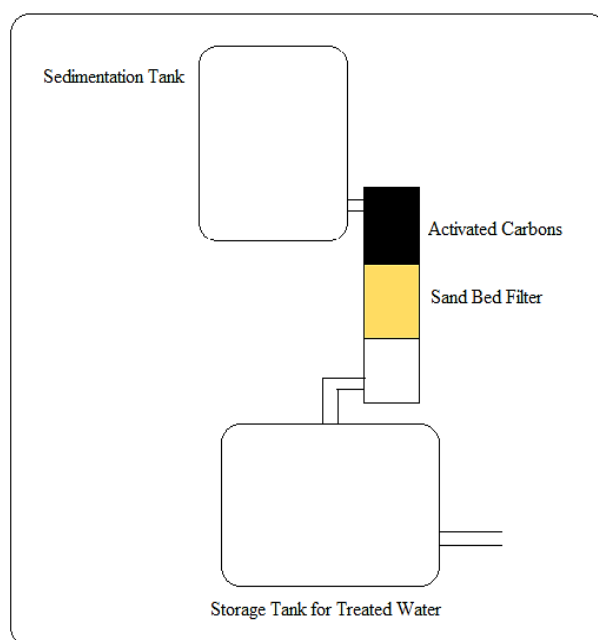
Greywater is a type of wastewater used for various activities like washing purpose, kitchen, and showers etc. excluding excreta arise from toilets. Hair's traces food constituents, household products and dirt can be observed in the composition of greywater. It may even appear unclean, but at the same time in some cases it can be a valuable for plants. The availability of nutritious elements in greywater released from homes are the basic cause of pollution, and these nutritious elements can be a productive fertilizer for plants [19]. In this study, the synthetic greywater was prepared from components usually present in graywater. The constituents including 85 mg/L of dextrin, 75 mg/L of ammonium chloride, 70 mg/L of yeast extract, 55 mg/L of each starchy food and washing soda, 30 mg/L of washing powder, 11.5 mg/L of sodium dihydrogen phosphate, 4.5 mg/L of arcanite, 10 ml/L of settled sewage and 0.1 ml/L of each shampoo and oil as shown in Table 1. The initial characterization of synthetic greywater was analyzed on 5 liters, while for the final application of iron chloride based-AC in combination with sand bed filter 30 liters of synthetic greywater was prepared. The recipe of synthetic greywater used in this study to analyze the efficiency  $\text{FeCl}_3$ -AC and sand bed filter was nearly of the same nature as real grey water [20].

**Table 1. The number of constituents used in the preparation of synthetic greywater**

Ingredients	Quantity	Formula
Dextrin	85 mg L <sup>-1</sup>	(C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> ) <sub>n</sub>
Ammonium Chloride	75 mg L <sup>-1</sup>	NH <sub>4</sub> Cl
Yeast extract	70 mg L <sup>-1</sup>	Nil
Flour (Starchy food)	55 mg L <sup>-1</sup>	-
Washing Soda	55 mg L <sup>-1</sup>	Na <sub>2</sub> CO <sub>3</sub>
Washing powder	30 mg L <sup>-1</sup>	Nil
Sodium di-hydrogen phosphate	11.5 mg L <sup>-1</sup>	NaH <sub>2</sub> PO <sub>4</sub>
Arcanite	4.5 mg L <sup>-1</sup>	K <sub>2</sub> SO <sub>4</sub>
Sewage	10 ml L <sup>-1</sup>	Nil
Shampoo	0.1 ml L <sup>-1</sup>	Nil
Cooking oil	0.1 ml L <sup>-1</sup>	Nil

#### 2.4. Fabrication of FeCl<sub>3</sub>-Activated Carbon Filter and Slow Sand Bed Filter Columns

In this regard two columns of transparent material having length 40 cm and 8 cm diameter were installed in vertical series, holding AC filter on the upside and sand filter on the downside. A locally available fabric was also used at the lower ends of both columns to separate the mixing of materials filled and hold them from the drain. Furthermore, both columns were connected to a sedimentation tank having a capacity of 30 liters. The sedimentation time for SGW was 3 hours before it flows towards filters. AC and sand columns were nearly filled up to 25 cm [21].

**Figure 1. Schematic Representation of Laboratory Scale Set Up**

#### 2.5. Characterization of Synthetic Grey Water

The structure of the synthetic grey water was reconstructed in the present study that is shown in Table 3. This is largely composed of household products including organic and inorganic pollutants from bathrooms, kitchen, and laundry. The characteristics of synthetic greywater were observed in this study were biochemical oxygen demand (BOD), chemical oxygen demand (COD), Turbidity, the potential of hydrogen (pH), and Total coliforms [22]. Aerobic bacteria utilize free oxygen for the decomposition of organic food present in wastewater and BOD is a valuation of that organic food. The consumptions of DO in wastewater is directly proportional to the amount of organic food presence. This test investigates the oxygen usage by bacteria needed them for stabilization of organic matter under measured environments. The BOD of the sample was measured through the titration method. In this assay DO initial was measured on the first day, and after five days DO final was measured. The difference between DO<sub>1</sub> and DO<sub>5</sub> was the BOD<sub>5</sub>. COD is the measure of the whole quantity of oxygen required for biological life and the decomposition of organic food. Chemical oxygen demand (COD) is basically the valuation of oxygen needed for the oxidation process of organic material into CO<sub>2</sub> and H<sub>2</sub>O. The method used for the COD was closed reflex calorimetric method. Turbidity is the

examination of comparative clearness of liquid. Turbidity is an appearance of the light scattered when it passes through the water. More the light dispersed, higher will be the turbidity value. Several impurities like clay, silt, algae, microscopic organisms, fine particles of organic and inorganic compounds. The equipment used to measure the amount of turbidity in synthetic greywater before and after the application of filter mediums was turbidity meter. The degree of acidity and basicity of a solution is known as pH. The value of the pH scale is divided into 14 figures, while 7 is the neutral point represent water. Above 7 all are alkaline and below 7 all are acidic in nature. The parameter was measured through pH meter. Total coliform calculation suggests an overall sign of the disorder of water. The more the value, the higher the contamination level of water. While total coliforms can originate from sources other than feces. The method used to analyse the measuring values of total coliforms at an initial and final stages in synthetic greywater was total coliforms colonies technique through membrane filtration.

**2.6. Flowchart**

Flowchart of the research methodology has been presented as below:

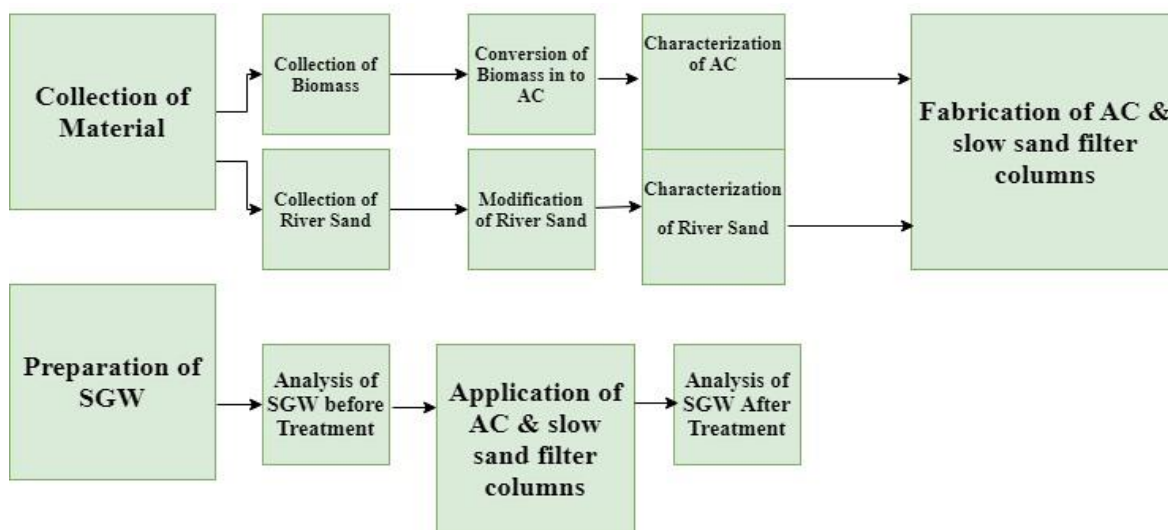


Figure 2. Flow Chart of the Methodology

**3. Results**

**3.1. Gradation Analysis of River Sand by Wet Sieving**

The sieve analysis of collected sand identify that the distribution of particle size for river sand was 0.075 mm (see Table 2). While the basic purpose to use sand filter column as an additional filter medium with activated carbon was to treat the amount of total coliforms may appears in synthetic greywater. The particle size of sand was the most prominent parameter in this consideration. A significant interaction between filter depth and the flow-through rate in the removal of E. coli ( $p < 0.05$ ) was observed which means that increasing the depth of the sand filter while slowing the filtration rate improved efficiency in E. coli removal of the raw water [23].

Table 2. The characterization of river sand through gradation analysis

Sieve #	Sieve Size	Retained in gm	% Retained	% Passing
# 4	4.75	00	00	100
# 10	2.0	00	00	100
# 40	0.425	0.86	00	99.91
# 200	0.075	957.18	97.46	2.45
Pan	---	5.54		

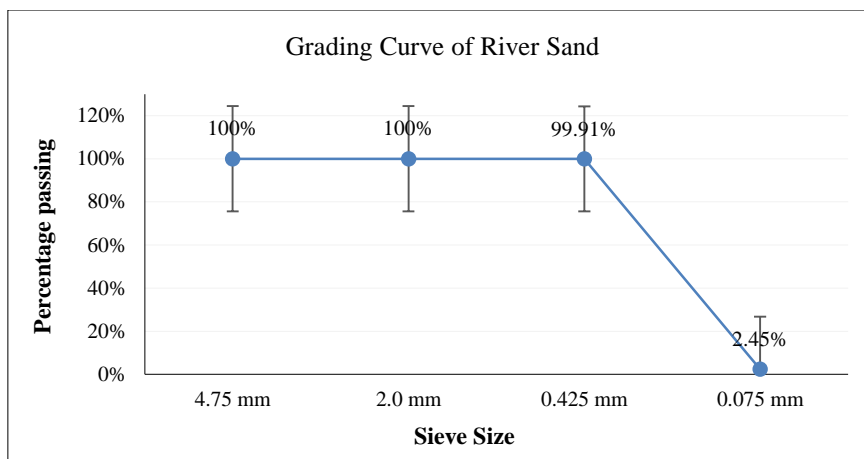


Figure 3. Gradation Analysis of River Sand

### 3.2. Determination of Particle Size of Powdered Activated Carbon

A particle size distribution caution can provide substantial operational assistance. This allows you to fix the adsorption of dirt on the surface of the adsorbents. During testing, typical particle sizes of the activated carbon was achieved. Though ASTM D-2862-97 (approved in 2004), 50 g of AC were investigated, while the main part of 50 g was stored in the opening of the screen 1.44 mm, moreover, the screen was opened 3.57 and 2.03 mm has 11.2 and 12.4 g, respectively, as shown in Table 3. Activated carbon with a particle size of less than about 0.4 mm is considered to be a poor filtering medium and is processed for sale in powder form [24].

Table 3. Determination of meshed particle size of Activated charcoal

U.S.S. Sieve #	Mean Opening (mm)	Retained %	Passing %	Weighted average
+4	5.74	8	92	4
4 × 8	3.57	22.4	69.6	11.2
8 × 12	2.03	24.8	44.8	12.4
12 × 16	1.44	32.6	12.2	16.3
16 × 20	1.02	10.4	1.8	5.2
18 × 20	0.92	1.8	00	0.9
Pan		100 %		50

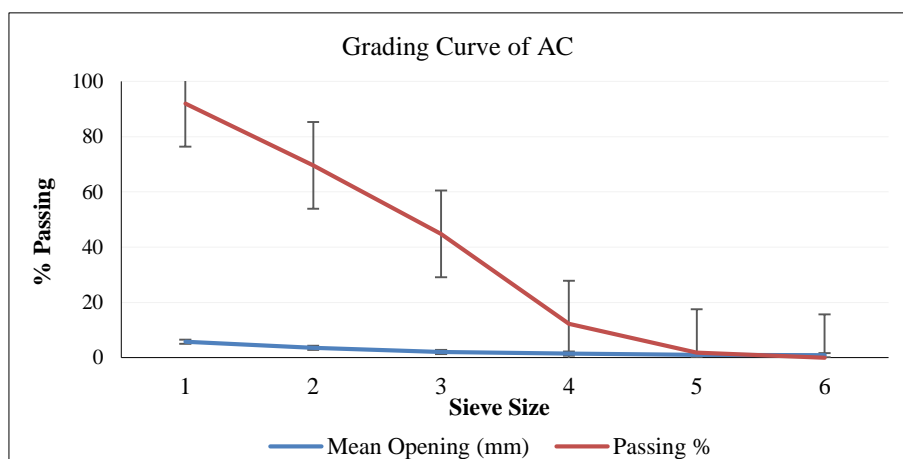


Figure 4. The particle size of Activated Carbon

### 3.3. Analysis of Synthetic Grey Water before and After Treatment with FeCl<sub>3</sub>-AC and Slow Sand Bed Filter

A noticeable effect in quality of distilled water was examined after the addition of ingredients used to prepare the synthetic greywater. The synthetic greywater was tested several times at an initial and final stages in order to obtain the accurate results. The sedimentation time provided to synthetic greywater was 3 hours. After sedimentation effluent

synthetic greywater flows to wards activated carbon and sand filter in a continuous stream. The SGW retention time for any column is zero and the two filters are proven to be efficiencies up to 60 liters. In addition, continuous changes were observed in all SGW parameters due to the addition of 10 ml/l of precipitated Sewage 2. The weight of the ingredients used to make the SGW is slightly different. Significantly changed the value of BOD<sub>5</sub>. The SGW values at an initial stage before the treatment were 15.2-40.6 mg/liter, and the results after applying the filter were 1.06-3.26, as shown in Table 4. The BOD<sub>5</sub> removal rate of activated carbon and sand bed filter can be 52.41 to 53.36 mg/liter [22].

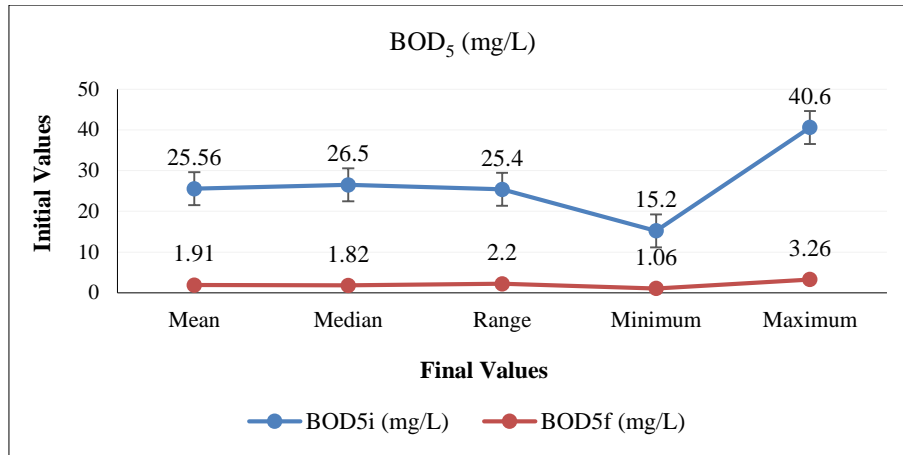


Figure 5. BOD<sub>5</sub> of synthetic grey water before and after treatment

The COD measures the value of the synthetic grey water before the treatment is above the BOD because many organic materials are chemically oxidized rather than biological. Before using the river sand filter and activated carbon, the COD value was 48-81 mg/l, and after application, the value was between 21.4-23.8 cm. Table 4. A pervious study noted that the COD reduction values were almost equal, the percentage exclusion rate was 65%, and the percentage reduction ranged from 54% to 70% [21].

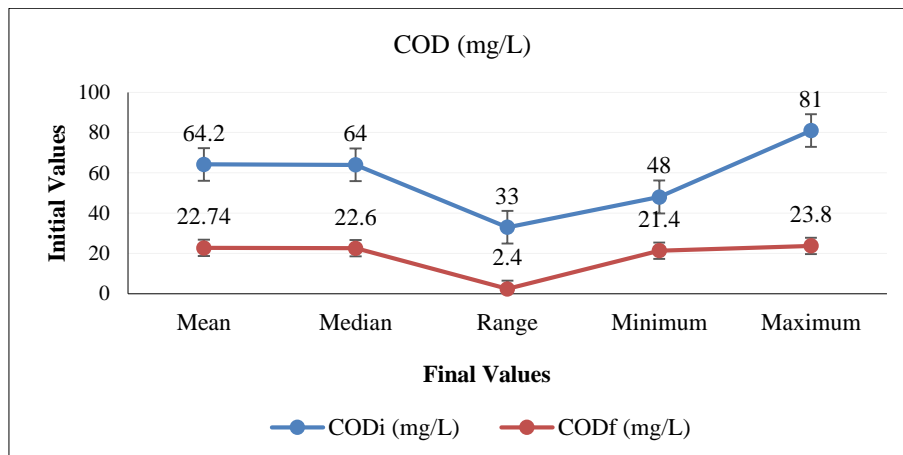


Figure 6. COD of synthetic grey water before and after treatment

Greywater can be composed of several contaminants which can affect through various ways. Turbidity is the parameter used to identify the presence of these complex nature pollutants. The turbidity of the finished grey water changes during the early stages prior to treatment. The measured value before treatment is 28.3-36.8 NTU, and the measured value after filtration is 1.93-3.2. A study showed that the use of filter sand and activated carbon keeps the turbidity below 4 NTU [13].

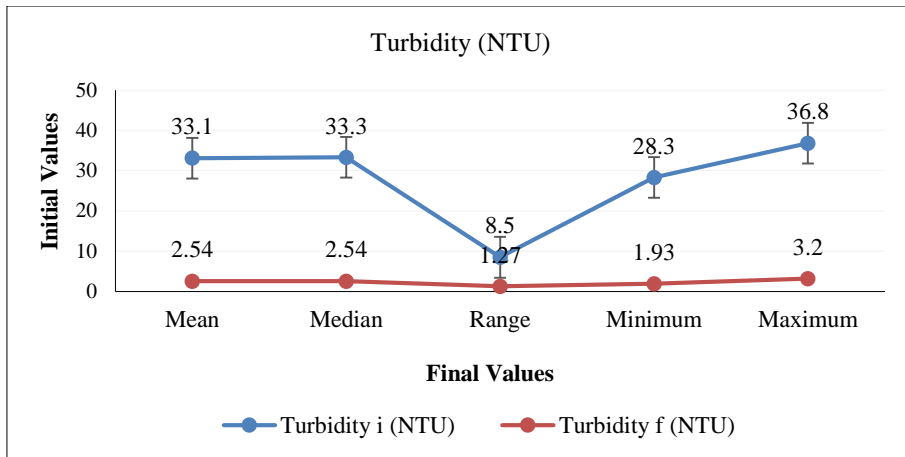


Figure 7. The turbidity of synthetic grey water before and after treatment

Due to the detergent used to make it, the properties of synthetic grey water are very strong alkaline. The pH of grey water (5-10.9) is more alkaline than the common household wastewater (5.9-7.7) [25]. The initial pH was observed between 9.2 and 10 and after filtration with FeCl<sub>3</sub>-Based activated carbon in combination with sand filter, the range was 7 to 8. The pH results were consistent with previous studies between 7.2 and 7.9 after treatment [26].

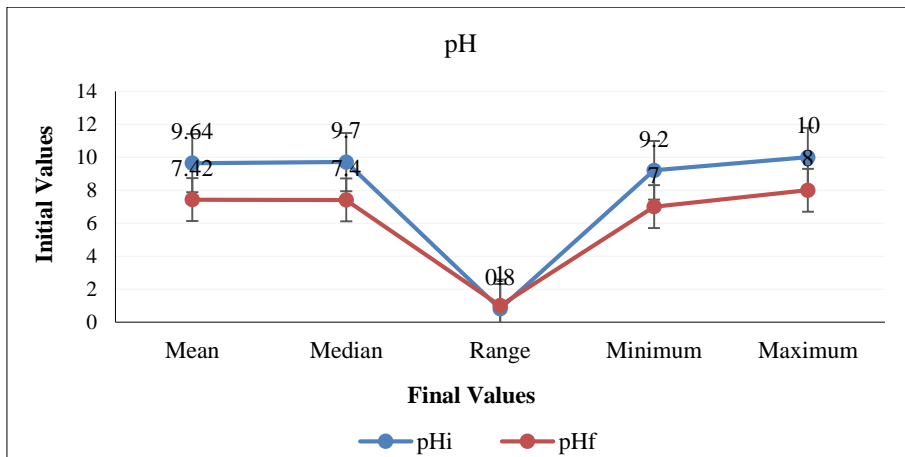


Figure 8. pH of synthetic grey water before and after treatment

Literature identified that the total coliforms in clear water ranging from 500 to  $2.4 \times 10^7$  CFU/100 ml [27]. In this research, the initial results of total coliforms in synthetic grey water was as high as 900-1500 CFU/100 ml, and even the amount of precipitated sewage mixed to SGW was only 10 ml/l, but the effect was significant. After applying the both filters i.e. iron chloride based Ac and sand filter, the final measuring values range were 6.32 to 46.87 CFU/100 ml, which clearly excludes the total SGW.

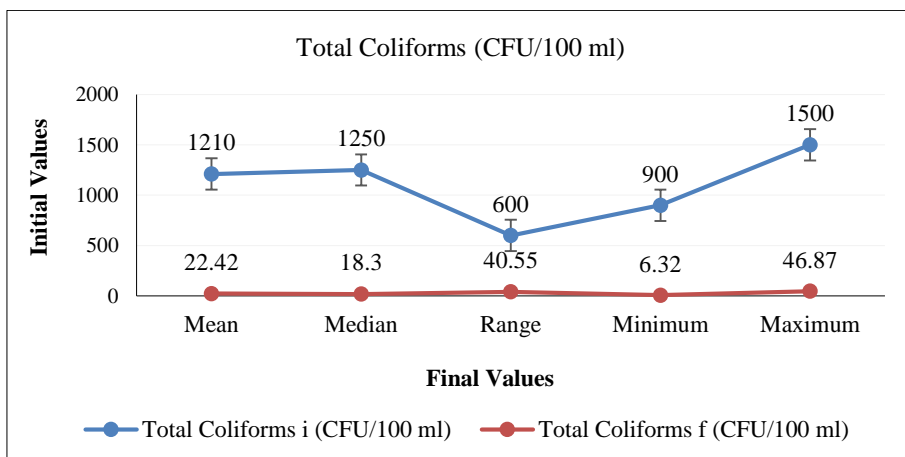
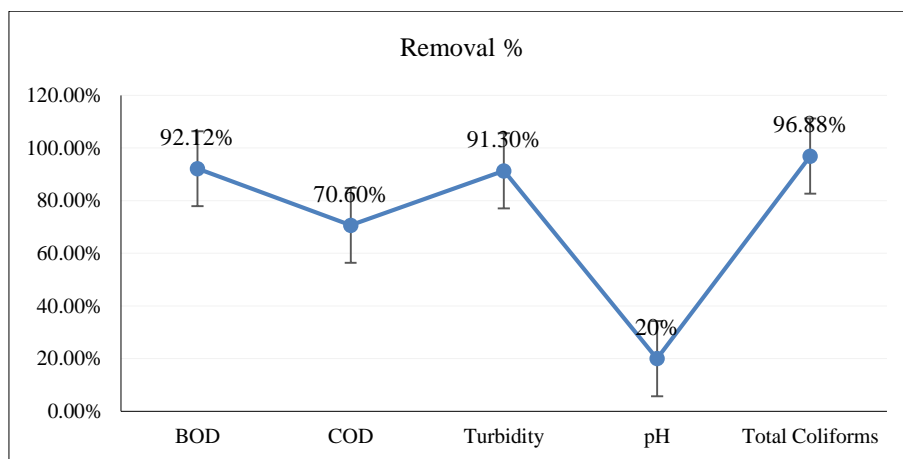


Figure 9. Total Coliforms of synthetic grey water before and after treatment

**Table 4. Characteristics of greywater before and after the treatment**

Parameters	Before Treatment (i)					After Treatment (f)				
	BOD <sub>5</sub> (mg/L)	COD (mg/L)	Turbidity (NTU)	pH	Total Coliforms (CFU/100 ml)	BOD <sub>5</sub> (mg/L)	COD (mg/L)	Turbidity (NTU)	pH	Total Coliforms (CFU/100 ml)
Mean	25.56	64.2	33.1	9.64	1210	1.91	22.74	2.54	7.42	22.42
Median	26.5	64	33.3	9.7	1250	1.82	22.6	2.54	7.4	18.3
Range	25.4	33	8.5	0.8	600	2.2	2.4	1.27	1	40.55
Minimum	15.2	48	28.3	9.2	900	1.06	21.4	1.93	7	6.32
Maximum	40.6	81	36.8	10	1500	3.26	23.8	3.2	8	46.87

**Figure 5. The removal percentage of FeCl<sub>3</sub>-AC and Slow Sand Bed Filter**

#### 4. Conclusion

Synthetic grey water was synthesized, and the effectiveness of AC in local agricultural waste and the effect of sand filter combined with AC in SGW were analyzed. The ingredients used to make the SGW are the main components of domestic products. The research analyzed that the combined filters (AC and river sand) were very operational in treating grey water. Compared with BOD, synthetic grey water has a higher COD value, but the combined elimination of the two filters is more efficient than COD. In addition, activated carbon and river sand have proven to be very effective at the pH of the synthetic grey water used for treatment, which is highly alkaline. During the treatment, the average removal efficiency of turbidity was analyzed to be 91.3%. Although the overall elimination rate of E. coli was also high, the average percentage was 97.9%. The total efficiency of activated carbon and river sand filters based on FeCl<sub>3</sub> is better, but the retention times of both columns are zero, and the retention time of SGW in both columns is studied to provide better results

#### 5. Acknowledgment

Authors would like to thank Dr. Khan Muhammad Brohi Dean faculty Architecture and Civil Mehran University of Engineering and Technology Jamshoro, Sindh, Pakistan for the direct involvement during the initial laboratory trials and continuous trial.

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