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Synthesis of Activated Carbon Via Sulphuric Acid and Iron Chloride and its Potential Application Synthetic Grey Water in Combination with Sand Bed Filter

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Abstract: Biomass is an economical and wide available source for the production of activated carbon (AC). In this study, the effect of H_2SO_4 and $FeCl_3$ in the production of AC from the cotton stalk (CS) was analyzed. Various techniques used to examine the characteristics of both prepared activated carbons (ACs). The techniques include scanning electron microscopy (SEM) which identified the surface modification, including its micro and macropores. Furthermore, crystalline modifications were measured through X-ray diffractometry (XRD) techniques. In last, Fouriertransforms infrared spectra (FTIR) method identified the presence of different functional groups on the surfaces of ACs. The major peaks during FTIR analysis of H_2SO_4-AC were observed in the range of 753.00, 877.52, 1180.09, 1557.42, 1574.55, 1695.10, 2110.76, 2320.81, 2359.11, 3360.30 cm^{-1}. Similarly, during the analysis of $FeCl_3$-AC,the major peaks were noticed at 877, 1156, 1557, 1568, 1683, 1695, 1916, 1991, 2112, and 2358 cm^{-1}. The removal efficiency of prepared ACs were analyzed on synthetic grey water (SGW) in combination with a sand filter. The pH value before treatment was 10 but after treatment with $FeCl_3$-AC and sand filter the value was up to 8, and 7.2 with H_2SO_4-AC and sand filter. Similarly,turbidity before treatment was 36.8 NTU but after filtration with $FeCl_3$-AC and H_2SO_4-AC in combination with the sand filter the values were 3.2 NTU and 1.6 NTU respectively. The BOD_5 of SGW before treatment was 40.6, and after the application of $FeCl_3$-AC in combination with sand filters the final value was 3.26 mg/l and H_2SO_4-AC in combination with sand filters, the final values was2 mg/l. The initial measuring value of SGW for electrical conductivity (EC) was 0.40 $uScm^{-1}$. After the treatment with ACs, a minor increase in the values of EC was examined, which was 1.6 µS/cm for $FeCl_3$-based AC combine with sand bed filter and 1.65 µS/cm for H_2SO_4-based AC in combination with a sand filter. The amount of total suspended solids (TSS) at an initial stage was 34 mg/L in SGW, while it was 0.68 mg/L after the application of H_2SO_4-based AC in combination with a sand filter and 1.61 mg/L after the application of $FeCl_3$-based AC combine with sand bed filter. Oil and fates were also observed with the initial value of 54 mg/L, which was 1.41 mg/l and 3.1 mg/l after the individual application of

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Sulphuric acid based AC and iron chloride based AC combined with sand respectively. In addition, the initial value of COD was 81 mg/L and after application of $FeCl_3$-AC in combination with the sandfilter, the value was up to 5.2. On another hand, the COD after treatment with H_2SO_4-based AC and sand bed filter was 2.3 mg/l. The value of total coliforms in synthetic grey water was 1500 CFU/100 ml. After the application of $FeCl_3$-AC and H_2SO_4-AC in combination with the sandfilter, the final measuring values were 6.32 CFU/100 ml and 4.21 CFU/100 ml respectively.

Keywords: *Activated carbon (AC), Activating agent, Bio-mass waste, Synthetic grey water (SGW)), Sand filter, Adsorbent.*

INTRODUCTION

The basic materials used in the production of AC are hard coal, coconut shells, and wood, but due to the absence of such material in different countries, the researchers are utilizing several types of other biomasses to replace them [1].The cheap materials used in its preparation of AC can be waste rubber tire, bamboo, nut shells, used tea and even bacterial cellulose [2-6], and all suggest materials have the capacity to reduce the production cost of material needed. The cotton stalk is the fourth largest crop of Pakistan and every year about 25100 million tons of cotton stalk waste produced in the country [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, after finalizing bio-mass the next most important step in chemical activation is the selection of an appropriate activating agent. Some are the popular activating agents are KOH, H_3PO_4, H_2SO_4, NaOH, $CaCl_2$, $ZnCl_2$ [8-13] etc. All these agents have unique properties and influencing behavior in the activation process [14-15]. Iron salts allow the preparation of materials having a high specific surface area (965 m^2 g^{-1}) 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 [16].When a sulfur-containing surface group is introduced, the removal of the molecule is enhanced due to the greater affinity between the reactant and the catalyst surface, which facilitates the production of very reactive hydroxyl radicals in the vicinity of the contaminant [17].Furthermore, some of the major applications of AC are storage of hydrogen and catalytic agent [18], transporting drugs [19], purifier and filter [20], and super-capacitor [21]. These all applications show a great adaptive behavior of an adsorbent and clarify its organized inert pore distribution which contains numerous functional groups [22]. Furthermore, Greywater (GW) is the whole domestic water arises from shower or bath, kitchen activities, and laundry purpose only excludes water containing excreta, but 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 GW [23]. The most common method used for the physical treatment of GW 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 by membrane filtration but it had some limitations on organic pollutants [24]. The reason to select SGW for the assessment of $FeCl_3$-Activated carbon efficiency instead of real grey water (RGW) was the variation in the characteristics of RGW [25].The objectives of this research were to study the effects of the salt based activating agent (Iron Chloride) and acid-based activating (Sulphuric acid) in the production of activated carbon and to analyze their efficiency on the synthetic greywater. The temperature (400^0C) and the volume of activating agent (4^{th} part of charcoal material) used in the activation process were at its minimum level, but the adsorbents (H_2SO_4-AC and $FeCl_3$-AC) obtained showed maximum level of efficiency during the treatment of synthetic greywater (SGW).

MATERIAL AND METHODS

Preparation and Characterization of Activated Carbons

The CS was carbonized at 550^0C for 1 hour in a furnace, and after carbonization 5 gm of each activating agent ($FeCl_3$and H_2SO_4) diluted in 100 ml distilled water were mixed up with 20 gm of charcoal and dried in an oven at 110^0C for 24 hours to eliminatethe moisture. In the last stage, the materials containing activating agents were again pyrolyzed at 400^0C for 1 hour. After pyrolysis, the activated carbons were cooled at room temperature and washed with distilled water to adjust the pH of activated carbons up to 7-8.5 [26].According to literature the pH range of activated in between 6 to 8 is suitable for sugar decolonization, water treatment etc. [27].The prepared ACs were characterized by various methods including Fourier Transform Infrared (FTIR) spectroscopy technique, Scanning Electron Microscope (SEM) and X-ray Diffractometry (XRD). CS is an agriculture waste which contains a high amount of lignin, cellulose, and hemicellulose. After the pyrolysis process, these components break down into various functional groups. FTIR was used to characterize the functional groups arises on the surface of activated carbon after pyrolysis. The machine model used for FTIR analysis was thermonicolet 5700 having movable KBR pin and deuterated triglycine sulfate sensor (DTGS). While the software for this machine was a commercial based IR spectrum analyzer OMNIC and its spectra recording range was 500-4000 cm^{-1} having a resolution of 4 cm^{-1}. SEM technique is a machine based technique, which analyzes the porosity of a material. After the pyrolysis of biomass (CS) at a high temperature, the presence of micro and macro pores was examined through SEM, whereas, the machine used in this method was a scanning electron microscope manufactured by Jeol Tokoyo Japan. XRD characterization is a non-destructive qualitative analyzation of any material. It measures the changes occurred in the crystalline structure of any material including its bulk composition, and homogenization. The diffractogram of both ACs obtained during this study from CS were analyzed on a phillippe x-ray diffractometry machine. The enetrning speed of graph in this machine was 2 $cm.m^{-1}$, while the XRD pattern was 2’2θ-20’2θ.

Fabrication of Activated Carbons and Sandbed 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 for each type of AC separately including $FeCl_3$-AC and H_2SO_4-AC. 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. While the sand used in this study was to characterize through gradation analysis method (see fig 1).

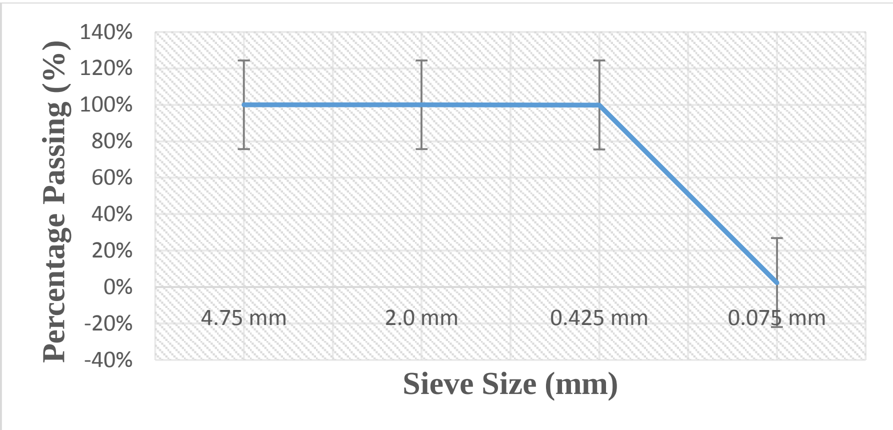

Fig. 1: Grading curve for filter media (Sand)

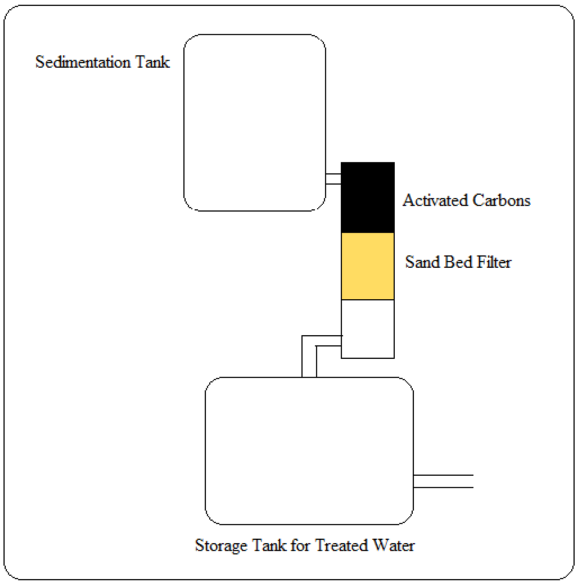

Figure 2: Schematic Representation of Laboratory Scale Set Up

Preparation and Characterization of Synthetic Grey Water (SGW)

The elementary source for the pollution of greywater is the number of household products released in it [28]. The composition of synthetic greywater (SGW) used in this study was almost of the same nature as released from homes. The chemical composition of SGW include starchy food (Dextrin=85mg/L), (flour=55), cleaning agents (Ammonium chloride=75mg/L), (Washing Soda=55mg/L), (Washing Powder=30mg/L), (Sodium Di-Hydrogen Phosphate=11.5mg/L), (Shampoo=0.1ml/L), baking material (Yeast=70mg/L), food ingredients (cooking oil=0.1ml/L), indicator of fecal contamination (sewage=10ml/L) and arcanite=4.5 mg/L as shown in table 1 [29]. After the preparation SGW, the next step was to characterize its quality before and after the applications of filter mediums. The parameters of SGW studied during this research work were pH, Turbidity, Biochemical Oxygen Demand (BOD_5), Electrical conductivity (EC), Oil and Fates, Total Suspended Solids (TSS), Chemical Oxygen Demand (COD), and Total Coliforms.

The methods and equipment used for pH, turbidity, BOD_5, COD,and total coliforms parameters were pH meter, turbidity meter, titration method, closes reflex method, and total coliform colonies through membrane method respectively. Electrical conductivity (EC) is one of the rapid methods to analyze the quality of water.

The HACH 8163 standard method was used for this meter while the equipment used was the conductivity meter. Total suspended solids (TSS)are the number of dry elements trapped by a filter. The method used to analyze TSS of synthetic grey water was Imhoff cone method, while the sample amount was 1000 ml.

Oil and fates of any effluent indicate the presence of miscellaneous components which are highly soluble in an organic solvent. The oil and fates characterization were examined through floatable oil and grease method in a separatory funnel apparatus. The sample volume taken for this test was 350 ml and 35 ml of trichloroethylene.

Table 1: Chemical Composition and Concentration of materials used in the preparation of synthetic greywater (SGW)

Ingredients	Quantity	Formula
Cellulose (dextrin)	85 mg/L	$(C_6H_{10}O_5)$ n
Ammonium Chloride	75 mg/L	NH_4Cl
Yeast	70 mg/L	-
Flour	55 mg/L	-
Washing soda	55 mg/L	Na_2CO_3
Washing Powder	30 mg/L	-
Sodium dihydrogen phosphate	11.5 mg/L	NaH_2PO_4
Arcanite	4.5 mg/L	K_2SO_4
Sewage	10 ml/L	-
Shampoo	0.1 ml/L	-
Cooking oil	0.1 ml/L	-

RESULTS AND DISCUSSIONS

Fourier Transform-Infrared (FTIR) Analysis

The Fourier Transform Infrared spectroscopy characterization of iron chloride based AC showed a number of peaks as shown in figure 3. The first peak observed during this analysis was at 876.99 cm^{-1}, which was the evidence for the availability of aryl hydrocarbons. The next peak observed at 1157.52 cm^{-1} were identified as alkali halides produced during the breakdown of chlorine present in $FeCl_3$. Subsequently, the peaks arisen at 1557.27 cm^{-1} and 1694.50 cm^{-1} were basically the confirmation of decomposition of amino acid present in CS, having the structure of C-O and member of the carboxylic group. Afterward, the next functional groups observed on the surface of activated carbon were aldehydes and alkynes having wavelength number of 1682.68 cm^{-1} and 2111.97 cm^{-1} respectively. The peak noticed at 1567.98 cm^{-1} were 4-ring structured alkenes having C=C double bonding. The variety of miscellaneous functional groups were also observed during FTIR analysis of $FeCl_3$-AC at 1990.04 cm^{-1}, and 2357.63 cm^{-1} these may be the oxides of iron breaks down at a temperature of 400^0C. The peak at 3323.49 cm^{-1} was the alkynes having structure RC≡CH. The alcohol group was also identified in this analysis having peaks number 3581.94 cm^{-1} and 3592.44 cm^{-1} with O-H starch and RCH_2OH arrangement. In last continues spectrum was noticed, that may be the presence of moisture in AC as acknowledged by Gonzalez [30].

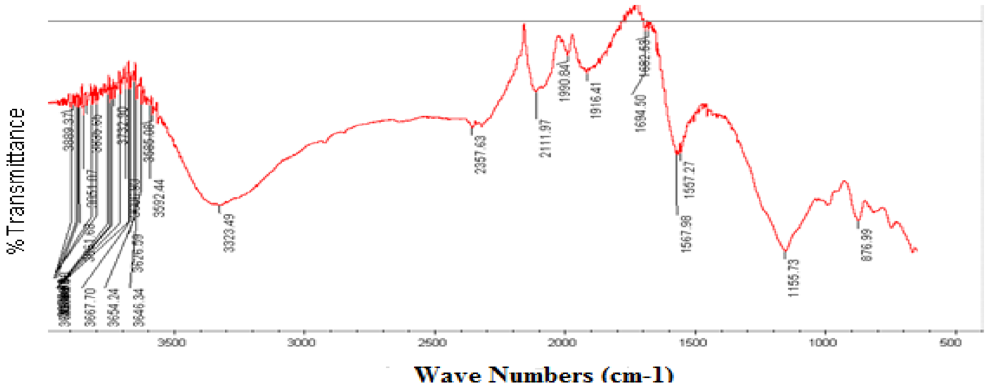

Fig. 3: Fourier Transform Infrared spectroscopy of Iron Chloride Based AC

The FTIR analysis of sulphuric acid based activated carbon identified very complex nature having a variety of functional groups present on its surface as shown in figure 4. The number of spectrum examined during this study including 3564.01 cm^{-1} was the alcohol group with O-H single stretch. The carboxylic group was also examined at the spectrum range of 3360.30 cm^{-1} with OH expansion. The miscellaneous groups with Si-H and N=C on the surface of H_2SO_4-AC were measured on continuous peaks at 2359.11 cm^{-1}, 2320.84 cm^{-1},and 2110.76 cm^{-1}. The stretch of C-O single bond and C=O double bond were the evidence of carboxylic groups at 1695.01 cm^{-1}, 1574.55 cm^{-1}, and 1557.42 cm^{-1} three continuous spectra. Another miscellaneous group was observed at 1180.09 cm^{-1} which showed S=O double bonding stretch. After the pyrolysis process of biomass, the lignin was converted into aromatic forms having C-H single bond. The peaks observed at 877.52 cm^{-1}, and 735.00 cm^{-1} were the evidence of that process [31].

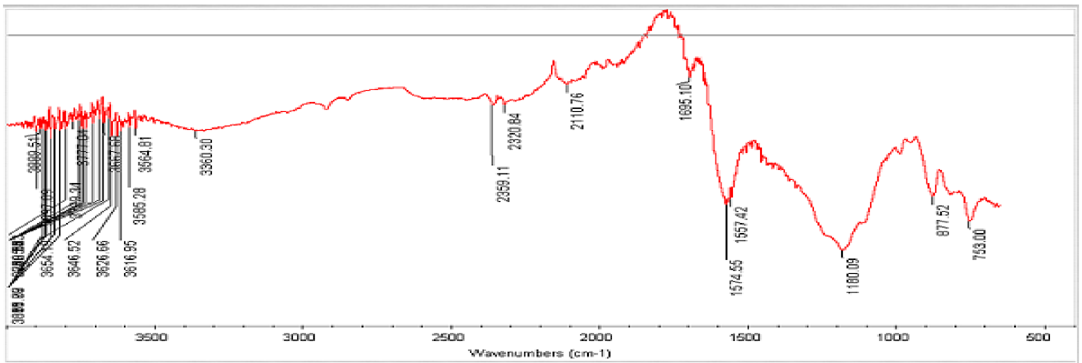

Fig. 4: Fourier Transform Infrared spectroscopy of Sulphuric Acid Based AC

X-Ray Diffractometry (XRD) of Activated Carbon

X-Ray Diffractometry (XRD) of iron chloride based activated carbon ($FeCl_3$-AC) extracted from biomass cotton stalk (CS) showed a continuous pattern (see fig. 5). The XRD analysis of $FeCl_3$-AC identified that the AC produced 400 ^{0}C is basically an amorphous solid. The continuous spectrum graph obtained during XRD analysis revealed the results of the scanning electron microscope (SEM) for $FeCl_3$-AC. The results of XRD showed that the precursor obtained from agriculture waste is basically composed of 8.5% crystalline nature while remaining 91.5% is amorphous [32].

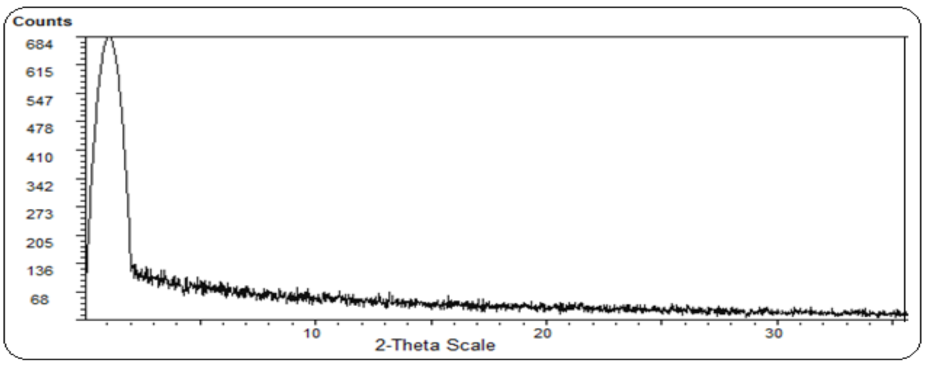

Fig. 5: X-ray Diffractogram of IronChloride Based AC

X-ray Diffractogram of sulphuric acid based activated carbon obtained from locally available agriculture waste cotton stalk exhibited a primary peak at 2θ=2, which was the identification of graphite as observed in another study [2]. The noise production of XRD signal at that peak was also the evidence that H_2SO_4-AC is amorphous in nature. The XRD results of H_2SO_4-AC also identified that the biomass breaks down on high temperature and converted into more active substances. The carbonization of biomass yield a compound representative layer of graphite. According to Lua and Yang the pyrolysis of biomaterial reduce its quantity and size, but the final product achieved by this process is more reactive than its parental form [33].

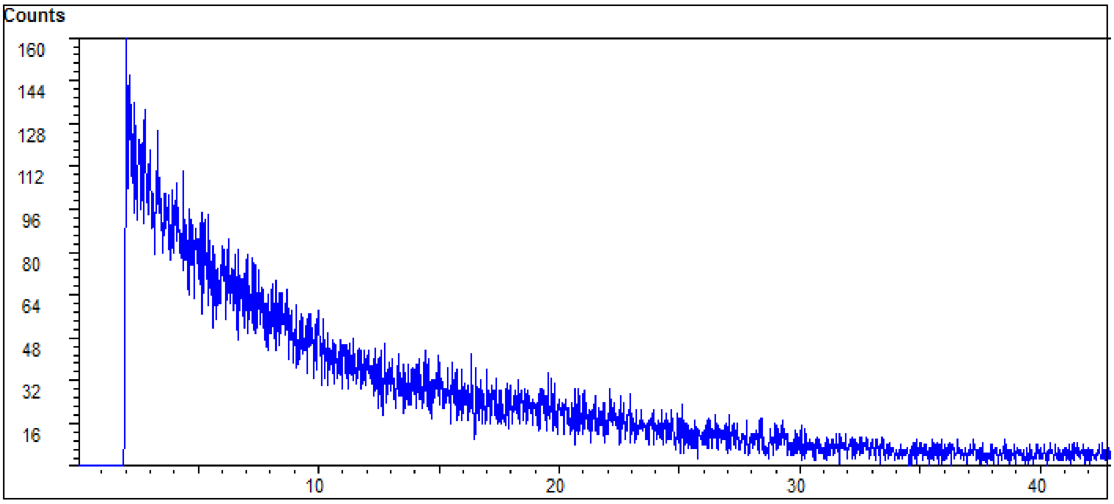

Fig. 6: X-Ray Diffractogram of sulphuric acid based AC

Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a technique used to examine the surface morphology of any material. SEM images of iron chloride based AC were recognized by using this technique as shown in figure 7. A uniform series of tunnels or a honeycomb structure were obtained in the images. While the amount of activating agent ($FeCl_3$)used as a 4th part of charcoal weight and diluted in 100 ml water was not observed in the SEM images. Several images were taken during this analysis having magnification range from 500 to 3000. A clear pattern of pores distribution was analyzed and on high magnification up to 3000 a more clear view for the presence of sub-pores was observed. SEM microscopy showed that iron chloride was a suitable activating agent even at lower amount for activation of biomass charcoal.

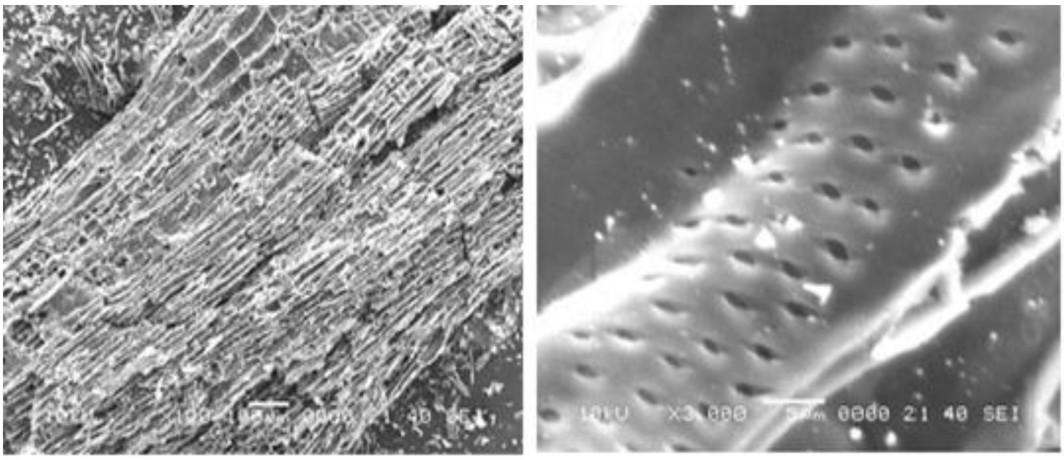

Figure 7: Scanning Electron Microscope Results of Iron Chloride based AC

A rigid solid material was observed during the SEM characterization of sulphuric acid based activated carbon as shown in figure 8. The magnification used for this analysis was in between 500 to 3000. While the images obtained in SEM classification were observed more porous with variety of sub pores as compare to iron chloride based AC. The magnification of 3000 clarify the presence of micro, macro and medium size pores. SEM images of sulphuric acid based AC showed a network of grooves which will be ultimately helpful during the adsorption process. A study suggested that rigid and grooves network are the results of activating agent and high temperature used in pyrolysis process [34].

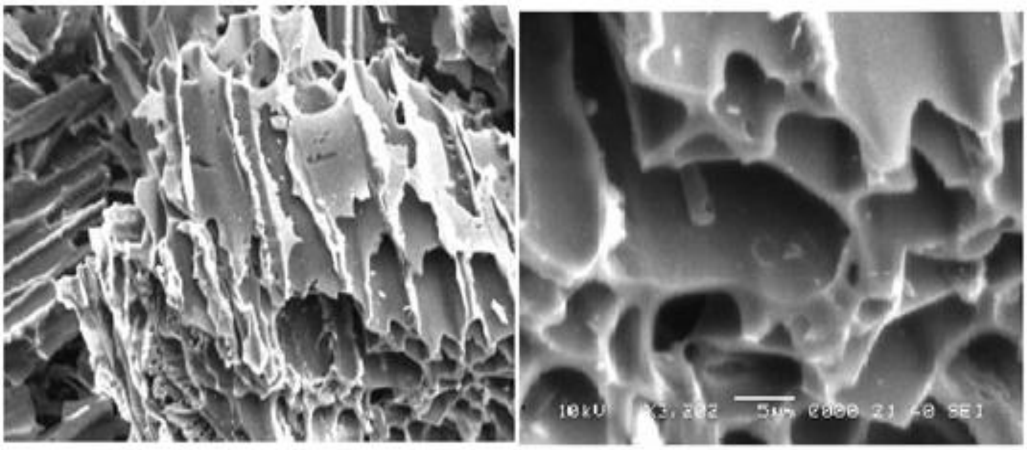

Figure 8: Scanning Electron Microscope Results of Sulphuric Acid Based AC

Analysis of SGW before and after Treatment with ACs and River Sand Filter

In an adsorption process the velocity of flow is directly proportional to depth of filter. The filter depth for greywater treatment and filter medium can be expressed in term of head loss.

l = α Lh

Where $\alpha = V^2a/g\ [1\text{-}e/\phi e^3]\ \sum (fx/d)$ (1)

f=150 (1-e)/ re + 1.75

re= ϕ (PVD)/u

Where, h is head loss, f is fraction factor, L is depth, d is dimeter of geometric mean, Va is approach velocity, Φ is shape factor, u is viscosity, e is porosity, x is weight of particles, g is gravity, p is density of water. To simplify the design, a graph between head loss and depth of filter media is shown as a figure 9.

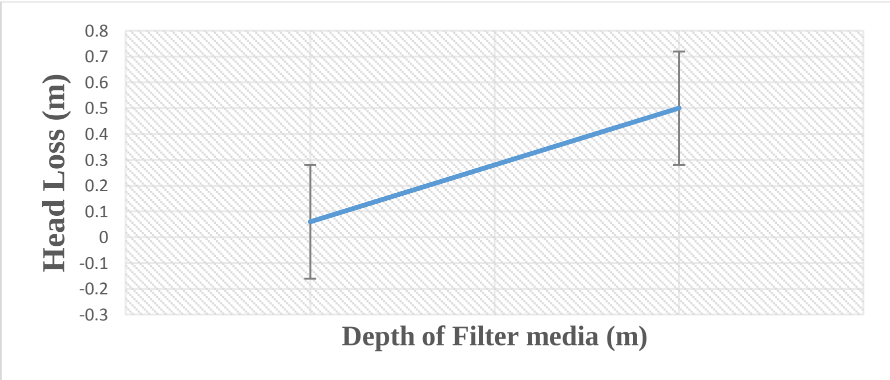

Figure 9: Design Chart of Head Loss versus Depth

The washing powder used in the preparation SGW in this study converted it into a strong alkaline in nature. As identified Schafer that a greywater can be more alkaline as compared to other domestic wastewater with pH range up to 11 [35].The preliminary value of SGW for pH was 10, but after the passing of SGW from H_2SO_4-AC and sand columns the pH value was 7.2[36].Similarly, the results of pH after treatment with $FeCl_3$-AC in combination with the sand filter was 8. The pH results were identical with a previous researchsuggested by Hourlier [29].

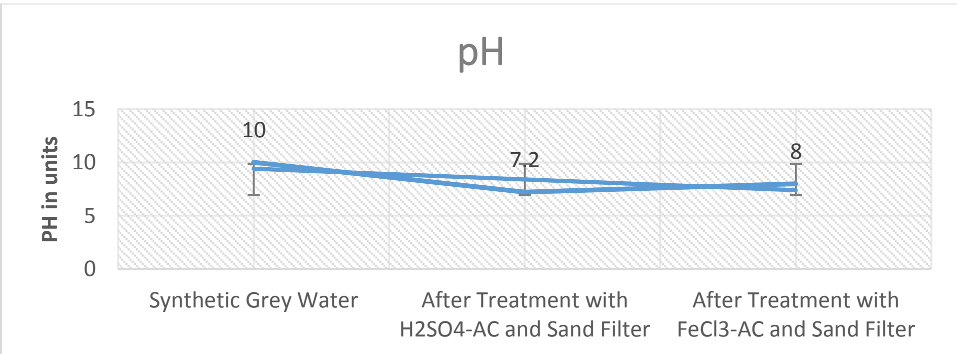

Figure 10: pH before and after the Application of filter media

Turbidity is the degree of water transparency. It is the most critical parameter which contains numerous elements. The turbidity of prepared SGW was fluctuating at an initial stage before treatment. Turbidity was 36.8 NTU at its initial stage, while after the application of H_2SO_4-AC the value was 1.6 NTU [37]. While the measuring value of turbidity after filtration with $FeCl_3$-AC and sand filter was 3.2.At the initial stage the value of total suspended solids was 34 mg/L. The measuring value after the application of sulphuric acid based activated carbon in combination with sand filter for total suspended solids (TSS) 0.68 mg/L. Similarly, when the synthetic grey water (SGW) was passed through iron chloride based activated carbon in combination with sand filter the final value for TSS was 3.1 mg/L. While presence of oil and fates was measured with the value of 54 mg/L at an initial stage bore the application of filter mediums. After the application of H_2SO_4-AC and $FeCl_3$-AC in combination with sand the final values were 0.68 mg/L and 1.61 mg/L respectively.

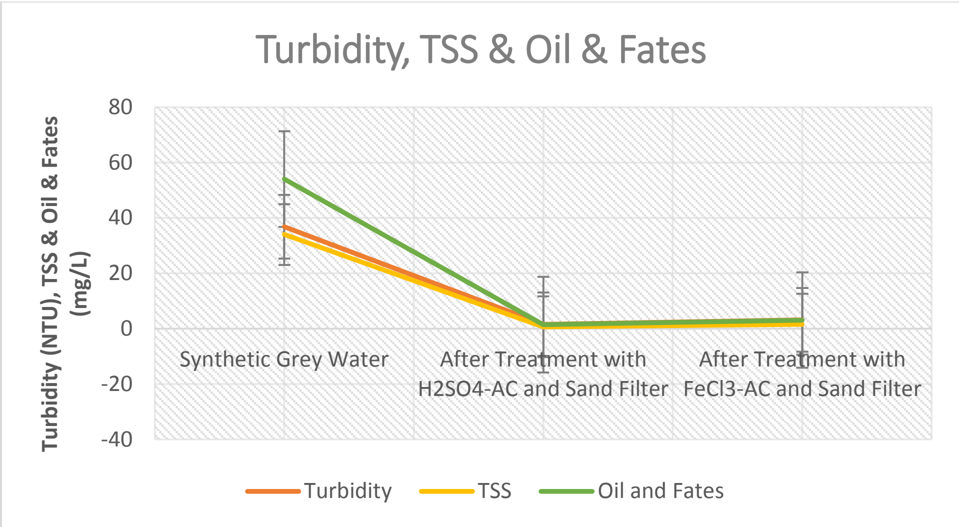

Figure 11: Turbidity, TSS and Oil & Fates before and after the Application of filter media

The initial values before the application of H_2SO_4-AC column for COD and BOD_5 were 81 mg/L and 40.6 mg/L respectively. After the treatment, the values for COD and BOD_5 were 2.3 mg/L and 2 mg/L respectively [38]. A significant variation was observed in the values of BOD_5. The result of BOD_5 after treatment with $FeCl_3$-AC and sand filter was 3.26 mg/L. A previous study also identified using same filter media the removal of BOD from 52.4 to 5.3 mg/L [39]. The COD measure values of synthetic grey water before treatment was higher than BOD because the many organic substances oxidized chemically rather than biologically. After the application of $FeCl_3$-AC and sand filter, the value of COD was 5.2 mg/L.

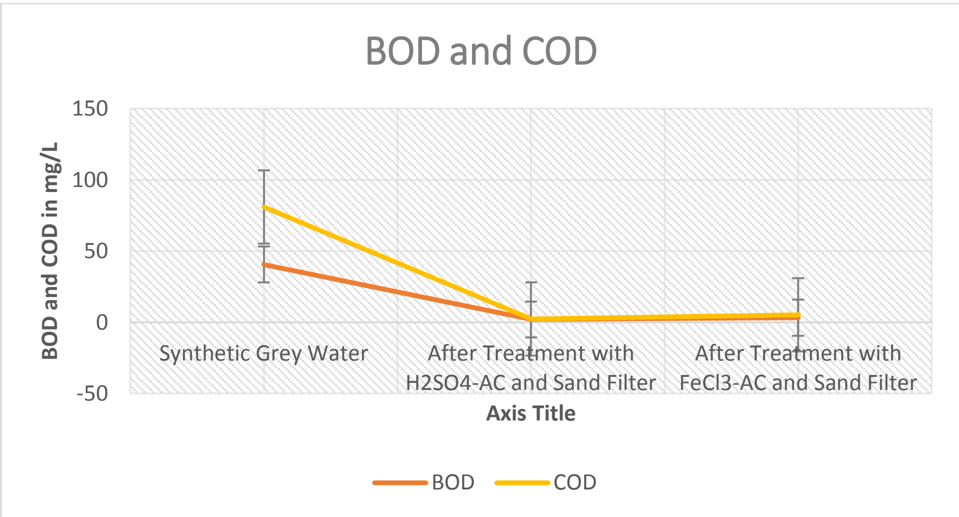

Figure 12: BOD and COD before and after the Application of filter media

Electrical conductivity (EC) of the SGW was found in a negligible quantity before the treatment with a value of 0.4 µS/cm, but after bothtreatments, a minor increase was observed in the values of EC. The value for $FeCl_3$-AC was1.6, while for H_2SO_4-AC the value was 1.65 µS/cm [40]. As suggest by Put hat granular activated carbon (GAC) can transfer electron between some compounds[41].

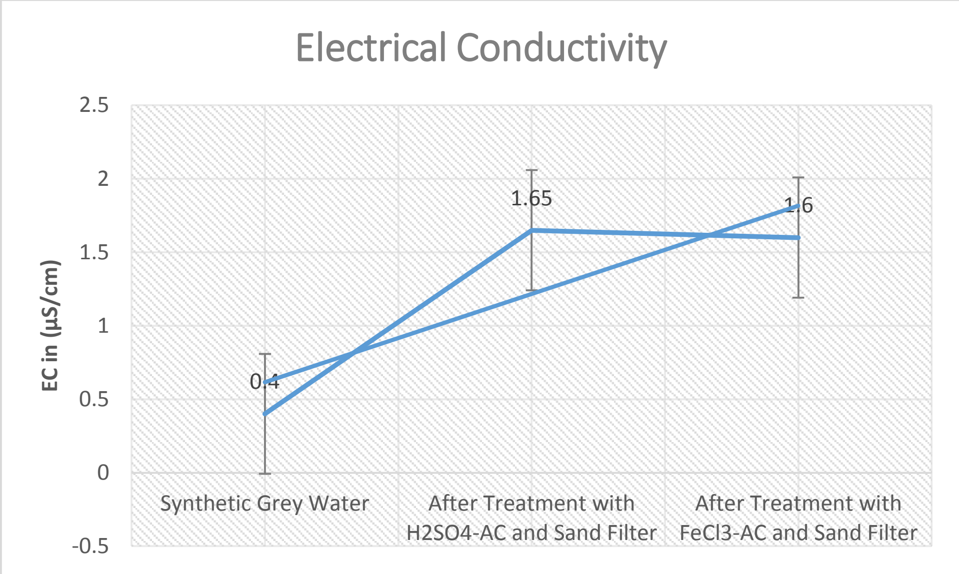

Figure 13: EC before and after the Application of filter media

While for total coliforms were 1500 CFU/100 ml initial stage but after treatment with H_2SO_4-AC and sand filter 4.21 CFU/100 ml. The previous study suggests that the range of total coliforms in a light greywater can be from 500 CFU/100ml to 2.3×10^7CFU/100ml [42]. The initial measuring value obtained during the characterization of total coliform in SGW was 1.5×10^2CFU/100ml. While the amount of sewage used to show the presence of harmful microorganisms was in a small amount of 10ml/L. After the application of both filters ($FeCl_3$-AC and sand filter), the final measuring value was 6.32 CFU/100 ml.

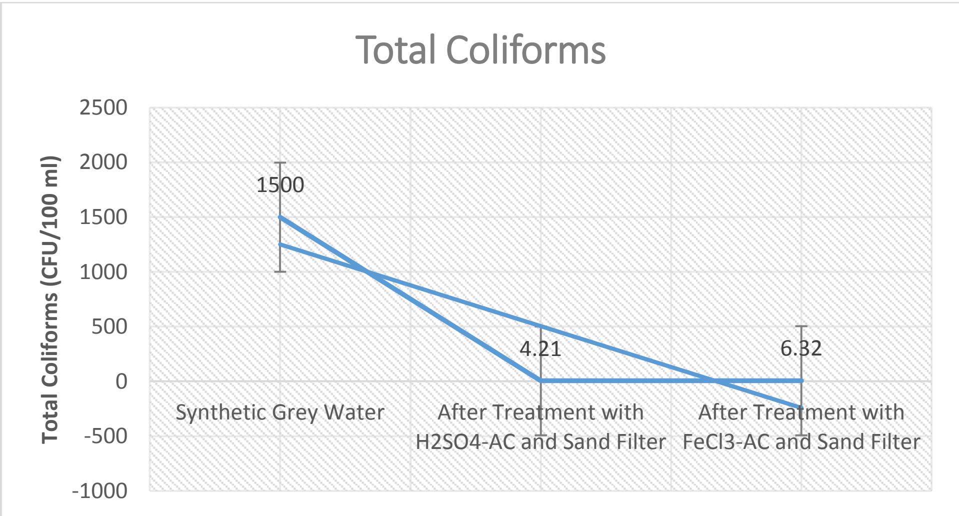

Figure 14: Total Coliforms before and after the Application of filter media

Table 2: Characterization of SGW before and after treatment

Parameters	Synthetic Grey Water	H_2SO_4-AC and Sand Filter	$FeCl_3$-AC and Sand Filter
pH	10	7.2	8
Turbidity (NTU)	36.8	1.6	3.2
BOD_5 (mg/L)	40.6	2	3.26
EC (µS/cm)	0.4	1.65	1.6
Oil and Fates (mg/L)	54	1.41	3.1
TSS (mg/L)	34	0.68	1.61
COD (mg/L)	81	2.3	5.2
Total Coliforms (CFU/100 ml)	1.5×10^2	4.21	6.32

CONCLUSION

Sulphuric acid (H_2SO_4) and iron chloride ($FeCl_3$) played an important role in the activation process with its minimum volume. While the characterization parameters taken to analyze both ACs were FTIR analysis, the XRD analysis, and SEM. The fumes and vapors generated during the production process of activated carbon reveal that the soft part of biomass contains fiber and cellulose. The results of SEM images showed more sequences of sub-pores on high magnifications. On another hand, even the lower activation temperature and amount of activating agents used in the process of preparation of ACs, but the adsorption rates of ACs were found still high on all selected parameters of synthetic grey water combined witha sand filter. The synthetic greywater prepared for the analyzation was obtained by using basic ingredients used in our homes. The research analyzed that activated carbon in combination with sand bed filter was an effective medium to treat the greywater. On another hand, AC and sand are easily available filter media that can remove organic and microbial contamination of greywater. In addition,the combination of H_2SO_4-AC and sand filter was found more efficient as compared to $FeCl_3$-AC combined with a sand filter for the treatment of SGW.

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