

**COMPARISON OF COMBINED FILTER AND ACTIVATED CARBON ON
TREATMENT OF SYNTHETIC GREYWATER BY PRODUCED MGCL₂ BASED
ACTIVATED CARBON FROM RICE STRAW WASTE**

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<http://doi.org/10.35410/IJAEB.2019.4412>

ABSTRACT

The aim of the study was to provide an opportunity to reuse of grey water after treatment in order to overcome the scarcity of freshwater in Pakistan. Treated grey water can be reliable source for consumption i.e. landscape, public parks and irrigation etc. Rice straw is considered as a waste material globally and burnt at fields after harvesting of paddy rice so it was used as a raw material to produce both the filters. It is stated that activated carbon (AC) prepared by agro-industrial waste are considered as low-cost generated as compared to industrially prepared activated carbon. Activated carbon was prepared in a muffle furnace and added activating agent (MgCl₂) in an inert atmosphere at the temperature 550 °C for two hours. Filters for application were fabricated by two different methods, one was filled by activated carbon and the other one partially filled by rice straw and remaining space was filled by activated carbon. Efficiency of both the filters was assessed by application on synthetic grey water. The FTIR results of MgCl₂ Based activated carbon showed 6 peaks which confirms the attachment of aromatics, carboxylic acids, amides, and miscellaneous classes on the surface. Whereas, FTIR of rice straw showed the functional groups of amines, aromatic, alkanes, alkanes and amines so it is concluded that different peaks have the same functional groups but with different structures. While the XRD analysis of produced activated carbon showed the peaks between 10 to 40° which is the confirmations of the amorphous crystalline structure and residual ash whereas rice straw XRD results shows that it is crystalline structure and having good stability for adsorption. Furthermore, SEM images authenticated the XRD results by showing the presence of MgCl₂ on the surface of activated carbon. It was also concluded from SEM micrographs that the high temperature and activating agent influences the structure of biomass (Rice Straw) and helps in converting it into a more use full material. SEM images of rice straw are showing uniform structure and a series of tunnels. Magnification in SEM at 3000, sub-pores were observed with the irregular shape and smooth surface. It was concluded that combined filter is more efficient than the activated carbon filter which has significantly removed BOD₅, COD, TSS, Turbidity and Oil & Fats up to 93%, 92%, 75%, 91% and 93%, respectively and pH of the synthetic grey water was neutralized from 9.2 to 7.8 by combined filter whereas activated carbon has neutralized up to 8.0.

Keywords: Rice Straw (RS), activated carbon (AC), MgCl₂, Biomass, Greywater Treatment, Combined Filter.

1. INTRODUCTION

Nowadays availability of fresh water in the world is less than 1%. Depletion of fresh water is increasing day by day and nowadays it has become environmental concern globally [1]. It was predicted that population will be increased to 9 billion in 2050 so the consumption of fresh water will also be increased [2]. This leads to adopt and introduce new technologies of extracting fresh water from wastewater or grey-water for industrial and agricultural usage [3]. Pakistan totally depends upon snowmelt and rainfall for restoration of fresh water resources [4]. As reported by UNO Pakistan is 7th number in the list of water stressed countries, resources of fresh water are only 177 MAF including 14% underground water and 86% surface-water. The studies were carried out and concluded that Pakistan might be facing 33 MAF fresh water scarcities by 2025 [5]. All sources from domestic wastewater except black-water (wastewater from toilets) is called as grey water, which is very important in developing countries. The improper disposal of wastewater is main cause of fatal diseases as well as environmental pollution [6]. Proper management of grey water includes gathering and recycling after treatment to avoid individuals in contact with it and restricts transfer of pathogens, the treatments of water bodies has positive roll to reduce nutrients and also eutrophication [7]. The recycling of grey water is experienced in all over the world to increase the availability of water and protects human health and beneficial for other environmental aspects [8]. Treated grey-water can be reliable source for consumption. Grey-water contains up to 70% of water, approximately 30% of organic fraction and 9 to 20% nutrients [9]. The treated water is mostly consumed for non-potable (not for drinking) purposes, such as landscape, public parks and irrigation etc. Furthermore non-potable water includes coolant for power plants or oil refineries and also for other industrial processes [10]. There are various methods to treat the grey water but adsorption is the method which is believed as an efficient and economical method for the treatment of grey water. It is also favored because it does not produce sludge [11]. The adsorbent is considered cost-effective if it is produced from natural resources or any waste material such as agriculture waste, [12]. In industrial point of view activated carbon is the considered as significant adsorbent [13]. Activated carbon is a complex porous structure which has energetic as well as chemical inhomogeneity. It was challenge to produce a low cost activated carbon with distinct pore sizes at low temperature [14]. There are many natural resources to produce high specific surface area and pore volume carbonaceous materials such as coconut shells, bagasse from sugarcane industries, coal, cotton stalks, rice husk, wood [15-19], etc. Rice straw is produced as a byproduct of rice crop and considered as agriculture residue. Rice straw is roughly produced 800 to 1,000 million tons per year, globally [20]. There are two processes for the production of activated carbon; chemical activation and physical activation. Single stage method of activated carbon in presence of chemical agent is known as chemical activation and a carbonaceous material followed by activation of prepared charcoal in presence of activating agent such as FeCl₃ or steam is known as two stage method [21]. There are different temperatures are used to produce activated carbon and different chemical activating agents are also used to activate such as KOH and K₂CO₃ [22], H₃PO₄ and ZnCl₃ [23].

2. MATERIALS AND METHODS

2.1 Preparation of rice straw

Initially, rice straw was shorten into small pieces of 1.5 cm and then washed to remove dirt and other impurities. Furthermore, it was dried in an oven at 105 °C for 24 hours to dehydrate. The material was prepared for both combined filter (RS & AC) and activated carbon filter [24].



Figure 1: Raw material (Rice Straw).

2.2 Preparation of activated carbon

Preparation was started with impregnation of with an activating agent ($MgCl_2$) 1:1 by weight then oven dried at 105 °C for 48 hours. For activation it was filled in crucibles having volume 100 ml and these crucibles were placed in steel container containing sand to create an inert atmosphere. Container was then covered with airtight lid for pyrolysis in a muffle furnace at 550 °C for 2 hours. After this process steel container was taken out from the muffle furnace and kept at room temperature for cooling [25].



Figure 2: Crucible and steel container used for activated carbon.

3. CHARACTERIZATION OF ACTIVATED CARBON

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

The equipment used for the FTIR spectrometer Analyzation was Thermo Nicolet 6700 having a changeable KBr optic and a detector made up of deuterated triglycine sulfate (DTGS) to examine

the standard pellets and samples. The standard regions of examination taken for all spectra were in mid-infrared (IR) of 4000-400 cm^{-1} having the resolution of 4 cm^{-1} with the accumulation rate 32 scans per spectra.

3.2 X-Ray Diffraction(XRD)

XRD analysis was carried by Philips XRD instrument, equipped with Cuka radiations of 40 KV and 30 mA, and having 2-theta of step size 0.05°. The scanning rate employed for spectra measure was 1°/minute.

3.3 Scanning Electron Microscopy(SEM)

Morphology, structure, and composition of nanostructures formed on the surface of activated carbon was observed by using SEM micrographs obtained on a JEOL, Tokyo, Japan machine.

4. PREPARATION OF SYNTHETIC GREY WATER

Synthetic greywater was prepared by using different ingredients such as Ammonium Chloride (NH_4Cl), Backing Yeast ($\text{C}_{19}\text{H}_{14}\text{O}_2$), Sodium carbonate (Na_2CO_3), Flour (fine grinded), Shower gel, Detergent powder, Polysaccharides($(\text{C}_6\text{H}_{10}\text{O}_5)_n$), Cooking oil, *Sewage*, Potassium Sulfate (K_2SO_4) and sodium DiHydrogen Phosphate which are shown in (Table 1) [26].

Table 1: List of synthetic grey water ingredients

Chemical Name	Concentration	Chemical formula
Polysaccharides	425 milligram Liter ⁻⁵	$(\text{C}_6\text{H}_{10}\text{O}_5)_n$
Ammonium Chloride	375 milligram Liter ⁻⁵	NH_4Cl
Backing Yeast	350 milligram Liter ⁻⁵	-
Flour (fine grinded)	275 milligram Liter ⁻⁵	-
Sodium Carbonate	275 milligram Liter ⁻⁵	Na_2CO_3
Detergent powder	150 milligram Liter ⁻⁵	N-A
Sodium Dihydrogen phosphate	57. milligram Liter ⁻⁵	NaH_2PO_4
Potassium sulfate	22.5 milligram Liter ⁻⁵	K_2SO_4
<i>Sewage</i>	50 milligram Liter ⁻⁵	-
Shower Gel	0.5 millilitre ⁻⁵	-
Cooking oil	0.5 millilitre ⁻⁵	-

5 FABRICATION OF DESIGNED MODEL

Transparent glass tank (as shown in figure. 3) was installed on the top of designed model for sedimentation purpose, which has volumetric capacity of 40 litres. Then both the filters were installed below the tank along with controlled valve for inlet and outlet flow. Columns were 50cm long having 2cm diameter. One column was filled with activated carbon and other with activated carbon and rice straw (Combined filter). Synthetic grey water was left for 3 hours to settle down then passed by both filters separately and samples were collected in containers. Diagram of designed model is shown in figure (4).



Figure 3: Designed model

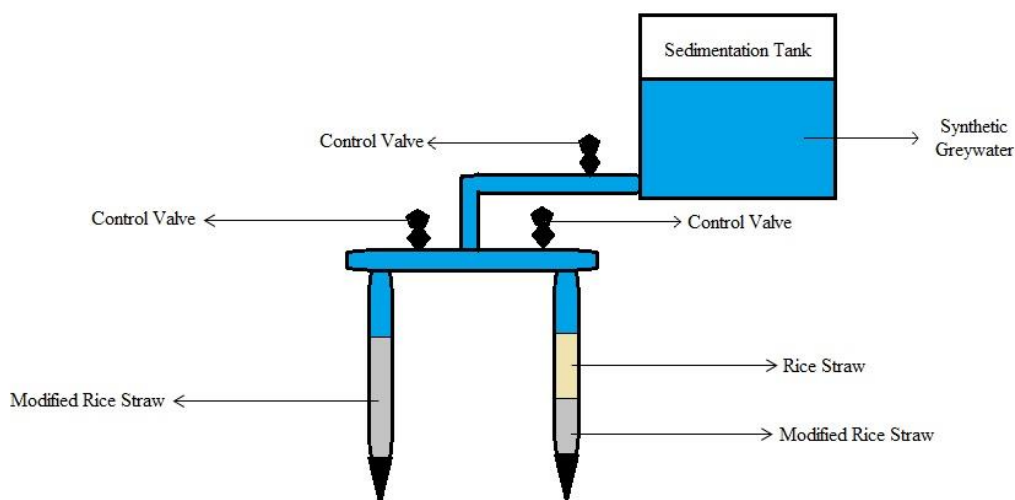


Figure 4: Diagram of designed model

6.RESULTS AND DISCUSSIONS

6.1 FTIR results for Activated Carbon

Rice straw activated carbon showed different peaks, 711 and 873 cm^{-1} are identifying aromatic class while the assignment of that peaks was C-H out of plan as discussed by Coates [27]. Whereas, 1062 cm^{-1} bend shows carboxylic group having RCO-OH structure and C-O assignments corresponding with Yeasmin and Mondal [28]. As stated by You [29] that 1633 cm^{-1} has amides group and having structure RCONH₂. Furthermore, miscellaneous substances observed at peak 2351 cm^{-1} having P-H phosphine and assignment were P-H phosphine sharp are matching with Javadian and Taghavi [30]. It was described by Bhuiyan [31] that 3265 cm^{-1} peaks have a carboxylic group having RCO-OH structure and dimer OH assignment.

Table 2: FTIR results of rice straw activated carbon.

S.No.	Peaks	Class	Structure	Assignment
1	711 cm^{-1}	Aromatics	1,2,3 tri-substance	C-H put plane
2	873 cm^{-1}		1,2,4 tri-substance	
3	1062 cm^{-1}	Carboxylic acid	RCO-OH	C-O stretch
4	1633 cm^{-1}	Amides	RCONH ₂	NH out of the plane
5	2351 cm^{-1}	Miscellaneous	P-H phosphine	P-H phosphine sharp
6	3265 cm^{-1}	Carboxylic Acid	RCO-OH	Dimer OH

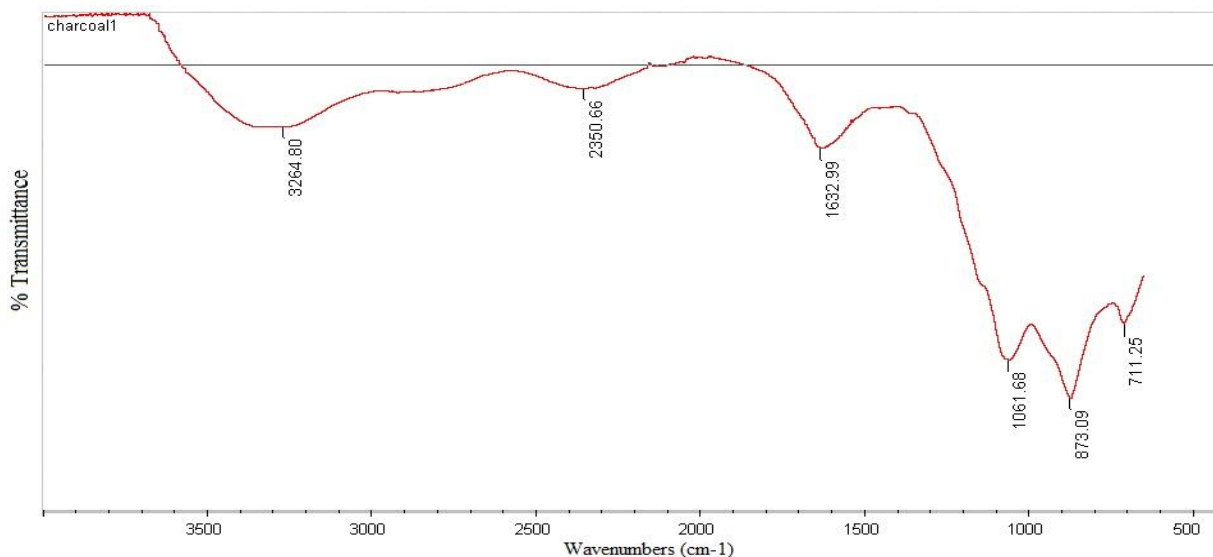


Figure5: FTIR Peaks of Activated Carbon.

6.2 FTIR results for rice straw

The FTIR peaks of rice straw were observed within the range of 400-4000 cm^{-1} . The first peak at 1034 cm^{-1} is showing amines functional group having the R₂NH structure with strong intensity and C-N stretch as discussed by, Sim [32]. The peak at 1421 cm^{-1} shows aromatic functional group with C-C in the ring structure and medium intensity, same results were discussed by Chen [33]. Peak at 1621 cm^{-1} showing a functional group of alkanes having Ar-CH=CHR structure and strong intensity, however, same peaks were observed by Aggarwal [34]. Spectrum at 2919 cm^{-1} showing a functional group of alkanes having RCH₂CH₃ structure and CH stretch which are too

weak and low in frequency as observed by Musa [35]. The last peak at 3345 cm^{-1} shows amines functional group having an R_2NH structure with weak intensity and stretching at NH, as described by Tay [36].

Table 3: FTIR results of rice straw

S.No.	Peaks	Class	Structure	Assignment
1.	1034 cm^{-1}	Amines	R_2NH	C-N stretch
2.	1421 cm^{-1}	Aromatic	C-C in ring	Ar C-C stretch
3.	1621 cm^{-1}	Alkanes	Ar-CH=CHR	-
4.	2919 cm^{-1}	Alkanes	RCH_2CH_3	CH stretch
5.	3345 cm^{-1}	Amines	R_2NH	NH stretch

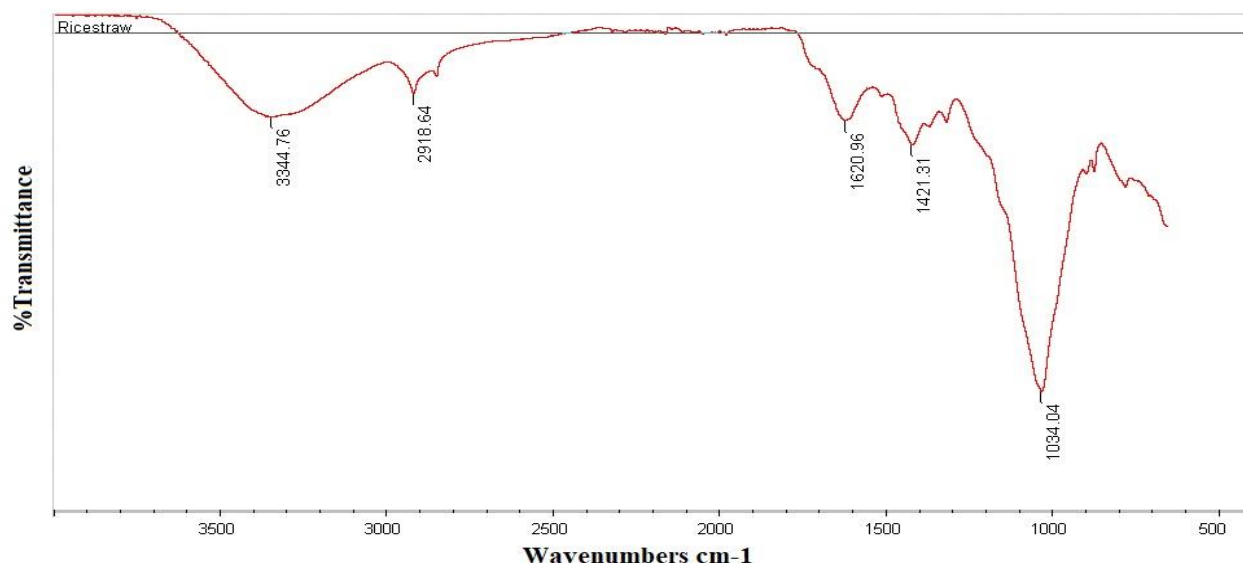


Figure 6: FTIR peaks of rice straw

6.3 XRD results for Activated Carbon

The crystalline structure of MgCl_2 -Based activated charcoal was analyzed with X-Ray Diffraction (shown in fig. 7). The three peaks were observed $2\theta=10^\circ$, $2\theta=15^\circ$ and $2\theta=40^\circ$ were confirming that activated carbon has an amorphous structure and sharp peaks having continuous pattern are identifying the existence of remaining ash and bits of metal in amorphous shape. The MgCl_2 based charcoal's X-Ray Diffraction examinations are suggesting that some amount of MgCl_2 existed in the adsorbent. From 3 peaks it was clarified that extracted adsorbent from rice straw didn't fall totally in the crystalline group. Whereas the main part of activated carbon shows graphite nature. The size of the adsorbent was ranging in 8-20 nm [31].

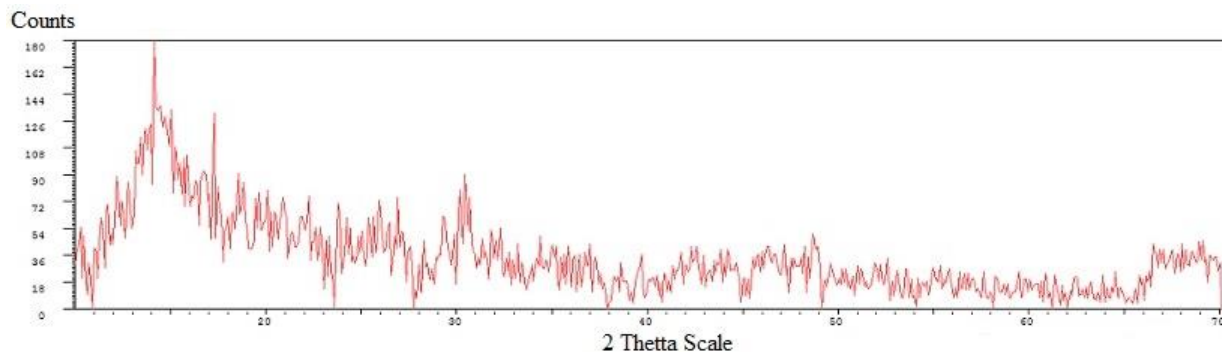


Figure 7: X-Ray Diffraction pattern of activated carbon.

6.4 XRD results for Rice Straw

The peak observed from 2θ to 20° theta are determining that rice straw was a crystalline material and $2\theta=25^\circ$ is showing the presence of silica, [37] and a continuous spectrum after 25° in theta scale showing that rice straw material has good stability for adsorption process as discussed by [38].

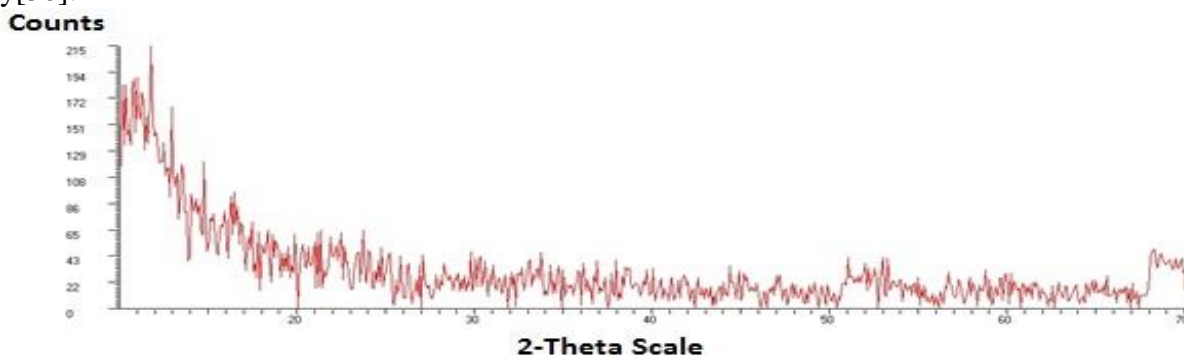


Figure8: X-Ray Diffraction of Rice Straw

6.5 SEM results for Activated Carbon

It was observed that Scanning electron microscopy images of $MgCl_2$ based charcoal were a nano-composite material having un-homogenized morphology. At 100 μm image showing the presence of $MgCl_2$ while dark part identifies the existence of rice straw charcoal. There was no pore structure in images [39]. Figures are shown below.

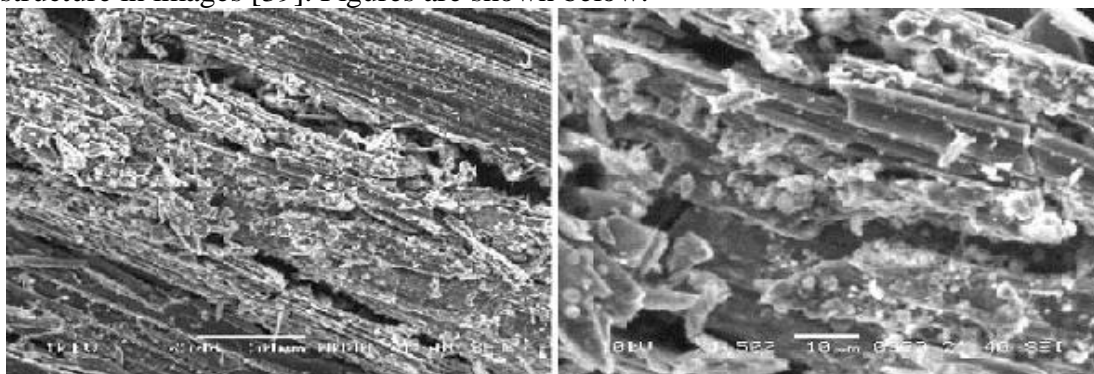


Figure 9:SEM images of activated carbon

6.6 SEM results for Rice Straw

The SEM images of rice straw showing that it has a uniform structure was observed by Ceyhan[40]. Up to 3000 magnification sub-pores can be observed. The morphology of images and irregular shape with different kinds of ranges and smooth surface as identified by Cao [41].

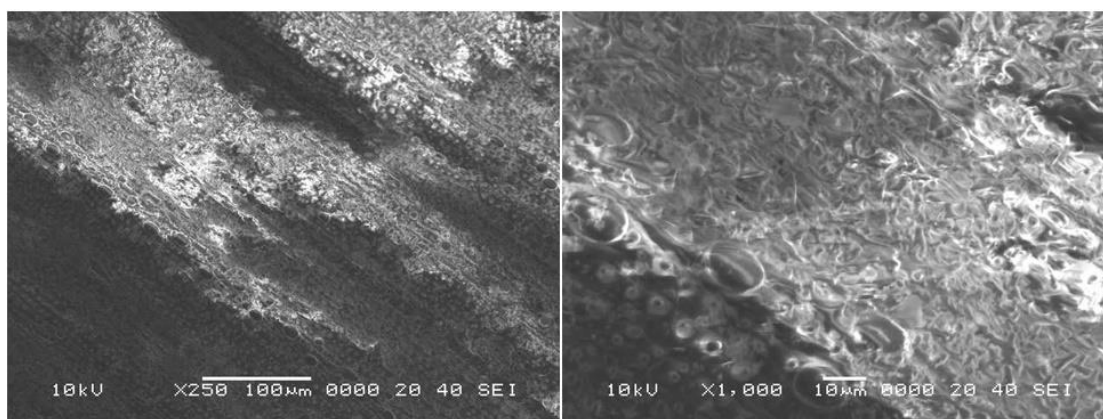


Figure 10: Scanning electron microscopy of rice straw

7. CHARACTERISTICS OF SYNTHETIC GREY WATER

The prepared synthetic grey water has pH 9.2, turbidity is 26 NTU, BOD₅ is 350 mg/L, COD is 500 mg/L, TSS is 435 mg/L, Oil and Fats are 40 ppm similar results were observed by [26],[42] and [43].

7.1 Application of activated carbon on synthetic grey water

The pH value after application has decreased up to 8.0 similar results were observed by [44]. While turbidity was decreased up to 89% and observed 3.8 NTU nearly same results were observed by [45]. BOD₅ after application was observed 100 mg/L (77% removal) and COD was decreased from 500 mg/L and reached at 175 mg/L (65% removal) as stated by [46] the produced activated carbon has capability to remove BOD₅ and COD more than 75% and 60%, respectively. TSS of synthetic grey water after application of activated carbon was observed 85 mg/L (80% removal). Values of oil and fats were decreased from 40 ppm to 4.5 ppm which is nearly 89%, similar results were observed by [47].

7.2 Application of combined filter on synthetic grey water

The pH value after application of combined filter has decreased up to 7.8, same results were observed by [48]. While turbidity was decreased up to 2.4 NTU (91% removal), [49] observed nearly same results. Whereas BOD₅ after application with combined filter was observed 27 mg/L (93% removal), same as observed by [50] and COD was decreased from 500 mg/L to 52 mg/L (92% removal), same results were concluded by, [51]. TSS of synthetic grey water after application of combined filter has become 110 mg/L (75% removal), same as described by [52].

Substantial decrease was observed in the value of oil and fats from 40 ppm up to 3.2 ppm (93% removal), results are related with [53].

Table 4: Comparison of efficiency of the both filters

Parameters	SGW Initial Results	Treated with activated carbon	Treated with Combined Filters (AC & RS)
pH	9.2	8.0	7.8
BOD ₅	350 mg/L	100 mg/L	27 mg/L
COD	500 mg/L	175 mg/L	52 mg/L
TSS	435 mg/L	85 mg/L	110 mg/L
Oil & Fats	40 ppm	4.5 ppm	3.2 ppm
Turbidity	26 NTU	3.8 NTU	2.4 NTU

8. CONCLUSION

The study has successfully provided a better way to utilize rice straw and to overcome the scarcity and depletion of freshwater in Pakistan. It was concluded that rice straw can be converted into activated carbon and low concentration greywater can be treated. Combined filter is more efficient than the activated carbon filter although FTIR results showed attachment of approximately same functional groups but XRD and SEM has further illustrated that the structure and shape is different in both the filters which has significant impact on its efficiency.

9. Acknowledgement

The authors are thankful to Mehran University of Engineering and Technology Jamshoro, Sindh Pakistan, to provide experimental and financial support for research work.

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