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Research Article

Removal of Cr^{6+} from Synthetic Polluted Water using Fe Modified Sugarcane Bagasse & Peanut shell Powder

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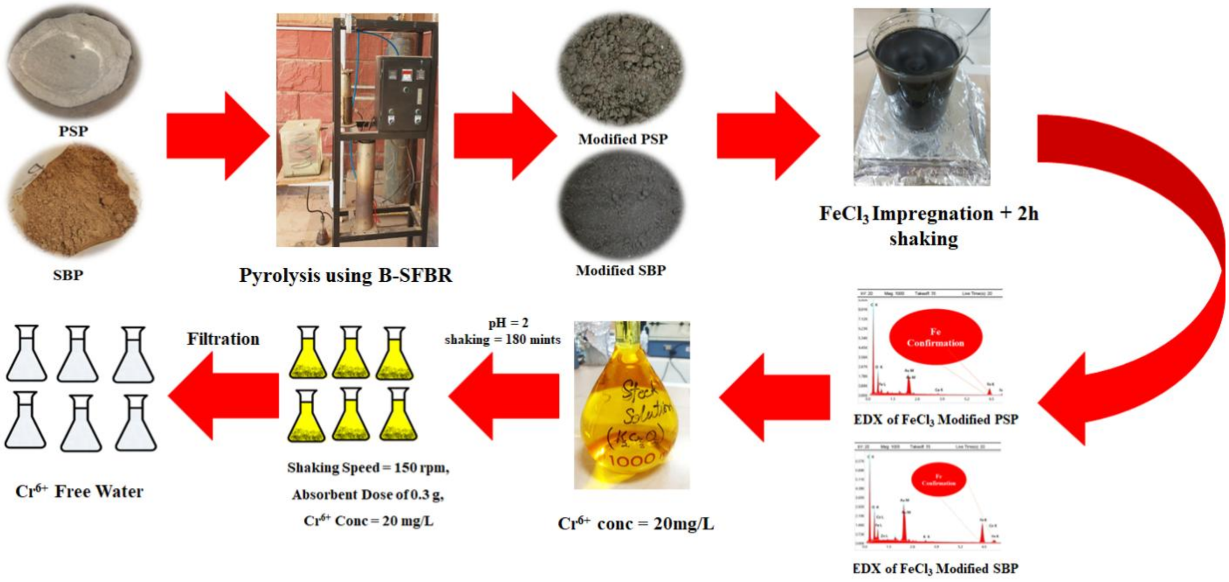
Abstract

Unprocessed waste is dumped in lakes and rivers which possess a severe environmental risk through heavy metal's contamination within the food chain, especially chromium Cr^{6+}. It applies for sustainable development goals by using green solid waste as a precursor. This research mainly focuses upon the analysis of time effect for Cr^{6+} removal by $FeCl_3$ modified biochar using sugarcane bagasse and peanut shell powder as biomass. Adsorbent preparation was done using Bench-Scale Fixed Bed Reactor (B-SFBR) and Cr^{6+} was found using the calorimetric method. Characterization was done by BET, SEM-EDX, and FTIR. The highest Cr^{6+} percentage removal was achieved by Modified Peanut shell Powder with 99.97% removal upon pH 2, shaking time 180 mints, speed = 150 rpm, dosage 0.3 g, Cr^{6+} conc 20 mg/L. Percentage Removal by Modified SB was 98.96% with Cr^{6+} conc 20 mg/L, dosage 0.3 g, pH 2, shaking speed 150 rpm, time 180 mints. Hence, the present experimental research concludes that $FeCl_3$ modified peanut shell powder shows greater Cr^{6+} removal efficiency up to 99.97 %.

Keywords: Water quality, Cr^{6+} pollution, Adsorbent, Sugarcane Bagasse powder, Peanut shell powder.

Graphical Abstract:



1. Introduction

As urbanization is increasing, water pollution is increasing, and availability of clean and safe drinking water becomes very less [1] . Pollution in water is increasing day by day because of the industrial revolution. From the last several years, the use of adsorption process for the removal of heavy metals and purification of water is being used. The most used and economical and low-cost method for the removal of heavy metals from water is done by using activated biochar as an adsorbent. These have a microporous composition having maximum surface area, porosity for the adsorption of heavy metals, any contaminant organic as well as inorganic impurities from water and air. The medium used to remove impurities called as adsorbents and materials or particles which absorb can be referred to as adsorbate. There are two main classifications of adsorbents subjected to involved forces, chemisorption and adsorbate-physisorption. Stable oxidations of Chromium in water are only two, trivalent Cr^{3+} and hexavalent chromium Cr^{6+}. Cr^{3+} is very critical for the nutrition of human for the digestion of glucose, it becomes very dangerous when the concentration is high above >150 mg L^{-1} for the physiology of the plant [2].

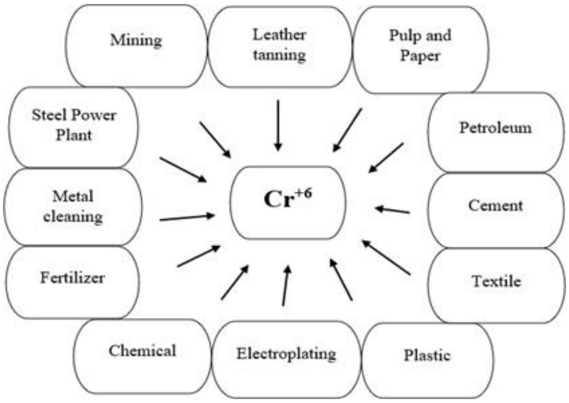


Figure 1. Industrial sources which lead to Cr^{6+} pollution [11] .

As chromium is carcinogenic in nature, it contributes to decrease or [3-7] WHO recommended the concentration of chromium in drinking water must be low that 50 µg/L. in Pakistan chromium concentration is very high owing release of water from textile, leather, metal finishing, cement, fabricationindustry [8-10].

Cr^{6+} present even at 0.05 mg/L concentration inside natural water is very carcinogenic [12]. Cr^{6+} when getting attached oxygen, it forms dichromate and chromate. These both are strongly water soluble and are less absorbed in soil as compared to water and lead to water contamination and pollution [13] .

In Pakistan, there are many unregistered and registered tanneries and footwear making units [14]. When these wastewater from gallons were analyzed, results showed a high amount of chromium concentration of about 592 mg/L [15] . In 2011 Karachi- Korangi chromium concentration in residue and filtrate wastewater was 18.75-170.12 mg/L and 15.2-185.5 mg/L [16].

Groundwater of Sialkot has 0.01-0.30 mg/L of chromium concentration [14]. In Sahiwal, drinking water we have 5.498 mg/L of chromium concentration [11]. Moreover, only one sample out of twelve had high concentrations of chromium about 0.6 mg/L from Sheikhupura [17]. These high concentration shows that industries do not treat water before they release their effluent into water in Pakistan and they do not act upon Environmental Law. Another study has used nonedible biomass (bagasse) for the synthesis and preparation of conducting resin [18]. Various methods are reported for the isolation of metals from industrial wastewater including ion [19-21] microfiltration, ultra-filtration, membrane filtration [22] reverse osmosis, cementation, electrocoagulation, solvent extraction, by using commercially made activated carbon, chemical precipitation [23-28] reduction of various adsorbent onto another adsorbents [29, 30]. These all methods of removal of heavy metals are not economical for developing or underdeveloped countries. Majorly, these methods are efficient for specifically at concentration 1-100 mg/L in a solution. The objective of this experimental research was the development of efficient adsorbent (modified biochar) made from locally available biomass present in considerable amount and its use for removal of Cr^{6+} from the aqueous solution. Secondly to check the effect of time for removal of Cr^{6+} using modified biochar made from sugarcane bagasse or peanut shell.

2. Materials and Methods

2.1 Solutions, Materials and Methods

Stock solution had been prepared by the addition of 2.83 g

$K_2Cr_2O_7$ in 1000 mL with Cr^{6+} conc of 20 mg/L in DI (Deionized) water (purchased from Sohail Chemicals, College Road, Rawalpindi, Pakistan). This prepared stock solution was farther used for the formulation of working solutions used in all the batch experiments. It was first diluted to 100 mL for preparation of solutions.

Table 1 Biomasses used in study.

Sr No.	Scientific Name	English name	Plant/Tree Parts	Local name
1.	Arachis hypogaea	Peanut	Shells	Mong Phali
2.	Saccharum officinarum	Sugarcane	Stalks	Ganna

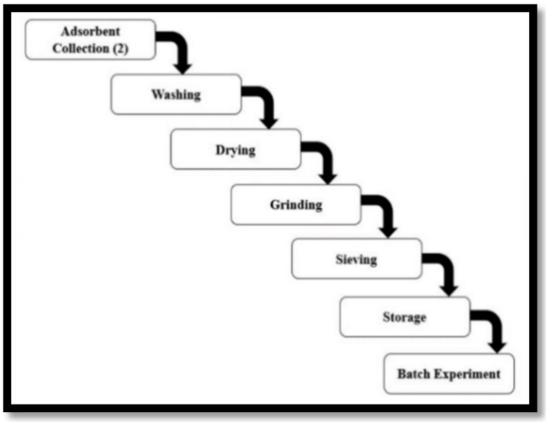


Figure 2. Flowchart of Adsorbent Preparation.

2.2 Preparation of Biochar using Bench-Scale Fixed Bed Reactor (B-SFBR).

Biomasses described in table 1 were used in the study. Preparation of adsorbent was done as mentioned in Fig 2 and biomasses are named in table 1. Pyrolysis was executed in a stainless-steel B-SFBR having an internal diameter of 10.8 cm and length of 50.4 cm. Fiber insulated with ceramic was draped all over the reactor for good temperature control which is optimum for pyrolysis process. To control the direct heat that reached to the biomass from the metal surface, a tough mica (silica-based) was used at the bottom of the insulated blanket. Mica sheets have the advantage of long lifetime of insulating material having efficient and better temperature management. Ice-bath and solution of NaCl were used to decrease and lower the temperature of the condenser. Furthermore, to collect biooil and to check the flow of nitrogen gas, a 250 mL Erlenmeyer flask was used, which has a rubber stopper to avoid the release of biooil in the working area. For the control of reactor temperature and to regulate the heating rate, Proportional-Integral-Derivative (PID) controllers were used. Pressures of 0.6 psi were used at the start for nitrogen purging. After purging, the pressure was reduced to 0.1 psi at all times of making biochar. For each batch test, a 100 g sample was put into the reactor's core before the start of the experiment through the top flange ofthe reactor. Thereafter, the reactor was airtight, and leakage was tested through a soap bubble test. Formerly heating the reactor flooded with N2 gas at the stream rate of 500 cm^3 /min. for 30 min to make sure anoxic in the interior atmosphere to prevent combustion of sample. The pyrolysis reactor was heated up to a prearranged temperature of 550°C to complete Thermo-Gravimetric Analysis (TGA) experiment with heating rate of 50°C/min. As the current study investigates, the pyrolysis of PSP (Peanut Shell Powder) and SBP (Sugarcane Bagasse Powder) have high thermal strength and the product is biochar.

Figure 3. (A) Raw Sugarcane Bagasse (SB), (B) SB Powder (C) SB after pyrolysis.

Figure 4. (A) Raw Sugarcane Bagasse (SB), (B) SB Powder (C) SB after pyrolysis.

2.3 Preparation of Modified / Activated Biochar (Adsorbent)

Activation / modification was done using FeCl3 which reacts with oxygen and makes iron oxide which is a well-accepted method for the modification of adsorbent (biochar). Iron oxide has a hydroxylated surface which has a high functional group of -OH and provides high adsorption capacity for metal removal like Cr^{6+}. For modifications iron having oxidation state of II and IIIare well used, Fe^{3+} is preferable. FeCl3 has a powerful buffering capacity. It provides H^+ ions in solution and makes colloids of ferric hydroxide. Fe^{3+} oxide/hydroxide colloid has high chromium removal efficiency at neutral pH [31]. Iron chloride is a conventional chemical reagent, that has robust buffering capacity. For activation, 10 g of biochar (PSP and SBP) were mixed with 500 mL FeCl3 solution with constant stirring at 70℃ for 2 h.

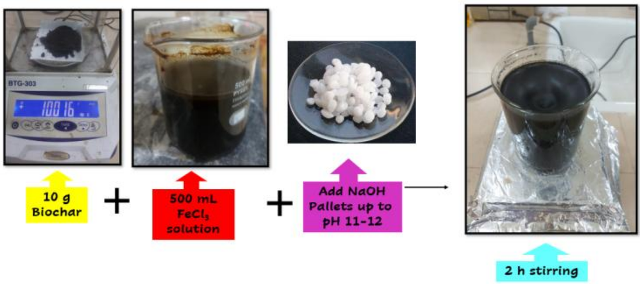


Figure 5. $FeCl_3$ Impregnation.

This solution is then washed and filtered using cloth to remove iron from the solution and to maintain pH at neutral. Filtered solution is now placed in a crucible and kept in at 90℃ for drying. During the formation of FeCl3 solution, pallet of NaOH was added to lower pH, filtration was done using Fabric having micropores, as washing and then squeezing Fe using cloth can remove iron in a better way. Washing was done using DI water up to 11-15 times to lower the pH of the adsorbent to neutral. The pH of filtrate was adjusted to 6 - 8 during modification. It was believed that hydrolysis of Fe salt would appear during the impregnation of Fe^{+3} ($Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + H^+$) (Wang et al., 2017). Colorimetric method was used to find the concentration of Cr. Colour development had been observed by adding up 0.25 mL (5 drops) of H_3PO_4 and maintaining p H of solution up to 2 or less than 4. Now transfer this solution in a 100 mL volumetric flask and add 2 mL of DPC solution and set it for 4-5 minutes for complete dissolution. The absorbance value was taken at 540 nm using 1cm glass cuvette.

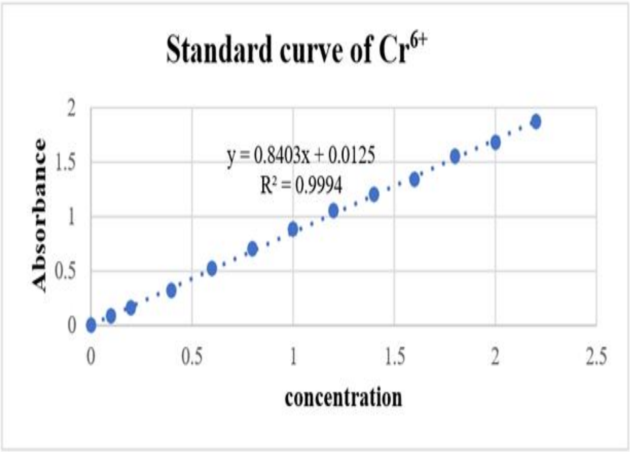


Figure 6. Standard Calibration Curve.

2.4 Adsorption Experiment of Activated Carbon

Batch experimentation for Cr^{6+} was done with concentration of 20 mg/L which is selected randomly. Removal was done by taking 10 mL of Cr^{6+} from stock solution in 250 m L Erlenmeyer flask with 0.3 g of Activated Carbon and fill distilled water up to 100 mL. The flask was entirely hermetically sealed and stirred at speed of 150 rpm at room temperature in shaker, for 10-180 minutes. Residual concentration of Cr^{6+} in the self-made contaminated aqueous solution was filter by vacuum filtration assembly and analyzed using UV spectrophotometer (Specord 200 plus Analytikjena Germany) with adsorption at 540 nm.

The removal percentage was calculated using the following equation.

$$\% \text{ removal} = \left(\frac{C_0 - C_f}{C_o}\right) 100$$

Here C_0 and C_f indicate the initial and residual metal concentrations (mg L^{-1}) respectively.

3. Result and Discussion

3.1 Effect of time on Cr^{6+} removal

Batch experiment was conducted with fixed variables as Cr^{6+} concentration of 20 mg/L, adsorbent dosage of 0.3 g, having pH 2 with time variation 10 – 180 minutes. Highest removal was observed at 180 min with removal efficiency of 98.96 by FeCl3 modified sugarcane bagasse. Table 3.1 is the removal percentage graph.

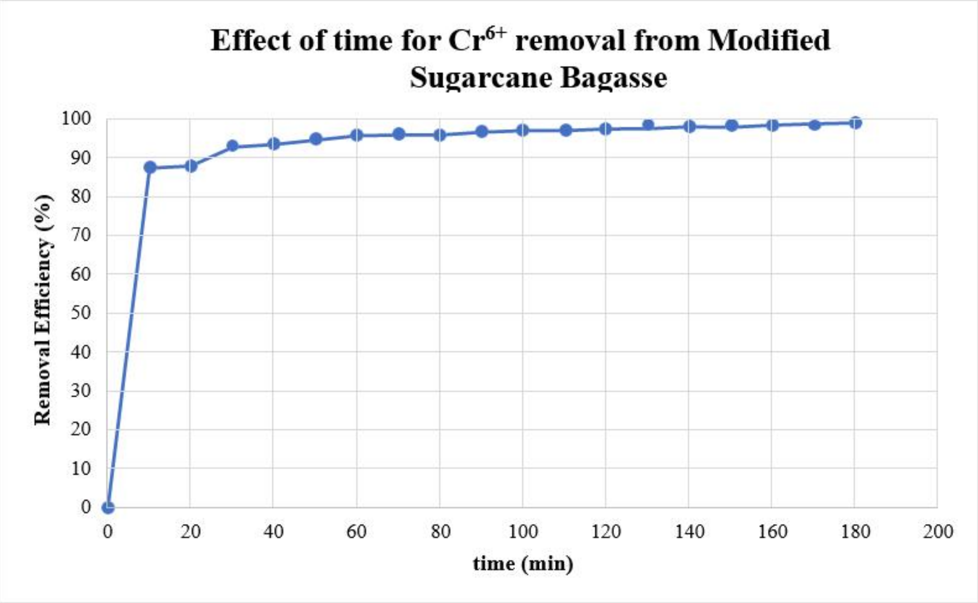


Figure 7. Effect of time for Cr^{6+} removal using Modified SBP (Adsorption conditions: T 28℃, Cr^{6+} conc 20 mg/L, dosage 0.3 g, pH 2).

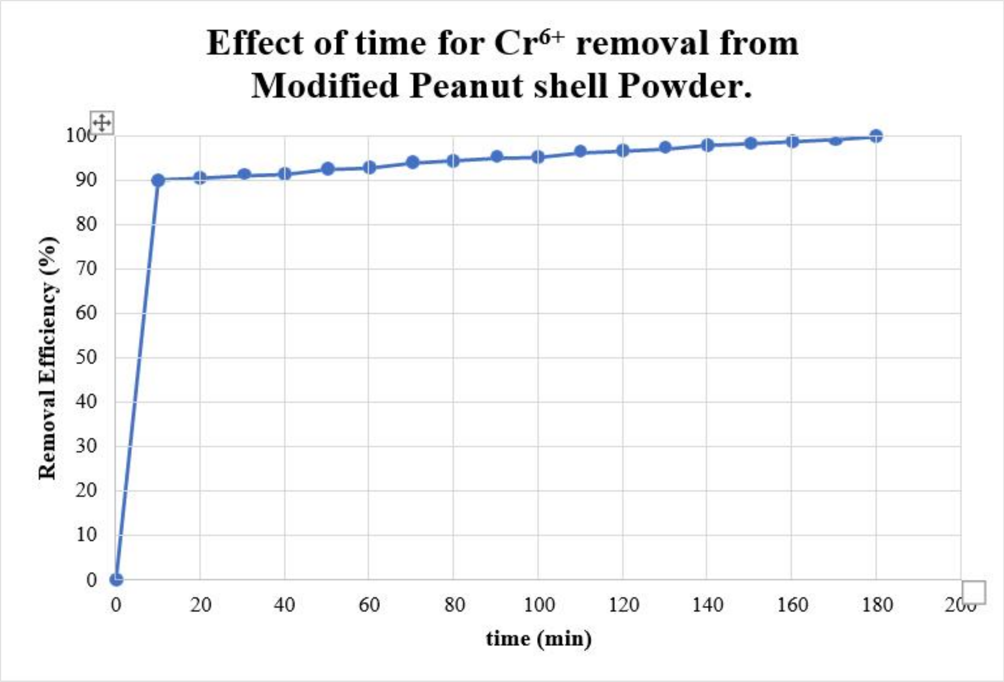


Figure 8. Effect of time for Cr^{6+} removal using Modified PSP (Adsorption conditions: temperature = 28℃, Cr^{6+} conc = 20 mg/L, absorbent dosage = 0.3 g, pH = 2.

Batch experiment was conducted with fixed variables as Cr^{6+} concentration of 20 mg/L, adsorbent dosage of 0.3 g, having pH 2 with time variation 10 – 180 minutes. Same sample treatment was applied with Modified Peanut Shell Powder (PSP) with absorbent dose of 0.3 g at pH 2, Cr^{6+} concentration of 20 mg/L and variable time of 10 - 180 minutes. Following is the removal percentage graph of APSP. Highest removal was observed at 180 min with removal efficiency of 99.97 by $FeCl_3$ modified peanut shell powder.

3.2 Thermal Analysis

TGA explained the thermal degradation performance of PSP and SBP by thermal analyzer was used for this analysis.

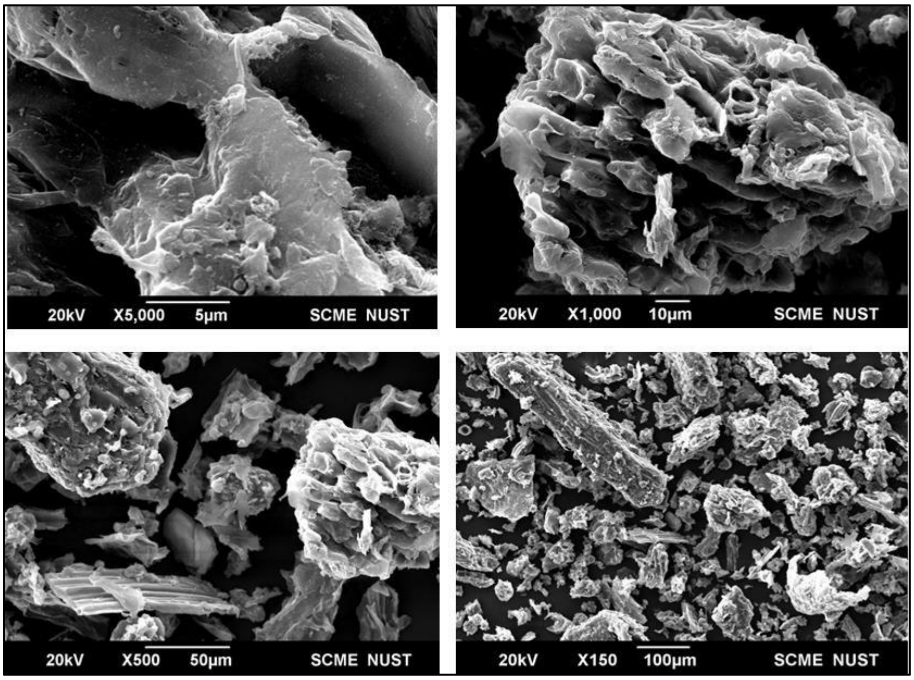


Figure 10. SEM images of Raw Peanut Shell Powder with variation of resolutions.

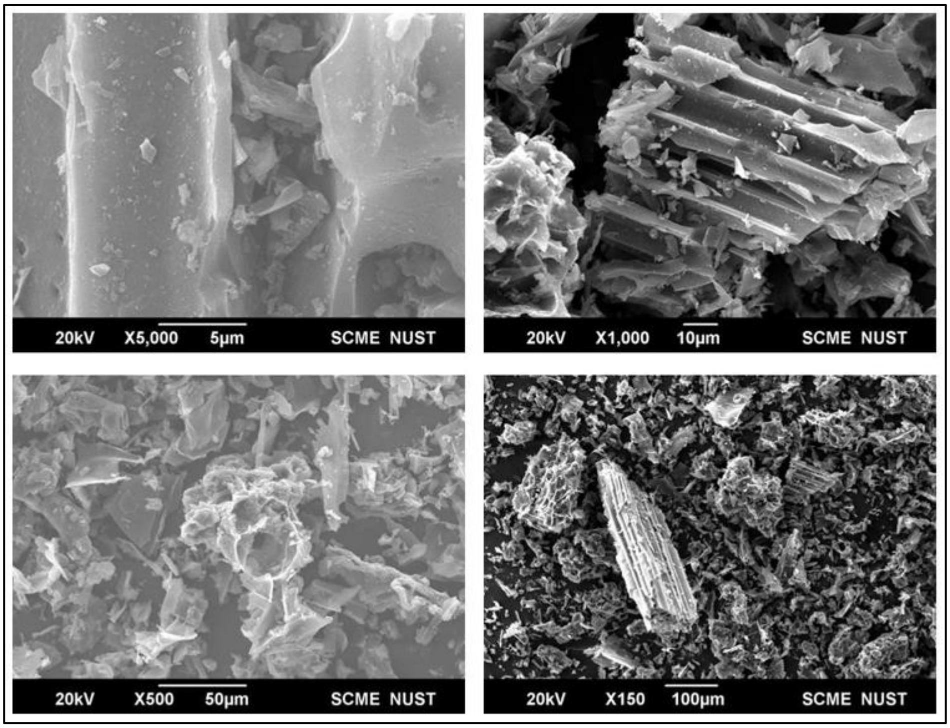
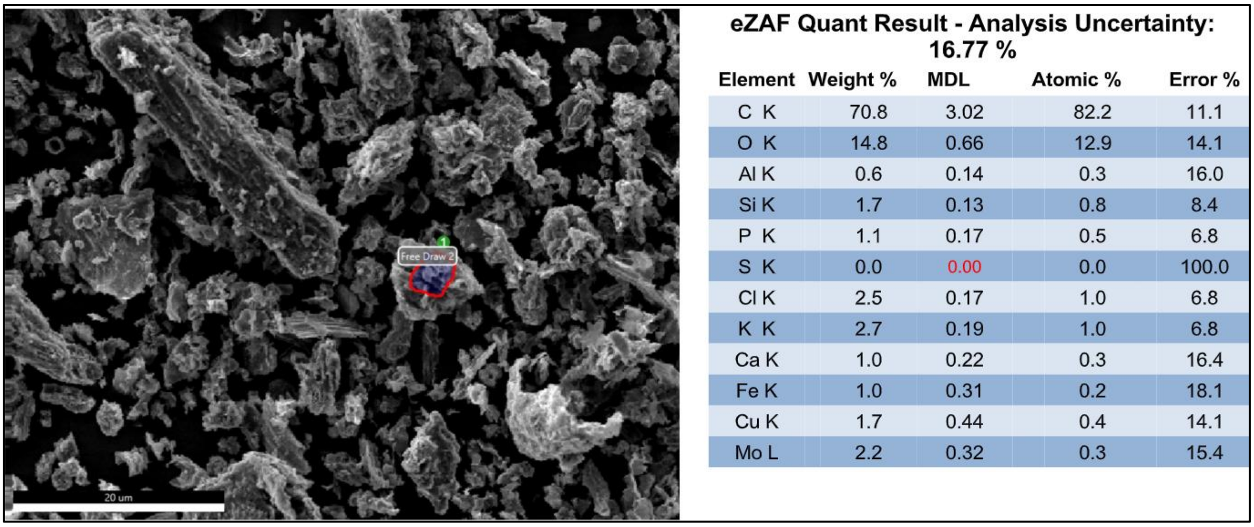


Figure 11. SEM images of Raw Sugarcane Bagasse Powder with variation of Resolutions.

Sample was examined at three heating rates (10, 20 and 30 °C/min) from room temperature up to 550°C in high purity N_2 gas with the flow rate of 200 mL/min was used for creation of inert atmosphere in system. Replicate analysis was performed thrice and then mean values were reported, with approximate error of ± 0.5 in absolute value.

Both green waste exhibit different spectra. Around 100 mass loss is responsible for decay of extractives, breaking solid biomass, moisture removal (referred to figure 9) [31].



eZAF Quant Result - Analysis Uncertainty: 16.77 %

Element	Weight %	MDL	Atomic %	Error %
C K	70.8	3.02	82.2	11.1
O K	14.8	0.66	12.9	14.1
Al K	0.6	0.14	0.3	16.0
Si K	1.7	0.13	0.8	8.4
P K	1.1	0.17	0.5	6.8
S K	0.0	0.00	0.0	100.0
Cl K	2.5	0.17	1.0	6.8
K K	2.7	0.19	1.0	6.8
Ca K	1.0	0.22	0.3	16.4
Fe K	1.0	0.31	0.2	18.1
Cu K	1.7	0.44	0.4	14.1
Mo L	2.2	0.32	0.3	15.4

Figure 12. Elemental Presentation of SEM-PSP.

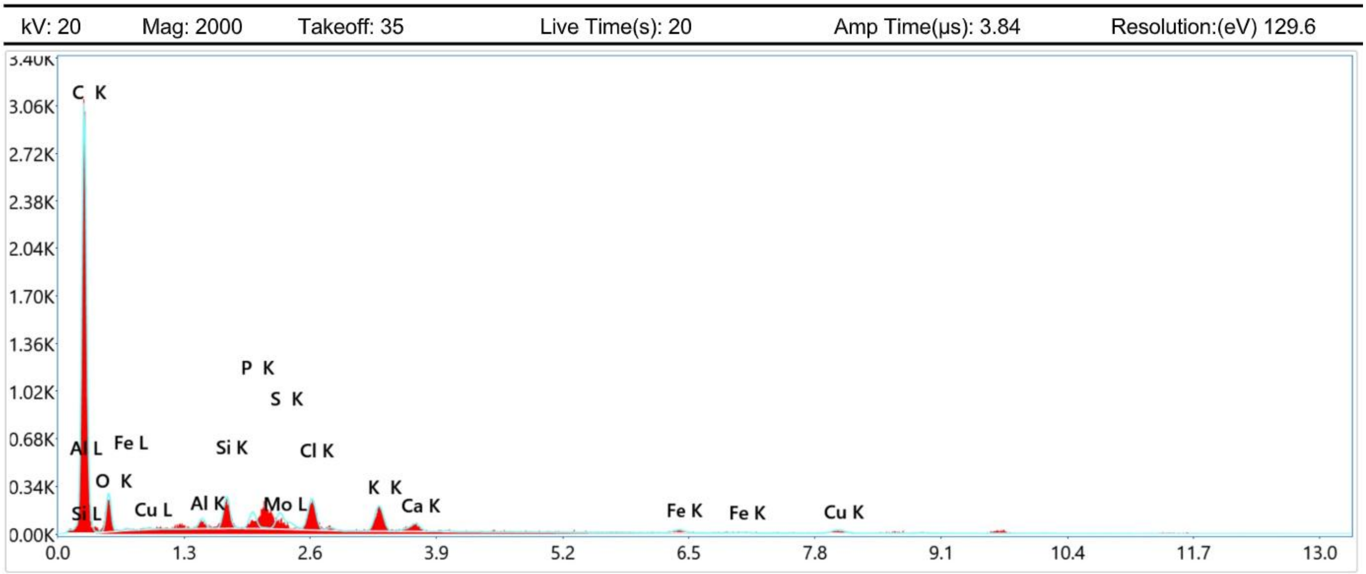
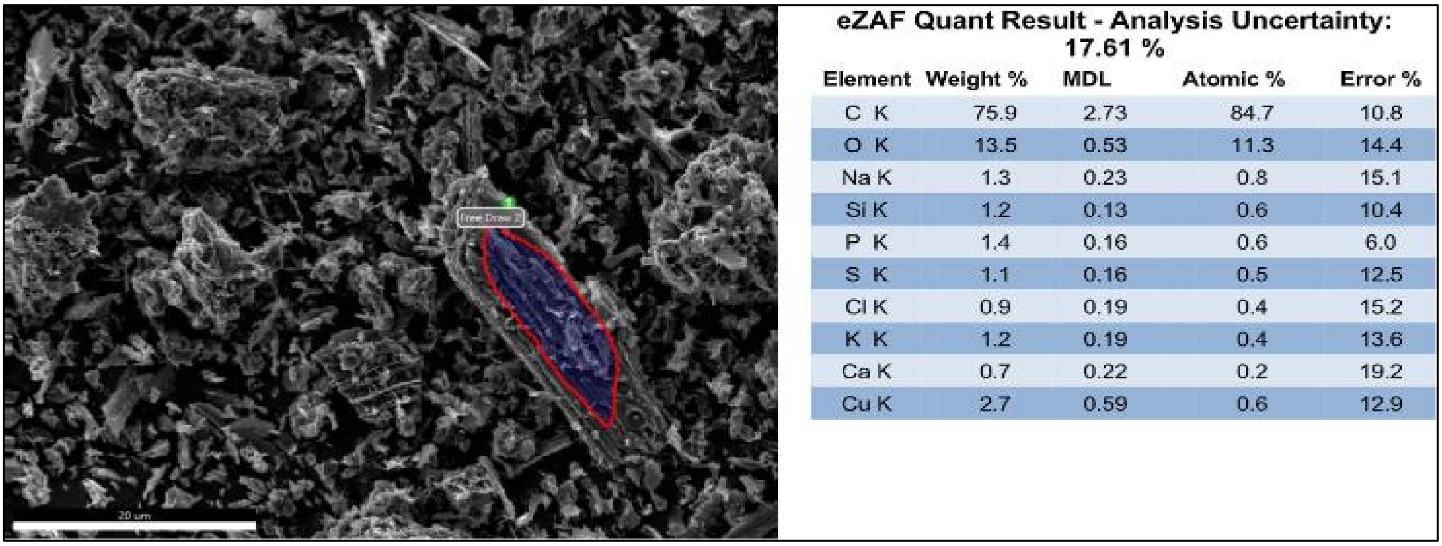


Figure 13. Energy Dispersive X-Ray Analysis of PSP.



eZAF Quant Result - Analysis Uncertainty: 17.61 %

Element	Weight %	MDL	Atomic %	Error %
C K	75.9	2.73	84.7	10.8
O K	13.5	0.53	11.3	14.4
Na K	1.3	0.23	0.8	15.1
Si K	1.2	0.13	0.6	10.4
P K	1.4	0.16	0.6	6.0
S K	1.1	0.16	0.5	12.5
Cl K	0.9	0.19	0.4	15.2
K K	1.2	0.19	0.4	13.6
Ca K	0.7	0.22	0.2	19.2
Cu K	2.7	0.59	0.6	12.9

Figure 14. Elemental Presentation of SEM-SBP.

PSP (Arachis hypogea) exhibit more mass loss, it starts to degrade at 200℃ and complete mass loss occurs at 530℃. Maximum weight loss was done during the temperature range of 300 - 500℃. Whereas SBP (Saccharum Officinarum) completed degraded at 569℃ and major weight loss occurs at 300℃ – 400℃

3.3 Characterization of Adsorbent.

3.3.1 Scanning Electron Microscopy (SEM)

SEM is an analytical technique which has its great importance in any research upon structure. As shown in the figure 10-17, the morphology of raw Peanut shell powder and bagasse is very rough. Fibers had irregular shaped as it is natural raw material. Elemental analysis proves impregnation of Fe on the biochar surface, same results was observed in [12].

3.3.2 Energy Dispersive X-ray spectroscopy (EDX)

Morphological study can be studied by SEM analysis JEOL- JSM-6490A Analytical Scanning Electron Microscope (USA) reveals the composition of biomasses. The surface of biomasses was rough and porous. EDX of FeCl3 modified shows the presence of iron attached with biomass. The peak of Au appears, it was used for coating on samples during elemental analysis represented in figure 10-19.

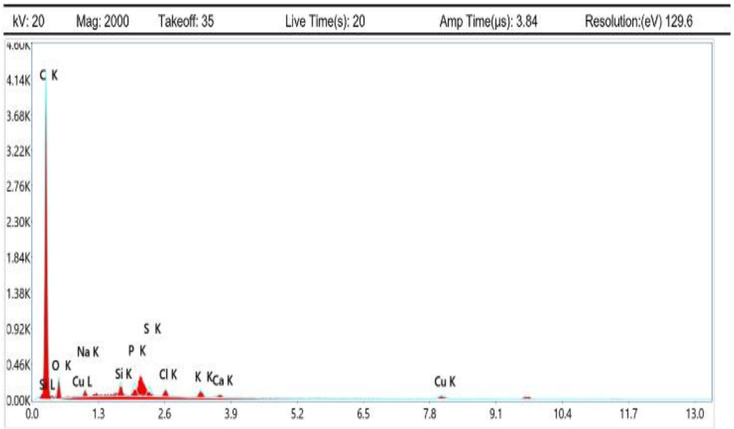
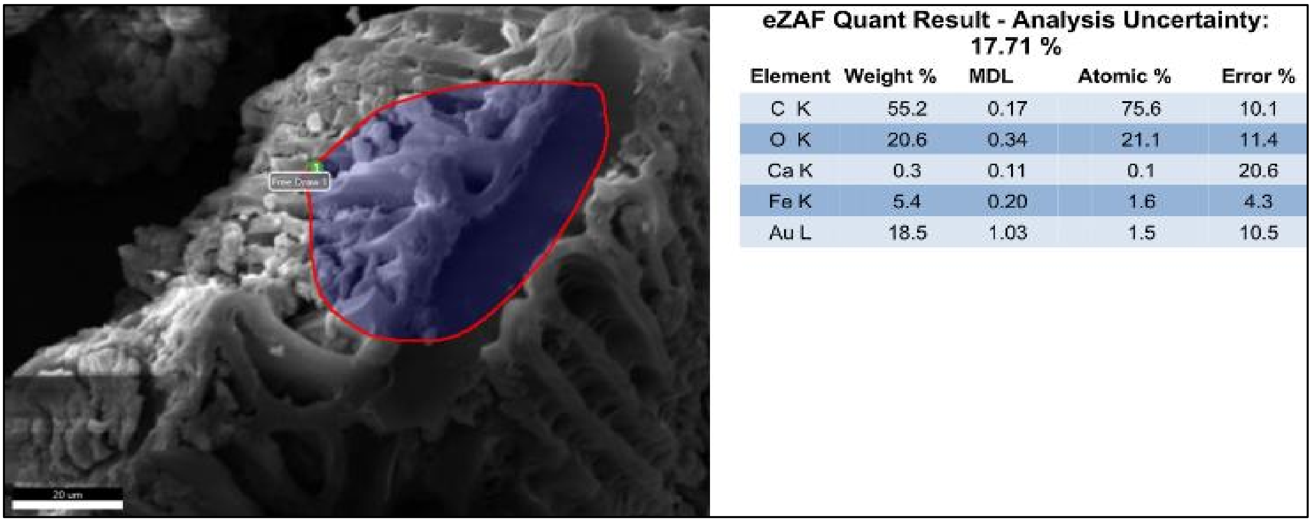


Figure 15. Energy Dispersive X-Ray Analysis of SBP.



eZAF Quant Result - Analysis Uncertainty: 17.71 %

Element	Weight %	MDL	Atomic %	Error %
C K	55.2	0.17	75.6	10.1
O K	20.6	0.34	21.1	11.4
Ca K	0.3	0.11	0.1	20.6
Fe K	5.4	0.20	1.6	4.3
Au L	18.5	1.03	1.5	10.5

Figure 16. Elemental Presentation of Modified PSP.

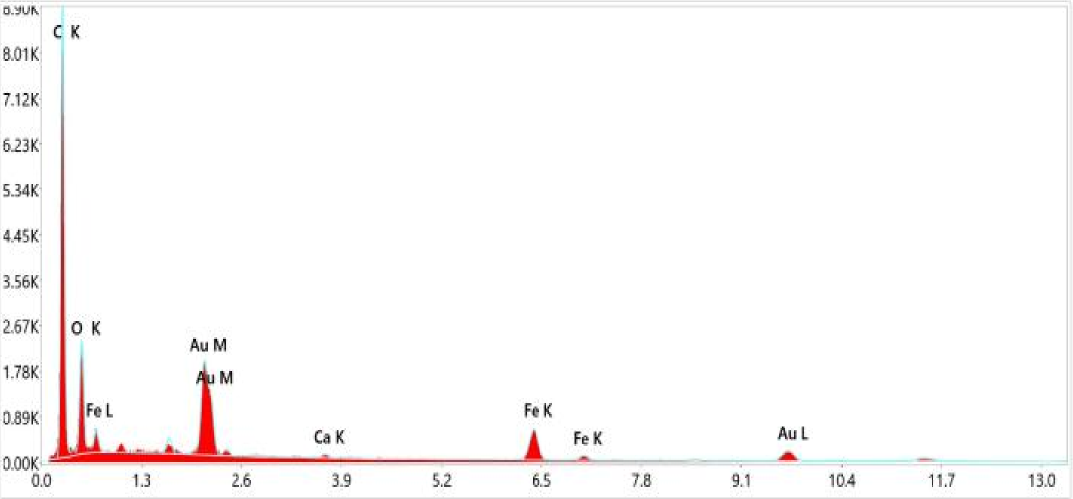
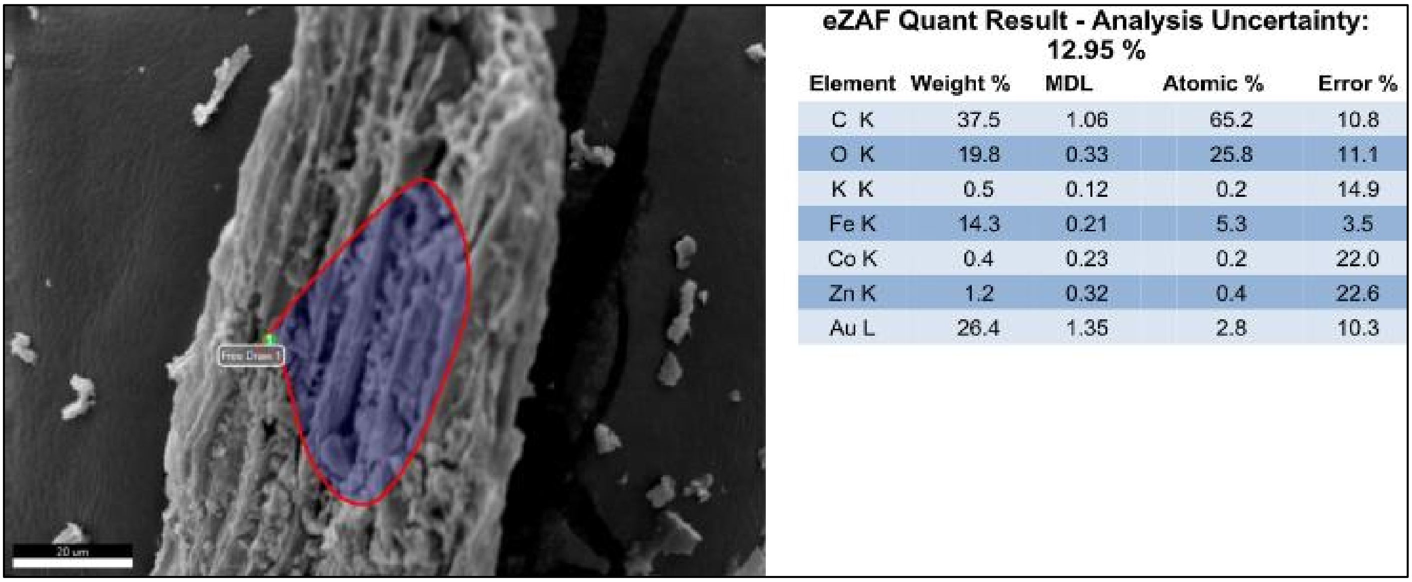


Figure 17. $FeCl_3$ Modified PSP.



eZAF Quant Result - Analysis Uncertainty: 12.95 %

Element	Weight %	MDL	Atomic %	Error %
C K	37.5	1.06	65.2	10.8
O K	19.8	0.33	25.8	11.1
K K	0.5	0.12	0.2	14.9
Fe K	14.3	0.21	5.3	3.5
Co K	0.4	0.23	0.2	22.0
Zn K	1.2	0.32	0.4	22.6
Au L	26.4	1.35	2.8	10.3

Figure 18. Elemental Presentation of Modified SBP.

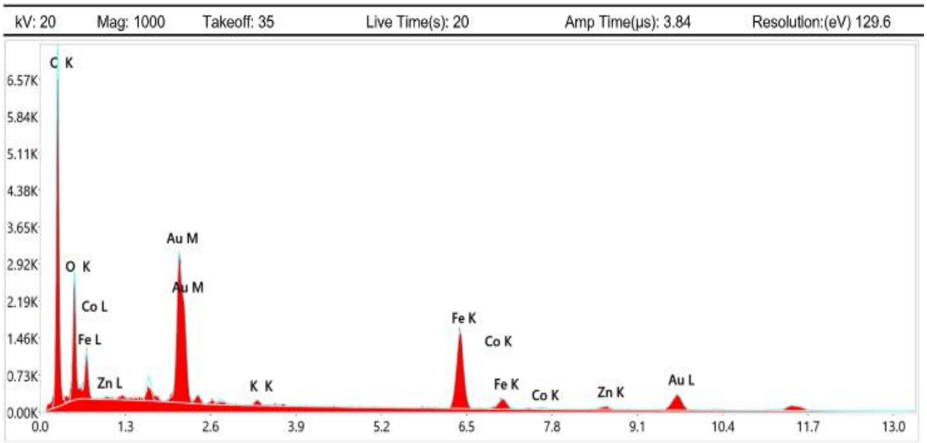


Figure 19. $FeCl_3$ Modified SBP.

3.3.3 BET Analysis

The BET (Brunauer, Emmett, and Teller) analysis is generally used to estimate the gas adsorption data and generate a specific surface area result which is expressed in units (m^2/g). This analysis was done using Quanta chrome Nova Win Instrument version 11.05 (Russia). The result of BET analysis shows that the value of raw and $FeCl_3$ modified peanut shell powder are 39.830 m²/g and 10.1157 m²/g, respectively, and raw sugarcane bagasse and $FeCl_3$ modified have 25.268 m²/g and 18.7709 m²/g respectively. The decrease in surface area for modified biomass was due to using ferric chloride which blocks the pores of biochar [32]. The surface area of peanut shell powder, sugarcane bagasse, modified or un-modified is shown in table 2. As biochar is negatively charged, and iron has positively charged that is why the removal efficiency was 99.97% using $FeCl_3$ activation method [33].

Table: 2. Surface area Analysis.

Sample	Single point SA (m²/g)	BET-SA (m²/g)	Pore Volume (cm³/g)	Pore Radius (Å)
Raw PSP	53.2132	39.830	0.054	16.044
$FeCl_3$ PSP	10.0048	10.1157	0.005144	21.101
Raw SB	49.6679	25.268	0.036	16.155
$FeCl_3$ SB	18.5476	18.7709	0.0134	29.7992

SA: Surface Area, PSP: Peanut Shell Powder, SBP: Sugarcane Bagasse

3.3.4 FTIR

Fourier Transform InfraRed (FTIR) technique applied to examine chemical structure and to validate the functional groups from adsorbent surface. For acquiring absorption spectra, all samples were furthermore grounded to roughly 0.5 wt.% with spectroscopic grade of KBr powder for homogenization and pallet formation. Discs (12.7 mm I.D and about 1 mm in thickness) were prepared using manual hydraulic press at 1000 psi (parts square per inch) for a pressing time of 0.5 min. The acquired result is shown in Fig 18 and analysis was done using Perkin Eimer Spectrum 100 FT-IR Spectrometer (USA) with 400-4000 cm^{-1} with a resolution of 2 cm^{-1}. Here a broad peak appears at 3000-3700 cm^{-1} which unveils -OH group of ketone or aldehyde [33]. The peak 2850-2930 represents $-CH_2$ aliphatic hydrocarbons vibrations and C-H group [34]. Sharp intense peak observed at 1602 cm^{-1} is due to lignin aromatic rings of C=O and C=C. Intense peak in the region 1030-1364 was due to stretching C-O of lignin, hemicellulose, and cellulose. Minor peak at 472 is due to Fe modification, refers to figure 20 [32].

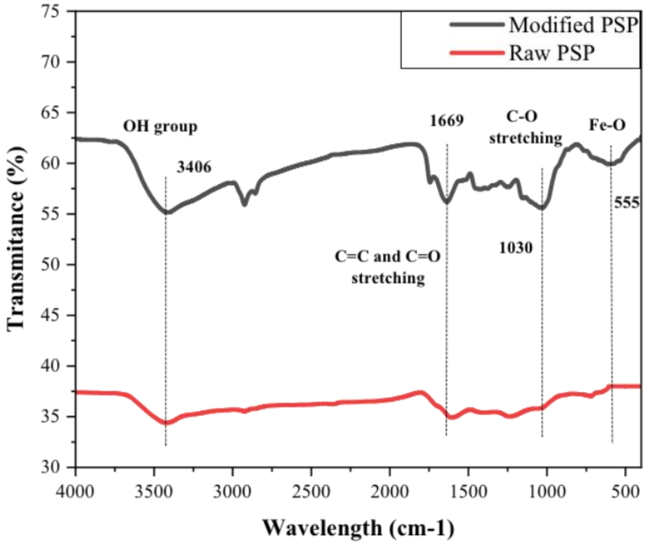
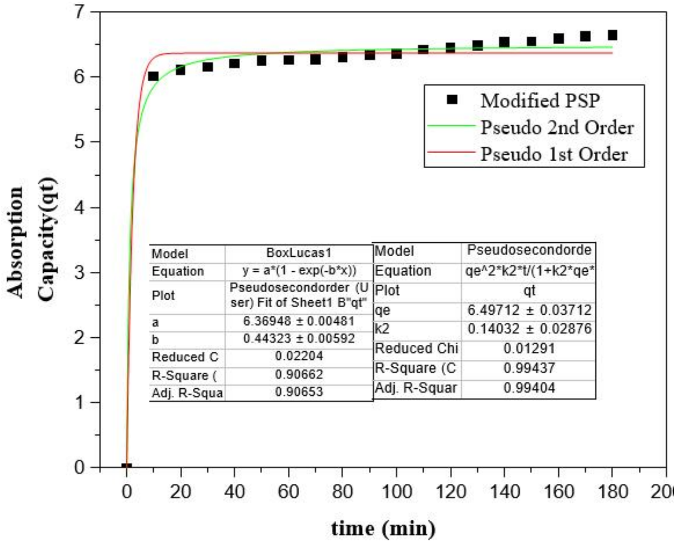


Figure 20 FTIR graph of raw SB and $FeCl_3$SBP.



Model	BoxLucas1
Equation	y = a*(1 - exp(-b*x))
Plot	Pseudosecondorder (User) Fit of Sheet1 B"qt"
a	6.36948 ± 0.00481
b	0.44323 ± 0.00592
Reduced C	0.02204
R-Square (	0.90662
Adj. R-Squa	0.90653

Model	Pseudosecondorde
Equation	qe^2*k2*t/(1+k2*qe*
Plot	qt
qe	6.49712 ± 0.03712
k2	0.14032 ± 0.02876
Reduced Chi	0.01291
R-Square (C	0.99437
Adj. R-Squar	0.99404

Figure 21. Pseudo 1st and 2nd order kinetics for Modified Peanut Shell Powder

3.4 Kinetics Isotherms

Kinetic Isotherms of Pseudo first order and second order were made using Origin Pro software figure 21-22. To explain kinetics adsorption of Chromium models of Pseudo 1st order and 2nd order were used, models were drawn in Origin software using following equations:

$$Q_t = Q_e (1 + e^{-k1t})$$

$$Q_t = k_2Q^2_et / 1 + k_2Q_et$$

Qe (mg/g) is an adsorbed metal at equilibrium. dynamics constants are k1 (min^{-1}), k2 (g/mg·min). For both models, the value of R2 and Chi-Square indicate error parameters. Equilibrium stage attain after 60 mints and best removal was at 160 mints.

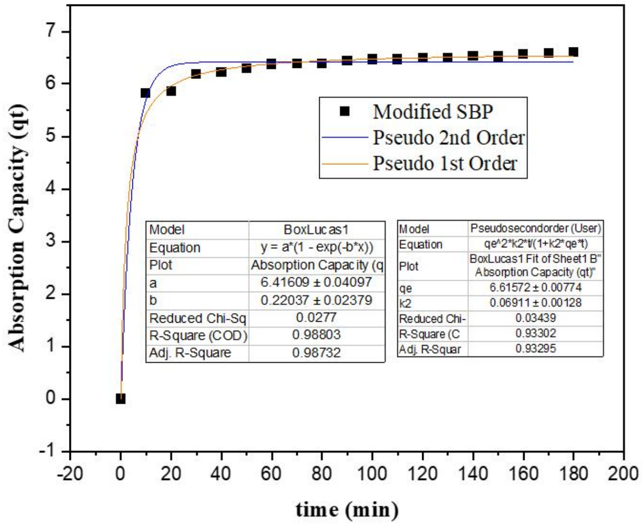


Figure 22. Pseudo 1st and 2nd order kinetics for Modified SBP

4. Conclusion

This is a relatively cheap technological solution for removal of Chromium heavy metal from water. $FeCl_3$ modified biochar was made using peanut shells and sugarcane bagasse. Comparative experimental study successfully reveals that carcinogenic heavy metal can be removed using $FeCl_3$ modified green waste biomasses up-to 98% as compared to modified sugarcane bagasse. To prevent Cr^{+6} water pollution this adsorption-based phenomenon is of great importance and uses waste as raw material. For this purpose, two different biomasses i.e., PSP and SBP were collected which are abundantly available in all over Pakistan and used for Cr^{+6} adsorption from synthetic polluted water. Removal percentage increases with time and was maximum at 180 mints with Cr^{+6} concentration of 20 mg/L, pH 2 having adsorbent dose of 0.3 g and shaking speed 150 rpm. Hence, conclude that Peanut shells can be used for obtaining 99.99% Cr removal if modified by $FeCl_3$.

Author Contributions

Dr. Said Akbar Khan (S.A.K); Methodology, Conceptualization Hijab Zehra (H.Z).; Formal analysis, H.Z.; Writing—original draft preparation, Mahnoor Zehra (M.Z).; Writing—review and editing. Supervision, S.A.K. All authors have read and granted to the published this version of manuscript.

Conflicts of Interest

There are no conflicts of interest reported by the writers.

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Data Availability statement

All the data which is presented in this article can be given on request from the first author.

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