Biosorption study of Cr (VI) from the aqueous solution using chemically modified biomass of a newly isolated edible mushroom waste

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Abstract

Despite some physiological role in human body, some metals, like hexavalent chromium, have no recognized biological benefit and are poisonous above tolerance limits. For the first time, a widely available Pleurotus sajor caju *(fungus) biomass was studied for the Cr (VI) adsorption from an aqueous solution involving physisorption, and chemisorption. Using the batch adsorption method, sorption experiments were conducted, and sorption parameters such as pH, contact time, adsorbent dose, and starting metal ion concentration were studied. The sorbent material was shown to remove up to 91.67% of the Cr (VI) at a pH value of 2. It was discovered that the maximum adsorption (q_{max}) was 239.98 mg/g. The Freundlich model was adopted for the equilibrium adsorption isotherm for Cr (VI). The biosorption kinetics was ascertained by fitting pseudo-first order and pseudo-second order kinetic models for the experimental data and the pseudo-second order model was found to better describe the data. The interaction of metal ions with the carboxyl, hydroxyl, and carbonyl groups present in biomass is primarily involved in metal ions uptake. This demonstrated the potential application of mushroom residues as a low-cost and promising bio-sorbent.*

Keywords

Bio-adsorbent; Heavy metals; Adsorption; Batch study; Water security.

1 Introduction

The aquatic environment is facing exceptional challenges and become one of the primary global concerns because of abandoned water pollution and unintended water security [1]. Aquatic pollution knowingly affects the water quality, visibility, and water reaction mechanisms including the lack of dissolved oxygen, biodegradation processes, and photosynthesis [2]. Heavy metals, dyes, and hazardous pigment effluents discharged from different sources like battery, electroplating, textile, pesticide, fertilizer, cosmetics, etc. contaminate the water sources which pose a warning to public health and ecosystems [3]. As opposed to organic contaminants, most of the heavy metals are toxic, carcinogenic, non-biodegradable and accumulate in living tissues through the food chain, causing severe damage of internal organs [4]. Thus, great attention regarding the elimination of toxic heavy metals, dyes, pigments, etc. is required using a novel, low cost, and feasible technology and material.

Specifically, Chromium is considered as a major pollutant in aquatic environment owing to its extensive use in leather, electroplating, ceramics, and other industries [5]. Mostly, the Cr(VI) forms like $HCrO_4^-$, CrO_4^{2-}, and $Cr_2O_7^{2-}$ are strongly oxidizing and are predominantly found in surface water and aerobic soils species [6,7]. The Cr(III) is essential element for the human body and other living organism however the Cr(VI) is 5-10 times toxic than Cr(III) and is classified among the top pollutants and listed as the 16th dangerous contaminant [8,9]. Due to high solubility, the Cr(VI) intake typically occurs through contaminated food, leakage, sea foods, skin or respiratory tract and can easily enters in stomach, kidney, and liver. The health issues related to Cr(VI) include the risk of lung, nasal, sinus cancer, ulcer, anemia, damage to sperm, detriment to the male reproductive system, asthma, abdominal pain, eye irritation, etc. [10,11].

The adverse health effects of Cr (VI) compelled us to solve the problem by finding a suitable method of removal from aquatic environments. Remediation of heavy metals from aqueous solutions is a topic of significant interest in environmental research. Among many, reverse osmosis, flotation, activated carbon adsorption, ion exchange, chemical reduction, precipitation, electrodialysis, and some traditional methods are in common practice to remove heavy metals from water and wastewater [12]. These are just a few examples of the methods used for the remediation of heavy metals from aqueous solutions. Researchers continue to explore innovative technologies and materials to improve the efficiency and cost-effectiveness of heavy metal removal processes. However, the mentioned approaches are typically costly and ineffective, especially at low metal ion concentrations between 1 and 100 mg/L [13].

Adsorption is a widely used method for the removal of heavy metals from water having low concentration of metal ions. Materials like activated carbon, clay minerals, zeolites, and biosorbents are commonly used for adsorption due to their high surface area and adsorption capacity [14]. The solid-phase byproduct of biomass pyrolysis, biomass carbon, has gained attention as a possible material to treat wastewater due to its rich functional group sites and better developed pore structure. Researchers have developed cutting-edge techniques including physicochemical activation, metal loading, and organic/inorganic modification to bring biochar from the lab to the commercial sector. These techniques have produced good results in terms of increasing specific surface area, optimizing the structure of the pore layer, and changing functional groups, all of which significantly improved the pore structure and surface functional groups [15].

Consequently, it is important to design a stable, earth-abundant, inexpensive, and highly effective adsorbent for water purification and heavy metal decontamination process, such as bio-adsorbent [16]. A known biotic mechanism cannot dissolve toxic heavy metals from water. Biosorption is a very common physiochemical mechanism that involves the adherent of atoms, ions, or molecules of dissolved solids from liquid to the surface of solid materials derived from organic sources [17]. Principally, the biosorption process involves a simple chemistry where the heavy metal ions in aqueous solution bound on the surface of an adsorbent due to an imbalance between two environments creating a driving force to be adsorbed onto adsorbent. The process continues till phase equilibrium is established between the amount of solid-bound adsorbate species and its fraction left behind in the solution [18]. Activated carbon, seaweed, microorganisms, fermented waste, and some other generated biomasses are only a few examples of the many diverse types of bio-sorbent sources that are defined by their wide source, cheap cost, and effective adsorption capability [16–19]. Moreover, mushroom-based biomasses are potent for the bioremediation of several heavy metals, dyes, organic pollutants, etc. The hemicellulose fungal cell wall, chitin, chitosan, polyuronide, polyphosphates, lipids, and proteins are the primal components of the mushroom fungus that portray the superior metal binding abilities [20,21]. Studies demonstrate the use of mushrooms and fungal biomass as effective biosorbents for the removal of various heavy metals from aqueous solutions and researchers are continuously exploring novel approaches and technologies to enhance the efficiency and sustainability

of heavy metal remediation processes using mushrooms [22–24].

Specifically, for the bioremediation of hexavalent chromium, fungal derived adsorbents are widely used, demonstrating effective removal from aqueous solutions. The mushroom substrate shows that it has an exceptional ability to adsorb Cr (VI) from water. The fungus species' cell wall contains chitin, which contributed to the likely binding of the Cr (VI) ions. Fungal cell walls are made up of 80–90% heteropolysaccharides, proteins, lipids, polyphosphates, and inorganic ions that act as adhesive material to create the walls. A common component of fungal cell walls that has the capacity to complex metal ions is chitin. Also many polysaccharides found in fungal cell walls are crucial for metal binding. It has also been discovered that some functional groups, specifically carboxyl groups, have the capacity to bind metal ions [25]. The potential of mushroom-derived waste biomaterials for the adsorption and extraction of heavy metals that are harmful from wastewater is interesting. Significant recycling potential is also accessible for the waste fungal biomass adsorbent that is created. following five cycles of adsorption and regeneration. Up to 85.5% of Cr (VI) ions are still able to be eliminated with good effectiveness [26].

Through a series of batch adsorption experiments, the study explores the effectiveness of oyster mushrooms (*Pleurotus platypus*) for the removal of Cr(III) and Cr(VI) from aqueous solutions. The results demonstrate that the ligand exchange process and electrostatic force of attractions are the main causes of the adsorption processes [27]. Other several mushroom(fungus) species were reported to show great efficacy for the bioremediation of Cr(VI). *Aspergillus carbonarius* performed well with 92.43% removal of Cr(VI) [28].

In this work, we derived modified *Pleurotus sajor caju* waste-derived biomass and focused on the efficacy towards biosorption of toxic heavy metal, Cr(VI), from water bodies. The impact of modified and raw *Pleurotus sajor caju* waste-derived biomass to remove Cr(VI) from synthetic aqueous solution was accessed. The 0.1 M NaOH modified *Pleurotus sajor caju* waste-derived biomass powder showed an increased sorption capacity of positively charged metal ions (Cr^{+6}) onto its surface than raw biomass possibly due to the interaction of metal to hemi cellulosic cell wall components. As a result, the addition of NaOH in biomass enhances their ability of metal binding and mechanical strength as well. The sorption property was explored by analyzing the effects of contact time, adsorbent dose, pH, adsorption isotherm, starting concentration, and batch kinetics. The SEM, EDS, and FT-IR analysis describe the surface structure and basic chemical groups in the biomass. Hence,

this work suggests and found a strategic functionalization of *Pleutorous sajo-coju* waste as potential bio-adsorbent to amplify the basic understanding of adsorption in a real aquatic environment.

2 Materials and Methods

2.1 Materials

All the chemicals (Sulphuric acid, Buffer tablets, Potassium dichromate, 1,5-diphenyl carbazide, Nitric acid, Sodium hydroxide, etc) of analytical grade were purchased from Thermo Fisher Scientific India. 1000ppm stock solution of potassium dichromate, 0.1 N nitric acid, 5N sulphuric acid, 2N nitric acid, 0.1M & 2N sodium hydroxide, 0.25% DCPI solution and buffer solutions (pH-4.0,7.0 & 9.2) were prepared in different volumes.

2.2 Experimental

2.2.1 Preparation of raw and modified adsorbent

The mushroom was washed, shade dried, then ground to a mess size of 250 microns. A part of powered mushroom underwent 0.1M NaOH treatment, followed by three hours of reflux at 80°C. The pH of the final product was then kept neutral by washing with distilled water and adding nitric acid. For two hours, the mixture was then dried in an oven set at 80°C. The products were then used for the adsorption studies and FTIR was used to confirm the extent of modification [29, 30].

Modifying bio-adsorbents enhanced adsorption capacities, but it will increase the adsorbents' cost. Therefore, a cost-benefit analysis is required. The modifying chemicals used in this study may cause impacts on the environment so the proper discharge procedures were followed.

2.2.2 Determination of λ_{max} and calibration

Various volume of working Cr(VI) solution containing 20mg/mL were taken in 25 mL volumetric flask, acidified with 1 mL of 5 N H_2SO_4, followed by addition of 1 mL of 0.25% of DCPI solution and remaining volume with distilled water. The absorption spectra of pink colored Cr(VI)-DPCI complex were recorded against blank solution. Among various absorption spectra, λ_{max} was recorded at 530 nm, using calibration curve [31, 32].

2.2.3 Batch adsorption study

An Erlenmeyer flask is pipetted with a known concentration of metal solution that has been pH-adjusted, and the predetermined quantity of adsorbent is then added. The flask is shaken for the

specified amount of time to ascertain the concentration of adsorbate. Adsorbate adsorption is dependent on a number of factors, including pH, contact time, adsorbent dosage, metal solution concentration, etc. One of these parameters was changed while keeping the others fixed in order to understand the adsorption mechanism. The equilibrium adsorption amount (q_e) and the percent metal uptake by adsorbent were expressed [32, 33].

$$q_e = \frac{(Ci - Ce)V}{w} mg/g \qquad (1)$$

where, q_e is the amount of metal ion adsorbed per unit mass of adsorbent, w is weight of adsorbent (g), C_i is Initial concentration of adsorbent dose (mg/L), C_e is equilibrium concentration of adsorbate (mg/L), v is volume of adsorbate.

The ratio of the adsorbate ion concentration before and after adsorption to the initial concentration of metal ions in aqueous solution determines the adsorbate removal percentage.

$$\text{Removal percentage}(R) = \frac{Ci - Ce}{Ci} x100 \ \% \qquad (2)$$

2.2.4 Adsorption isotherm

The amount of adsorbate adsorbed per mass of adsorbent and the quantity of unabsorbed adsorbate remaining in the solutions at equilibrium time are related by a curve known as the adsorption isotherm. The most significant isotherms for analyzing the adsorptive behavior of an adsorbate and an adsorbent are the Langmuir and Freundlich adsorption isotherms.

2.2.4.1. Langmuir adsorption isotherm

The linear form of this isotherm is represented by the expression

$$\frac{1}{qe} = \frac{1}{q_m} + \frac{1}{q_m K_L C_e} \qquad (3)$$

where, q_e is maximum adsorption of bio-sorbent at equilibrium (mg/L), q_m is theoretical monolayer saturation capacity(mg/L), K_L is Langmuir equilibrium constant (L/mg).

Langmuir constant q_m and K_L can be obtained as slope ($\frac{1}{q_m}$) and intercept ($\frac{1}{q_m K_L}$) respectively from the linear plot of ($\frac{C_e}{q_e}$) Vs C_e. By utilizing a dimensionless constant separation factor or equilibrium parameter, the fundamental properties of the Langmuir adsorption isotherm can be used to estimate the affinity between the biosorbent and the biosorbate.

$$R_L = 1/1 + K_L C_i. \qquad (4)$$

2.2.4.2. Freundlich adsorption isotherm

The Freundlich adsorption isotherm assumes a heterogeneous adsorption surface and active sites with different energy. This isotherm can be explained by equation 5 [34].

$$q_e = K_F C_e^{1/n} \qquad (5)$$

where, K_F is Freundlich constant (L/g), n = Freundlich exponent (g/L).

The value of these factors affects the isotherm's curvature and steepness, while the value of n reveals the adsorbate's affinity for the adsorbent. This isotherm's linear form is written as:

$$\log(q_e) = log(K_F) + \frac{1}{n} \log(C_e) \qquad (6)$$

2.2.5 Adsorption kinetics

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2.2.5.1. Pseudo first order kinetic model

The pseudo first order kinetics proposed by Lagergren for adsorption analysis can be express in equation 7 [35].

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \qquad (7)$$

where, q_e is amount of Cr(VI) per gram on the surface of adsorbent at the equilibrium(mg/g), q_t is amount of Cr(VI)per gram on the surface of adsorbent at time 't'(mg/g), k_1 is equilibrium rate constant of pseudo-first order kinetics.

After integration and applying boundary condition, t = 0 to t = t, qt = 0, and $q_t = q_t$ the linear form of the equation (7).

$$loq(q_e - loq(q_t - q_t) = loq\ q_e - \frac{k_1 t}{2.303} \qquad (8)$$

K_1 is calculated from the linear plot of log (q_e-q_t) against time.

2.2.5.2. Pseudo second order kinetic model

The pseudo second order rate equation for adsorption analysis [36].

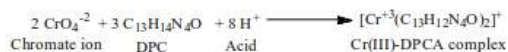$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \qquad (9)$$

After integration and boundary condition qt = 0 at t=0, qt = qt at t = t equation (9).

$$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e t}t \qquad (10)$$

where, k_2 is equilibrium rate constant for pseudo second order kinetics, k_2 and q_e can be calculated from the graph of t/q_t against t.

2.2.6 Spectrophotometric methods for the determination of Cr (VI)

DPCI method is quite suitable for the determination of Cr(VI) in aqueous solution. The absorbance of the solution measured at maximum wavelength is 530 nm.

$$2\,CrO_4^{-2} + 3\,C_{13}H_{14}N_4O + 8\,H^+ \longrightarrow [Cr^{+3}(C_{13}H_{12}N_4O)_2]^+$$

Chromate ion   DPC   Acid   Cr(III)-DPCA complex

2.2.7 Material characterization

The Fourier transform Infra-red (FTIR) spectroscopy was done at the Central Department of Chemistry, Tribhuvan University, Kathmandu. The Spectra of RPSC and MPSC were obtained with a BRUKER spectrometer model, using potassium bromide (KBr) pellet method. The surface morphologies of MPSC and RPSC samples were char-

acterized by using Field emission scanning electron microscope (FE-SEM, Model: Supra 40 V P instrument Zeiss Co., Germany) and energy dispersive X-ray spectroscopy (EDS) based elemental composition in the Center for University-Wide Research Facilities (CURF) of Jeonbuk National University, South Korea.

3 Result and Discussion

By adopting a most generalized and simple hydroxyl functionalization technique, the waste of edible mushroom *Pleutorous sajo-coju* was prepared as an active biomass for heavy metal (Cr^{+6}) removal from aqueous solution (**Scheme 1**, details in experimental section). On optimizing different physicochemical parameters such initial pH, initial adsorbate concentration, contact time, adsorbed dosage, and desorption research, the sorption of Cr (VI) in an aqueous solution was investigated based on raw and functionalized *PSC*.
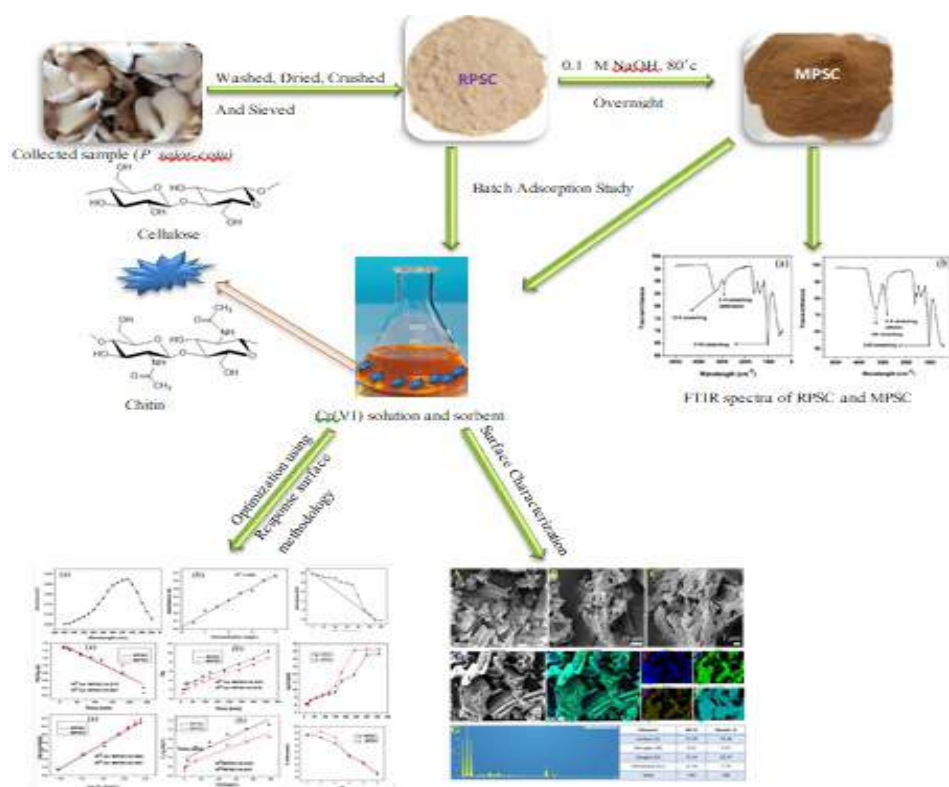
Scheme 1:Schematic representation showing preparation and modification of bio-adsorbent from Pluorotous sajor-caju (fungus), application for removal of Cr (VI) from aqueous solution and characterization.

3.1 Maximum absorbance and calibration curve study

The maximum absorption wavelength for Cr (VI) obtained at 530 nm was used to prepare calibration curve, shown in Figure (1 a, b).

3.2 Effect of contact time for Cr (VI) DPCI complex formation

The effect of contact time on adsorption of Cr (VI) at initial concentration of 50 mg/L were studied. The **Figure 2** showed the experimental data where

a rapid uptake of metal ion occurs initially and absorbance value was nearly constant from 0 to 20 minutes, the absorbance value within 20 minutes was calculated using DPCI solution. The relationship between time and absorbance is shown in **Figure 2**.

3.3 Effect of pH for absorption of Cr (VI) on to MPSC and RPSC

Figure 3 illustrates how pH affects the adsorption of Cr (VI) onto RPSC and MPSC. In the instance of RPSC, removal of Cr (VI) was found to be reduced as pH increased; the highest removal value was shown to be at 1.0, which is an optimum pH

for RPSC. However, the highest removal value for MPCS was shown to be 2.0, and this pH is optimum for MPSC.

3.4 Effect of contact time for adsorption of Cr (VI) onto RPSC and MPSC

The batch adsorption of a 20 mg/L metal solution with a 25 mg adsorbent was studied by assessing the impact of contact time over different time periods ranging from 10 minutes to 360 minutes. The impact of contact time on the adsorption of Cr (VI) onto RPSC and MPSC is shown in **Figure 4**. Adsorption was discovered to be expanding as time value has increased.
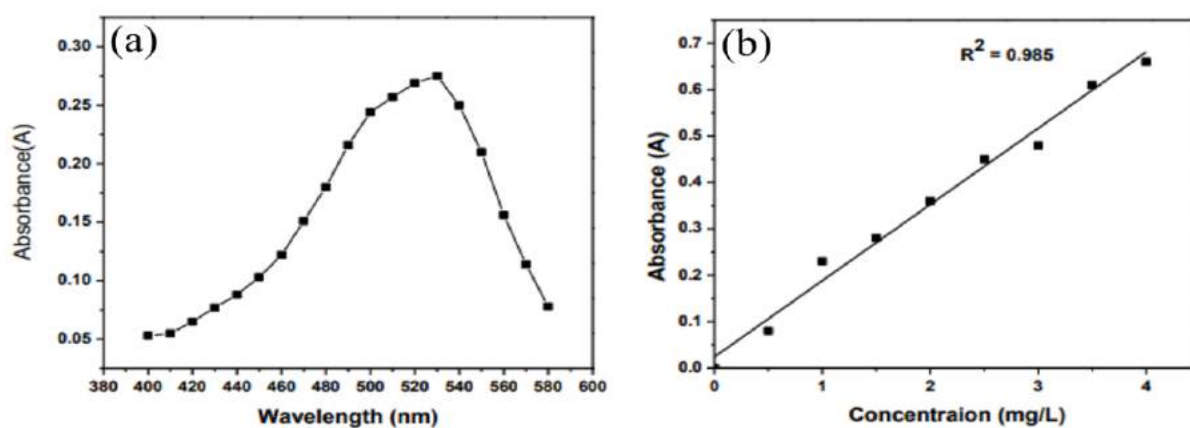

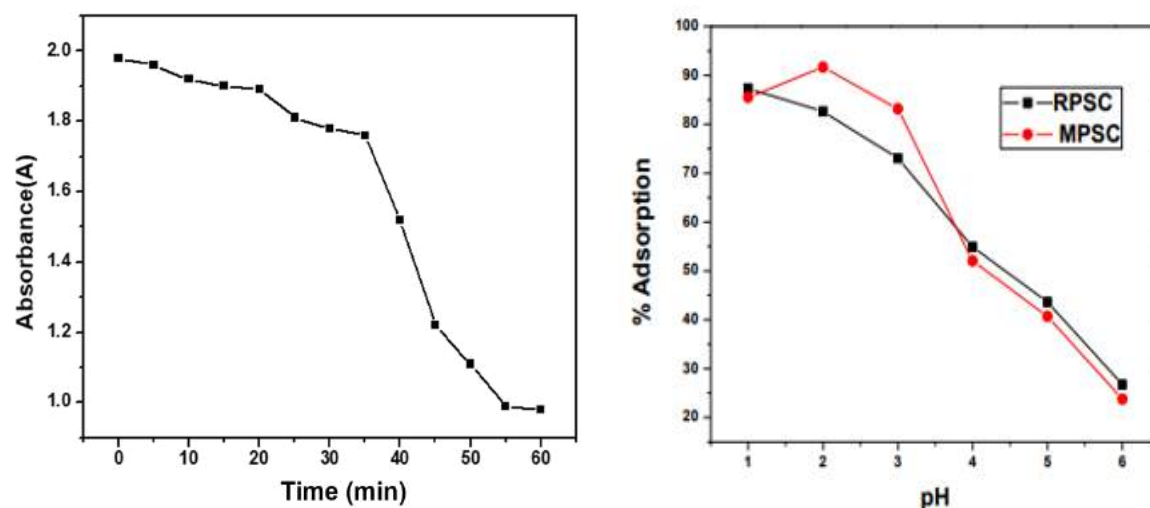Figure 1: (a) Determination of $_{max}$ for Cr (VI)-DPCI complex (b) calibration curve.

Figure 2: Effect of time on Cr (VI)-DPCI complex formation.

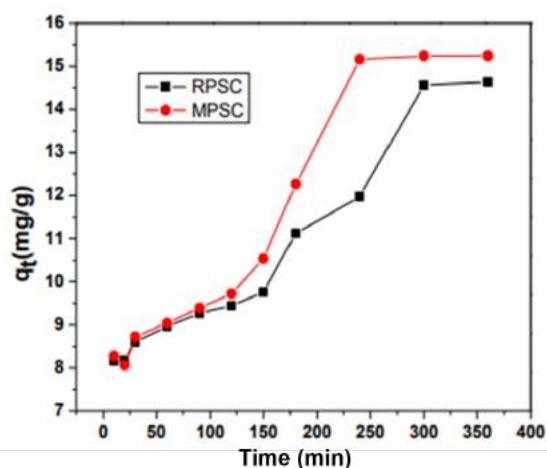Figure 3: Effect of pH on Cr (VI) adsorption by RPSC and MPSC.

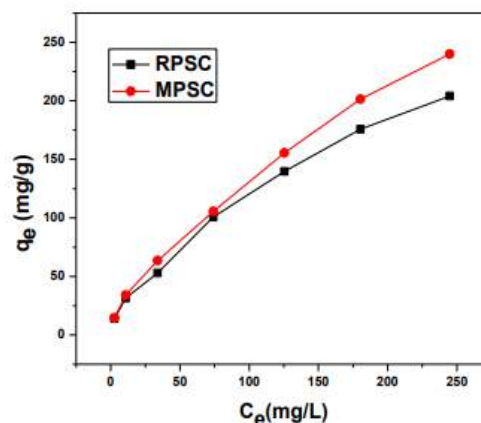Figure 4: Effect of contact time for the adsorption of Cr (VI) onto RPSC and MPSC.

Figure 5: Adsorption isotherm of Cr (VI) onto RPSC and MPSC.

3.5 Effect of initial Cr (VI) ion concentration

For MPSC, the quantity adsorbed increased from 11.61 mg/g to 201.90 mg/g, whereas for RPSC, the amount adsorbed increased from 13.82 mg/g to 204.12 mg/g. The adsorption increased from lower concentration to higher concentration ranges (20 mg/L to 500 mg/L). These results demonstrate that, as the amounts of adsorbent increases, so does adsorption. The effects of the initial adsorbate concentration for RPSC and MPSC are shown in **Figure 5**.

3.6 Batch isotherm studies

The Freundlich and Langmuir isotherms exhibited a linear relationship with Cr (VI) adsorption onto MPSC and RPSC. **Figures 6 (a)** and **(b)** illustrate respectively, the Langmuir isotherm and the Freundlich isotherm whose R^2 value indicates that Freundlich isotherm is best fitted over Langmuir isotherm. This result indicates that Freundlich isotherm has greater applicability. The insight into the sorption favorability in terms of correlation coefficient indicates the active sites of MPSC were dispersed throughout and the ions are adsorbed chemically on the surface.
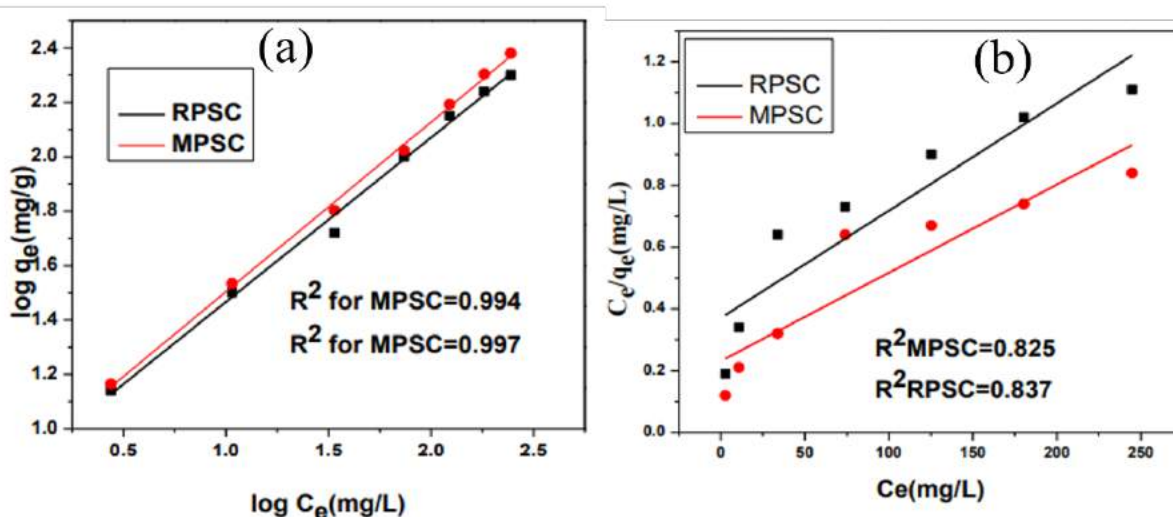

Figure 6: (a) Langmuir isotherm and (b) Freundlich isotherm plot for the adsorption of Cr (VI) onto RPSC and MPSC.

3.7 Batch kinetic modeling

The kinetic data for Cr (VI) adsorption onto MPSC and RPSC was studied using pseudo-first-order and pseudo-second-order kinetic models. The comparative plot of these models is represented in **Figures 7 (a) and (b)**, respectively. From the kinetic plots for the adsorption of Cr (VI), it was found that the value of the correlation coefficient (R^2) for the pseudo-second-order kinetic model was higher (close to one) than that of the pseudo-first-order. The results of the experimental data were better described by pseudo-second-order kinetics. This kinetic model signifies the adsorption behavior of adsorbent across the complete range.
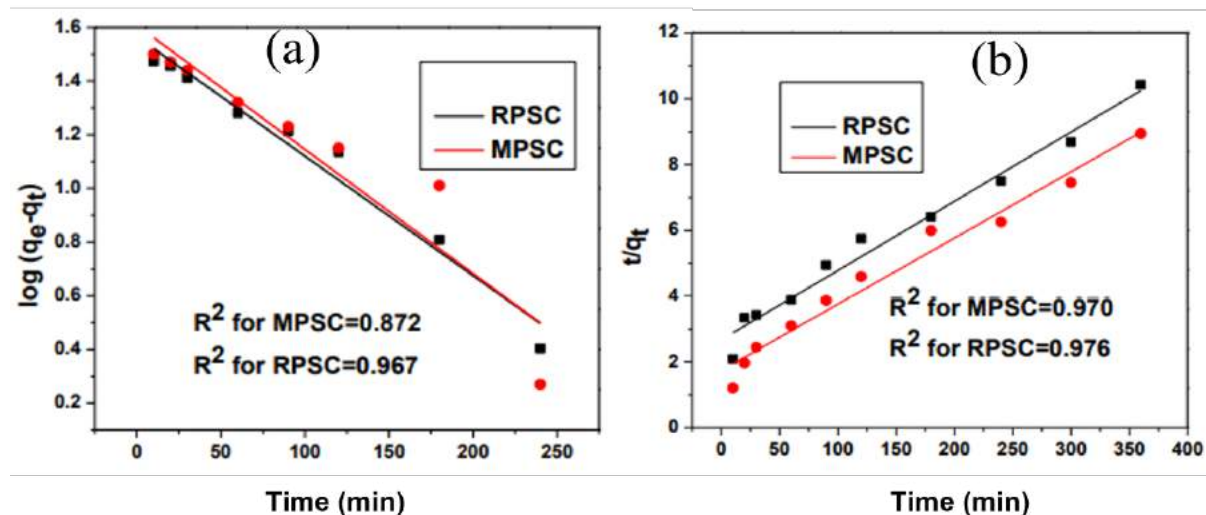

Figure 7: Pseudo (a) first and (b) second order kinetic model for the adsorption of Cr (VI) onto RPSC and MPSC.

3.8 Effect of adsorbent dose

The effect of the adsorbent dose for the adsorption of Cr (VI) onto MPSC and RPSC, was studied at room temperature by varying the adsorbent amount from 25 mg to 200 mg which indicates that the adsorbent dose is directly proportional with the adsorption yet showed constant value of the % adsorption beyond 100 mg for MPSC and 160 mg for RPSC, respectively. In addition to this, % adsorption was remarkably higher in case of MPSC over RPSC for the same doze of adsorbent. **Figure 8** shows the effects of adsorbent dose for the adsorption of % of Cr (VI).

3.9 FTIR analysis

FTIR analysis was performed to investigate the surface functional groups of MPSC and RPSC whose spectra have been shown in **Figure 9 (a) and 9 (b),** respectively. The graph of MPSC indicated the presence of -OH, C=O, N=N=N, N=C=S, C=N, N-O, etc., which are responsible for the adsorption of Cr (V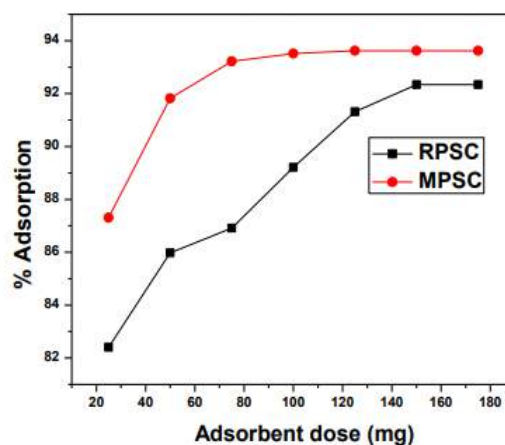I). Compounds containing these functional groups have enough ability to bind chromium as they contain double bond active sites.

Figure 8: Effect of adsorbent dose for the adsorption of Cr (VI) onto RPSC and MPSC.
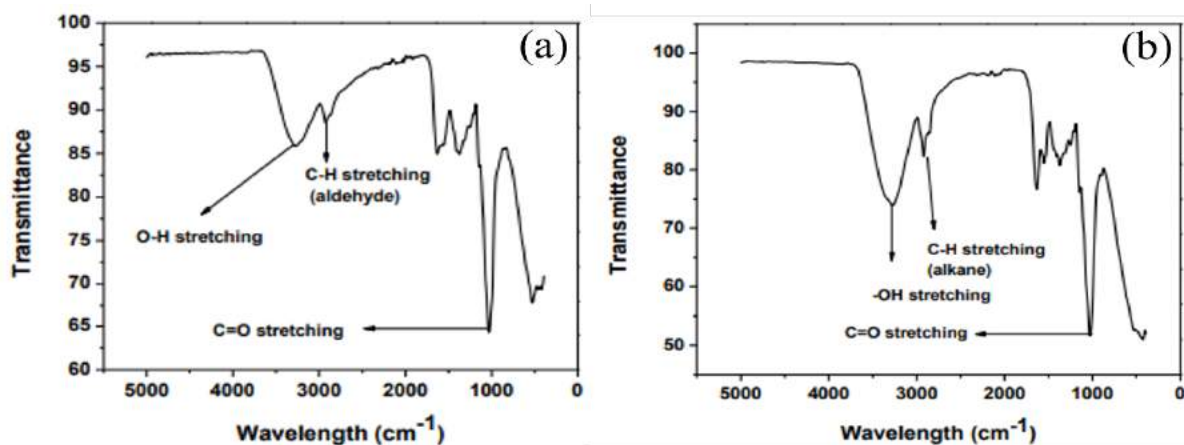
Figure 9: FTIR spectra of (a) RPSC and (b) MPSC.

3.10 FE-SEM and EDS analysis

Figure 10 displays the FESEM analysis of the adsorbent samples both before and after adsorption along with EDS mapping and EDS spectrum. Prior to adsorption, the FESEM images at different magnitudes for MPSC revealed a porous structure and the presence of more free sites **(Figure 10 A-C)**. The high magnification image of MPSC in **Figure 10C** indicates the distinctive porosity, and awful rough surfaces with broken edges thus providing valuable sites for metal ion adsorption on the surface of adsorbent. After adsorption, the surface morphology of sorbent MPSC **(Figure 10D)** showed some changes that signify the effective adsorption of metal ions on the surface of the adsorbent. The morphological change as a result of the

accumulation of Cr (VI) ions on MPSC led to the smooth and brighter surfaces might be due to interaction of functional group of sorbents with targeted metal ions. This revealed that a higher concentration of Cr (VI) had been adsorbed using the bio-sorbent also confirmed from the EDS elemental mapping obtained from the FESEM. The post-adsorption EDS pattern of MPSC **(Figure 10E-I)** revealed characteristic colour mapping images for carbon, nitrogen, oxygen, and chromium. Moreover, the EDS spectrum and the elemental distribution in **Figure 10J** clearly depicts the significant amount of chromium and other element revealing the potency of bio-adsorbent for efficient removal of toxic metal from aqueous solution. Overall, the rough surface of MPSC has a considerable effect on the capacity for adsorption [37].

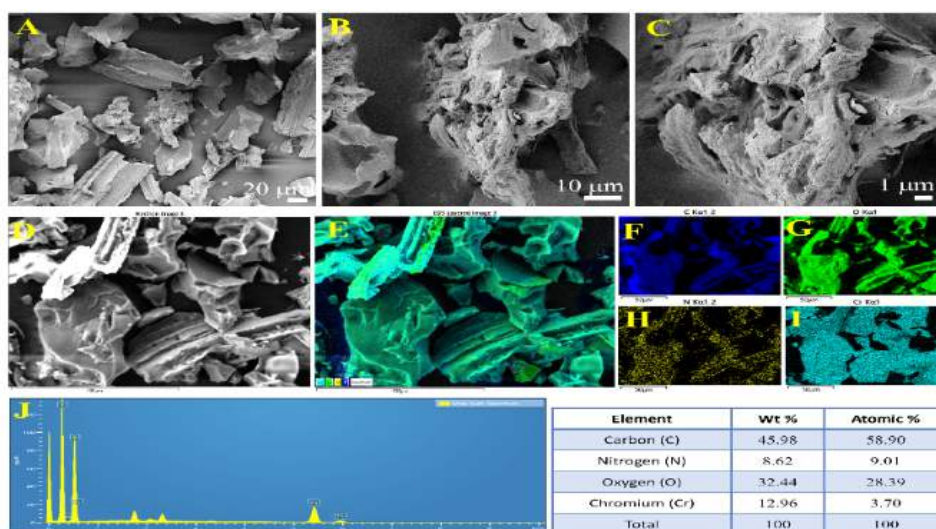| Element	Wt %	Atomic %
Carbon (C)	45.98	58.90
Nitrogen (N)	8.62	9.01
Oxygen (O)	32.44	28.39
Chromium (Cr)	12.96	3.70
Total	100	100

Figure 10: FE-SEM image of (a, b, c) MPSE, (d) MPSC after adsorption and (e, f, g, h, i) FESEM-EDS colour mapping of MPSC, (J) EDS spectra of MPSC and corresponding atomic composition..

4 Conclusion

This study provides promising evidence for the use of *Pleurotus sajor-coju* as the potent source of a new adsorbent due to high abundance and low costing procedure. Modified *Pleurotus sajor-coju* was found to be more effective adsorbent than raw one, for the removal of Cr (VI). FTIR analysis confirms the presence of azide, thiocyanate, alcohol, nitro, sulphonyl chloride and oxime groups on NaOH modified *Pleurotus sajor-coju* (MPSC) which are responsible for adsorbing Cr (VI) from aqueous solution. The mass of the adsorbents were reported to be amorphous, however certain peaks displayed the crystallinity of cellulose. Additionally, the SEM analysis of the surface morphology revealed a rough, uneven surface with a distinct dark area of pores and voids. These circumstances favor the adsorption of metals. Various results illustrate that the adsorption of Cr (VI) increase with the increase in contact time, concentration of the solution and adsorbent doze. The mechanism of adsorption is better described by pseudo-second order kinetics with the Freundlich isotherm giving the best fit over the Langmuir isotherm.

Acknowledgment

Conflict of interest

The authors have no financial and other conflict of interest.

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