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Optimization of Chemical Impregnation Methods for the Regeneration of Spent Activated Carbon

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KEYWORDS
Activated char, Recycle, Adsorption, Water purification

ABSTRACT:
The regeneration of spent activated carbon through chemical impregnation is an effective method to restore its adsorptive capacity. This process involves treating the spent activated carbon with specific chemicals to remove adsorbed contaminants and regenerate the carbon. this study aimed to investigate the characteristics of the regenerated activated carbon for potential utilization in water purification applications. Spent activated carbon was collected from the used cartridge of the activated carbon of the water purification unit. It has a higher carbon content and is readily available. The spent carbon was activated using 1N KOH at the optimum activation duration. The present study involves the elemental analysis of spent activated carbon prior and post-activation. Characterization as CHNS (elemental analysis), FTIR, SEM, and important index iodine number studies during this work. The present project aims to look into the potential regeneration of spent activated carbon. Well-developed pores and activation sites in regenerated carbon have been observed.

1. Introduction

Activated carbon is used in current scenarios in various sectors like pharmaceuticals, wastewater treatment, food and beverage, cosmetic industry, etc. Activated carbon can be regenerated. To eliminate the impurities that have been adsorbed and partially restore its adsorption capacity [1]. Reactivation, incineration, and landfilling Currently, there are three ways to get rid of spent active carbon. around 95% of powdered active carbon gets disposed of in landfills since it is the least expensive option, and roughly 68% of granular active carbon is reactivated. Because of this, there is a resource and waste management issue with the continuous use and disposal of powdered AC that needs to be properly handled. Reactivating spent AC is, therefore a sustainable strategy; nevertheless, reactivating carbon cannot be done at a reasonable cost.[2]. The regeneration and reuse of SAC is also a practical way to reduce secondary pollution and conserve natural resources [3]. There have been numerous reports of regeneration techniques, such as thermal desorption with liquid water under subcritical conditions, solvent regeneration, biological regeneration, microwave regeneration, and electrochemical regeneration [4]On the other hand, chemical activation is a highly effective way to improve the pore structure of activated carbon. By processing raw materials with appropriate chemical activators at optimal temperatures, activated carbon is produced with outstanding qualities. The employment of activator agents such as phosphoric acid (H_3PO_4), zinc chloride ($ZnCl_2$), potassium carbonate (K_2CO_3), potassium hydroxide (KOH), and sulfuric acid (H_2SO_4) produces activated carbon of exceptional quality. Maximizing the chemical activation process is critical to ensuring activated carbon's economic viability as an absorbent [5], [6].

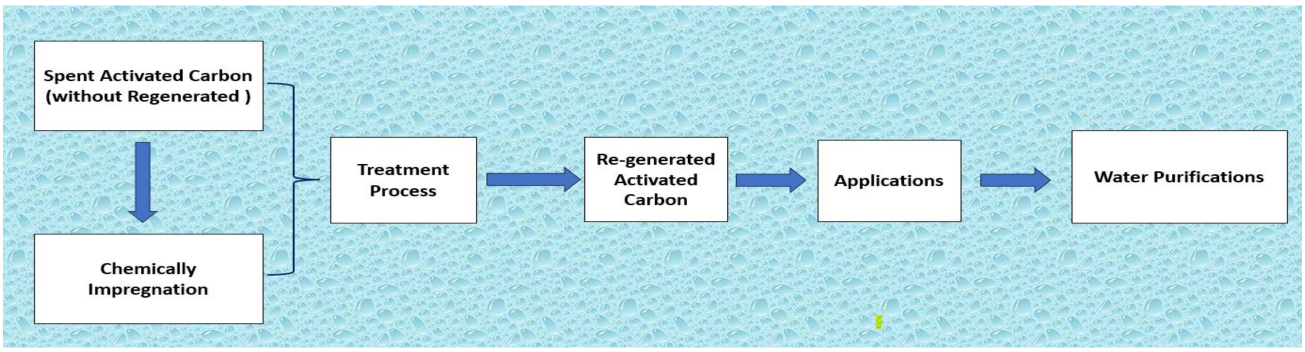

Fig. 1. Process flow of activated carbon.

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Activated carbon is a microcrystalline material with a high surface area and porosity, making it highly effective as an adsorbent [7]. The type of raw materials utilized will surely result in variations. The surface area of the carbon that is activated can also differ based on the raw ingredients, temperature, and type of activation applied. [8]. Low-temperature carbonization is ideal for producing efficient activated carbon as it promotes the development of a porous structure [9]. Hydrochloric acid is one of the best activating agents as it modifies the graphite structure, resulting in high adsorption capacity [10]. The content of activated carbon is dependent on the source material and processing method utilized [11].

The utilization of carbon-based materials in the purification of wastewater applications can make a sustainable approach to remediation in wastewater purification. In the present work, SAC is used to regenerate & activated through chemical treatment methods. Characterization is performed to reveal the carbon content, char yield, and activated carbon properties.

2. Experimental

2.1 Materials

The following material is used as follows

2.2 Chemicals Used

In the present work, the following chemicals were used. The sources of the chemicals and their acronyms used are given in Table I.

Table I List of chemicals used and their sources

Sr. No.	Chemicals	Acronym	Source
1	Spent Activated Carbon	SAC	Cartridge from RO system
2	Potassium Hydroxide	KOH	Central Drug House, New Delhi
3	Hydrochloric acid	HCl	Loba Chemie Pvt Ltd
4	Potassium iodide	KI	Loba Chemie Pvt Ltd
5	Iodine	I	Central Drug House, New Delhi
6	Potassium iodate	KIO_3	Thermo Fisher Scientific India Pvt Ltd
7	Starch solution	$(C_6H_{10}O_5)n$	SD Fine Chemicals (SDFCL)
8	Sodium thiosulphate	$Na_2S_2O_3$	Loba Chemie Pvt Ltd

2.3 Preparation for Regenerated Spent Activated Carbon (SAC)

The raw material used for the preparation of activated carbon is spent activated carbon without re-generated.

2.4 Raw Material Collection

Spent activated carbon as a raw material collected from the activated carbon cartridge of the RO system. SAC was stored in bags. (Fig. 2).

Fig. 2. SAC sample.

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2.5 Drying of SAC

The samples of SAC were put into trays. These trays were put first to dry in the oven for 15 minutes at 80 degrees centigrade.

2.6 Sieving

The SAC samples were sieved in 200 and 100-grade mesh to separate the larger particles and achieve a homogeneous particle size (Fig. 3). Sieving is important for the development of fine particle size of activated carbon, which is required for proper distribution in the matrix.

Fig. 3. Prepared SAC sample after sieving.

2.7 Impregnation

The char was collected in a beaker and treated with KOH Solution using various impregnation ratios 5:1.

2.8 Magnetic Stirrer Process

The beaker was placed on a magnetic stirrer for 2 hours of activation.

2.9 Washing and Drying

The obtained regenerated spent activated carbon was rinsed with 1N dilute HCL for one hour and then thoroughly washed with distilled water multiple times until the sample reached a pH of 6-7. The obtained regenerated SAC has been dried in an oven at 110 ºC for 4 hours before being kept in desiccators.

2.2.1 Methods

The activated carbon samples from plastic waste wrappers were studied for percentage of the yield for spent activated carbon, CHNS elemental analysis, Iodine number, SEM (Scanning Electron microscope), FTIR (Fourier Transform Infrared Spectroscopy), and the presence of corresponding functional groups.

2.2.2 Percentage of the Yield

For the spent activated carbon, the percentage of yield is an important parameter. The amount that was produced from the dried precursor can be calculated by using the formula. Eq. (1) [12].

$$\text{Yield \%} = 100\text{x} \frac{\text{Weight of activated carbon produced (g)}}{\text{Weight of dried precursor used (g)}} \quad (1)$$

2.2.3 CHNS Elemental Analyzer

The CHNS elemental analyzer is used to find and determine the percentages of carbon (C), hydrogen (H), nitrogen (N), and sulphur (S) in a sample.
Elemental analysis on developed samples was recorded by CHNSO Elemental Analyser, Elementar (Unicube), from CIPET Lucknow. Characteristics of char and developed activated carbon have been studied to analyze the Carbon (C), Hydrogen (H), Nitrogen (N), and Sulphur (S) % percentages in all our samples.

2.2.4 Iodine Number

The ASTM D 4607-94 test method establishes a defined process for determining the iodine number of activated carbon. The iodine number is the quantity of iodine in milligrams absorbed by one gram of activated carbon under certain test conditions. It is represented in the SI unit mg/g. The method employs an iodometric titration in which alkaline iodine (0.1N) is titrated against sodium thiosulfate (0.1N), with a 1% starch solution serving as an indicator. The procedure is taking a blank reading followed by a reading with activated carbon [13], [14].

2.2.5 Scanning Electron Microscope (SEM)

A Scanning Electron Microscope (SEM) is used to find the surface properties and morphological structure. The morphological images on developed samples were recorded by a Scanning Electron Microscope (SEM) Jeol (KM 6000 plus), from CIPET Lucknow. Characteristics of char and developed activated carbon have been studied to

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analyze the morphological changes in all our samples.

2.2.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is used to find the chemical bonds present in prepared samples. The functional group ability on developed samples was recorded by Fourier Transform Infra-Red Spectrometer Thermo-Scientific (Nicolet 6700), from USIC BBAU Lucknow. Characteristics of cha and developed activated carbon have been studied to analyze the variation of functional groups in all our samples [15].

3.0 Results and Discussion

3.1 Percentage of Yield of Activated Carbon

Spent activation carbon was undergone for impregnation chemically, and the activated carbon has been developed. As per the impregnation with KOH Solution at 5:1, impregnation ratios have been performed to develop the activated carbon. the result of % yield for the samples has been shown in Table II. For the KOH/SAC ratio of 5:1, the activated carbon yield calculation is 18.24 %.

Table II. Yield of activated carbon

Sr.no.	Ratio of KOH/Char	Char wt.(gm.)	After activation with the KOH process, AC wt.(gm.)	After drying (gm.)	Yield % of AC
1	5:1	1.0	0.98	0.96	96 %

3.2 CHNS Analysis

The CHNS elemental analyzer is utilized to confirm the presence of carbon, hydrogen, nitrogen, and sulphur in organic matter and other materials.
The results of this analysis are displayed in Table III. The carbon content of the source material SAC without regenerated was found to be 73.45 %. After the chemically regenerated SAC sample, the carbon content was observed to be nearly 80%. The high carbon content results show that it is suitable for use in activated carbon preparation [16]

Table III. CHNS Analysis

		CHNS Elemental Analysis			
Sr. No.	Ratios	C %	H %	N %	S %
1	SAC	73.45	3.12	0.59	0.56
2	5:1 SAC	79.80	2.20	0.40	0.73

3.3 Iodine Number of Activated Carbon

The iodine number value for the spent activated carbon is observed at 318.72 mg/g. The value of iodine number for our SAC samples with chemically impregnation has been shown in Table IV.

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Table IV. Iodine number of activated carbon

Plastic waste wrappers	Ratio	Weight of AC (gm.)	Iodine Number (mg/g)
Without regenerated SAC	5:1	0.5	346.72
Spent Activated Carbon	5:1	0.5	1140.48

The activated carbon's iodine number, which indicates its adsorption ability, typically ranges from 500 to 1200 mg/g, making it an excellent adsorbent [17].

3.4 Scanning Electron Microscope (SEM)

SEM of SAC without regeneration is shown in (Fig. 4a), and images after Chemically activation by KOH impregnation ratios 5:1 are shown in (Fig. 4b), The progressive increase in pore creation during carbon conversion to activated carbon reveals a large outer surface with uneven cracks and pores that occur throughout the activation stage. SEM images show a network of interconnected voids and pores throughout the samples. The distribution of voids indicated the uniformity of the morphology in samples and is desirable for consistent performance in applications. Bright areas representing carbon-rich zones are clearly visible in the SEM images of samples.

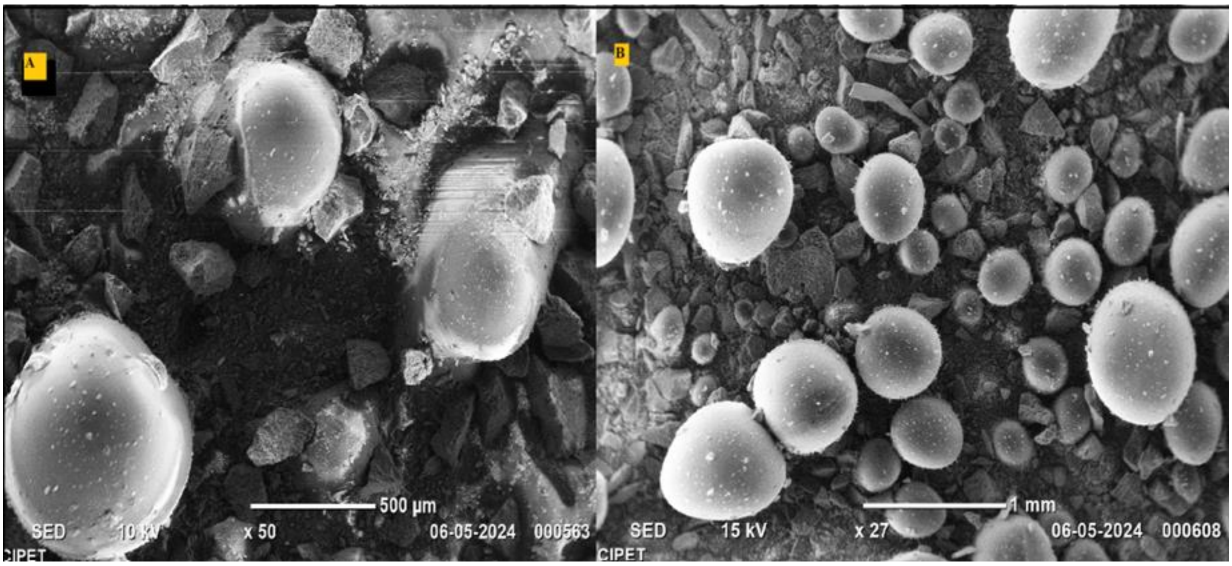

Fig. 4. SEM analysis **(a)** 5:1 without regeneration **(b)** 5:1 SAC impregnated with KOH.

3.5 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was carried out for the SAC sample and regenerated with 5:1 KOH solution by chemical impregnation to determine the change in functional groups present in both samples in Fig. 5. The peaks at 1093 and 1144 for C-O stretching vibration have been found significantly in the regenerated SAC, while peak 1203 was present in untreated SAC indicating same functional group. So, the presence of aliphatic ether has been confirmed. The peaks 1705 with strong intensity confirm the condensed aromatic groups only in the treated SAC sample regenerated. The peaks at 2006 cm^{-1} indicated N=C=S stretching of isothiocyanate, the peak at 2387 having stretching vibrations of C–H in aliphatic compounds, and the peak at 2617 confirming O-H stretching carboxylic acid post-treatment SAC sample [18], [19] . These peaks are not found in untreated SAC, it indicates the alkaline reaction with various deposits present in saturated spent activated carbon during the regeneration process. The O-H carboxylic acid stretching was found at peaks 2802 in untreated SAC and its absence in regenerated SAC confirms the removal of the carboxylic group due to the treatment. N=H stretch of amine salt was located at 2914 in the spectra of the spent activated carbon. O-H bond is significantly present the both samples, confirming the presence of the hydroxyl group. The rest inherited groups relevant to the carbon family are common in both samples and have no effect of alkaline chemical impregnation [20].

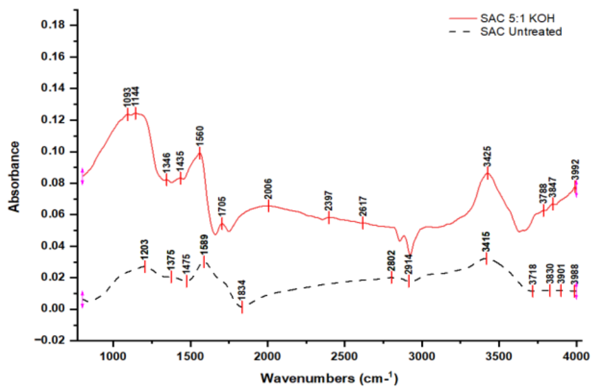

Fig. 5. FTIR spectra of SAC untreated & SAC 5:1 KOH regenerated.

4.0 CONCLUSION

In the present work, Spent activated carbon was regenerated through a chemical treatment process methodology, and the yield of activated carbon was observed at 18.24%. CHNS value validates the carbon % present in activated carbon, and more than 79 % of carbon was reported in samples. The obtained iodine value of the 5:1 ratio is 1140.48 mg/g, which indicates the adsorption potential and validates the formation of activated carbon with this impregnation ratio. SEM analysis explores the morphological structure of activated carbon and reveals the network of interconnected voids and pores throughout the samples. FTIR analysis confirms the presence of inherited functional groups similar to backbone carbon. Some chemical reactions with the deposited scale of spent activated carbon have also been seen during chemical impregnation. The results unambiguously reveal that activated carbon is highly effective in absorbing contaminants in water, particularly those originating from industrial effluents.

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Data availability

The data that support the findings of this study are available on request from the corresponding author.

Author contributions

Ravi Verma Formal analysis, Investigation, Original draft, Writing - review & editing.
Dr. Manish Choudhary Supervision, Validation, Writing - review & editing.

Conflict of interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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