Nanoporous activated carbon from Bel fruit (*Aegle marmelos*) shell as efficient bio-adsorbent for river water treatment

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Abstract: Bel fruit *(Aegle marmelos)* shells were activated using phosphoric acid to synthesize nanoporous-activated carbons (ACs). The different ratios of bel shell powder to phosphoric acid were used to optimize the activating agent and then carbonized at 400 ℃ for three hours in a nitrogen atmosphere. The thermal stability of bel shell powder was determined using thermogravimetric analysis. Surface functional groups existing on the surface of bel shell-activated carbon were characterized using Boehm titration and Fourier Transform Infrared Spectroscopy (FTIR). The pore size was determined using iodine and methylene blue adsorption and surface morphology was analyzed using Scanning Electron Microscopy (SEM). The iodine adsorption was maximum (850.364 mg/g) at an impregnation ratio of 1:1.0 (precursor: phosphoric acid, BSC_1.0). The oxygenated functional groups such as hydroxyl, carbonyl and carboxyl present on the surface of the activated carbon (BSC_1.0) were confirmed by Boehm titration and FTIR analysis. Both Langmuir and Freundlich models fit well in methylene blue adsorption. The coefficient of the determinant was comparatively higher for the Langmuir model than the Freundlich model with an adsorption capacity of 227.27 mg/g. The remediation efficiency of bel shell-activated carbon was determined by treating Bagmati river water with BSC_1.0. The river water was black and found to be extremely polluted with -214 mV ORP, after treatment water turned into clean and clear and ORP enhanced to 91.0 mV. It removed more than 80% of contaminants from the river water, and significantly improved water quality to the WHO limits. Therefore, it is concluded that activated carbon from bel shells can be effectively used as bio-adsorbents for the remediation of Bagmati River water.

Keywords: Activated carbon; Bel shell; Remediation; Water pollution.

Introduction

Several techniques such as bio-degradation, advanced oxidation process, coagulation, membrane separation, adsorption, ion exchange, precipitation, activated sludge, electrochemical conversion, photo-catalytic degradation, catalytic ozonation etc. were employed for treating contaminated water[1]. However, most of these techniques are expensive, inefficient and/or generate large amounts of waste products that requirefurther disposal[1,2]. Recently adsorption techniques have gained popularity in water treatment[3]. It is a critical and popular tool used throughout municipal and industrial water treatment facilities[3,4]. Activated carbon (AC), a solid, micro-crystalline, non-graphitic form of black carbonaceous material with a porous structure, has been

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considered a unique and versatile adsorbent because of its high surface area, nanoporous structure, excellent adsorption capacity, and high degree of surface reactivity[5]. Recently, AC has been profoundly used as a powerful adsorbent for water purification by removing major environmental contaminants and undesirable components. Adsorption of pollutants from water is possible by developing a porous structure on the surfaceactivated carbon. Several factors, including activation technique, temperature, activating agent, impregnation ratio, duration of carbonisation etc., affect the properties of activated carbon, particularly its pore size[6,7]. Conferring to the definition from the International Union of Pure and Applied Chemistry (IUPAC), porous materials are categorized into three classes based on different pore sizes: micro-porous (<2 nm), mesoporous (2–50 nm) and macro-porous (>50 nm) materials. Such hierarchically porous materials reveal a porous hierarchy in which the porosity and structure traverse multiple length scales from micro- to meso- and macropores[8]. Generally, activated carbon is produced by physical and chemical activation methods. Physical activation consists of a two-step process. Char obtained from the carbonization of raw materials is gasified with either steam or oxidizing gases (N_2, O_2 CO_2, etc) at higher than 800 °C temperature[9]. It mainly involves diffusion, resulting in limited pore formation at lower temperatures but high temperatures lead to well-defined pores. The relatively long activation time and lower carbon yield are the main drawbacks of physical activation[10]. In chemical activation, the precursor material is treated with suitable chemical activating agents before carbonization[11]. This method also known as wet oxidation, is usually applied to raw materials consisting of cellulose, also called biomass resources[10]. The simultaneous carbonisation and activation process aids in decomposing lignocellulosic materials and eliminating impurities, resulting in well-developed pores[12]. The activating agents are mostly dehydrating agents that affect the pyrolytic decomposition and resist the production of tar and volatile matter with subsequent changes in the thermal degradation of precursors, hence contributing to the porous structure of carbon materials and yield percentage[13]. The commonly used activating agents are alkaline groups such as potassium hydroxide (KOH), sodium hydroxide (NaOH), calcium chloride ($CaCl_2$) and potassium carbonate (K_2CO_3), acidic groups such as phosphoric acid (H_3PO_4) and sulphuric acid (H_2SO_4), intermediate metal salts such as $ZnCl_2$[10]. Benefits of chemical activation include increased product yields and decreased energy expenses. The selection of precursor material notably impacts the properties of the resulting activated carbon[14]. The synthesis of nanoporous carbon from biomass/non-biomass and/or other wastes has gained the utmost attention for environmental and energy applications[9]. The production of activated carbon from readily available waste materials has been the subject of research interest for the low-cost adsorbent[2-4, 12-14]. *Aegle marmelos* (bel) is a common fruit found throughout South Asia[15]. The inner portion of the bel fruit consists of edible sweet and thick aromatic pulps having several medicinal uses. The pulps are highly nutritive having phenols, tannins, glycosides, alkaloids etc.[15] It has a hard and woody shell and is considered an agro-waste product. The studies reported that biochar from the bel shell contains micro-pores useful for the chemisorption of dye from an aqueous solution[16].

Safe water is enormously important for human beings to survive. Water should have a pleasant taste and colour, be devoid of harmful substances and pathogens, and be safe for household use without causing any harm[17]. When water exhibits high levels of cloudiness, unpleasant taste, and smell, along with the presence of microorganisms and undesirable chemicals in amounts that threaten health, it is deemed polluted[18,19]. The water quality of the Bagmati River in Nepal, particularly as it flows through the Kathmandu Valley, has significantly deteriorated over recent decades. This trend of increasing pollution is due to several factors, including inadequate wastewater treatment, urbanization, and direct discharge of untreated sewage and industrial effluents into the river[20]. According to studies, the

river's water quality mostly exceeds safe limits for water quality parameters[21]. Addressing these challenges is crucial to protecting the health of both the Bagmati River ecosystem and the population relying on it in Nepal. In this study bel shells activated carbons were prepared using the chemical activation process and characterized using different techniques. The bel shell activated carbon (BSC_1.0) was utilized to remediate the Bagmati River water.

Materials and Methods

Preparation of activated carbon

The bel fruits were collected from Tanahu, Nepal and shells were separated and washed with distilled water to remove impurities, then dried at 110ºC in an air oven for 24 hours. The dried shells were crushed into powder using the electric grinder sieved to get uniform-sized particles. The powder was mixed with the required amount of phosphoric acid (H_3PO_4) in the ratio of 1:1, 1:1.5, and 1:2 by weight and represented by BSC_1.0, BSC_1.5, and BSC_2.0, respectively. The mixture was kept for 24 hours and then carbonized at 400ºC in a tube furnace for 3 hours. After cooling the carbonized carbon was washed with the 1% $NaHCO_3$ then with the distilled water until the pH to neutralize the acid[22]. The activated carbons were dried in an oven at 110ºC for 24 hours and sieved with a mesh of 185µm.

Characterization of activated carbon

The amount of iodine adsorbed by the activated carbons was determined by the ASTM D4607-94 method. 0.1 g of activated carbon was mixed in 5mL of 5% HCl, boiled and subsequently cooled to room temperature. The resulting mixture was shaken vigorously by adding 10mL of 0.05 M iodine solution and titrated with 0.05M sodium thiosulphate solution after filtration[18]. The iodine number was calculated from the equation (1);

$$Iodine\ number\ (I_n) = \frac{wt.\ of\ iodine\ adsorbed\ on\ carbon\ (mg)}{wt.\ of\ activated\ carbon\ (g)} \quad (1)$$

The methylene blue number was determined using multipoint adsorption isotherm. 25 mg of activated carbon was added to 25 mL methylene blue solution at different concentrations (25, 50, 100, 150 and 200 mg/L). An equilibrium concentration of methylene blue was spectrophotometrically determined after continuous shaking for three hours. Equation (2) was used to determine the methylene blue number.

$$MB_N = \frac{(C_o - C_e)V}{M} \quad (2)$$

Where, the initial and equilibrium concentrations of methylene blue (mg/L) were represented by C_o and C_e, respectively. The volume of methylene blue and the mass of activated carbon were represented by V in litre and M in gram, respectively.

A point of zero charge (pH_{zpc}) was determined by adjusting the initial pH at 2, 3, 4, 5, 6, 7, 8, 9 and 10 of 0.1M $NaNO_3$ solution and then 0.1 g of activated carbon was added to 50 mL solution. The mixture was agitated for three hours and the final pH was accurately measured. The pH_{zpc} value was evaluated from the ΔpH versus the initial pH curve[18-23]. The Boehm titration is utilized to determine the acidic and basic functional groups on the surface of activated carbon. 0.1 g of activated carbon was mixed with 25 ml of 0.05 M Na_2CO_3, $NaHCO_3$, and NaOH separately, and titrated with 0.05 M HCl after shaking for 3 hours. The amount consumed by $NaHCO_3$ represents the presence of phenolic groups the difference between the amounts consumed by NaOH and Na_2CO_3 is the carboxylic groups and the difference between the amount consumed by Na_2CO_3 and $NaHCO_3$ is lactonic groups present on the surface of activated carbon[24].

Instrumental analysis

The surface functional groups of activated carbon were qualitatively determined by Fourier-transform infrared (FTIR) spectroscopy (NICOLET iS20, Thermo-Fisher Scientific, Waltham, MA, USA). A spectroscopic technique called Raman scattering spectroscopy (NRS-3100, JASCO, Tokyo, Japan) was used to determine graphitic and defective carbons. A scanning electron microscope (SEM) (S-4800, Hitachi Co., Ltd. Tokyo, Japan) operated at 10 kV and 10 µA was used to evaluate the surface morphology of an adsorbent.

Remediation of the Bagmati River water

The remediation efficiency of bel shell-derived activated carbon was determined by treating heavily polluted Bagmati River water with BSC_1.0. The physico-chemical parameters such as pH, hardness, alkalinity, conductivity, dissolved oxygen, chloride, sulphate, phosphate, iron, chromium ion concentrations etc. were analyzed following standard procedure before and after treatments with BSC_1.0[25,26].

Results and Discussion

The proximate analysis determined the moisture, volatile, ash and fixed carbon content in bel shell powder. The moisture, volatile matter, fixed-carbon and ash content were 10.54, 68.929, 19.894 and 0.637 %, respectively. The less than 1% ash and nearly 20% fixed carbon in the precursor suggested that the powder of bel shell is suitable as a precursor for an efficient activated carbon. The thermogravimetric curves of precursor (BSP) and phosphoric acid-treated precursor (BSC) show that the weight of the precursor remained almost constant until 200 ºC and then decreased (Fig.1). The weight was excessively reduced between 250℃ and 300 ºC and continued till 400 ºC and was not significantly reduced with a further increase in temperature. The acid-treated bel shell powder shows a slightly different curve. The weight loss started at 100 ºC till 350 ºC and remained almost constant with a further increase in temperature. After complete pyrolysis, the remaining weight of precursors was about 20% whereas 40% remained in the acid-treated bel shell powder. Below 200 ºC loss in weight is due to the evaporation of moisture or water of crystallization in the precursors. More than 40 % of BSC and more than 70 % of BSP weight loss occurred in between 200℃ to 400 ºC due to the pyrolytic decomposition of hemicellulose, cellulose, and lower molecular weight lignin components[27,28]. A slight decrease in weight above 400 ºC is due to the evaporation of tar and less volatile materials trapped in the carbon networks. The insignificant weight loss above 400 ºC indicates the completion of pyrolysis hence the chemical activation was performed at 400 °C[28]. powder (BSP) and acid-treated bel shell powder (BSC).

The amount of activating agent required to develop maximum pore on the surface of bel shell powder was determined by varying ratios of precursor and

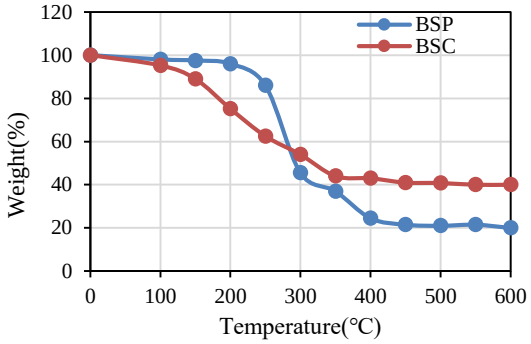


Figure 1: Thermogravimetric analysis of bel shell

phosphoric acid. The effect of the activating agent was determined by analyzing the amounts of iodine adsorbed (iodine numbers) by activated carbons prepared by using different ratios of precursor and phosphoric acid such as 1:1 (BSC_1.0), 1:1.5 (BSC_1.5) and 1:2.0 (BSC_2.0) by weight (Fig 2). The iodine numbers of activated carbons ranged from 736 to 850 mg/g. The iodine number was a maximum for BSC_1.0 (850.36 mg/g). From the maximum value, it is considered that the phosphoric acid completely and more extensively reacts with the surface of carbon at an impregnation ratio of precursor to phosphoric acid of 1:1.0[29]. The methylene blue adsorption capacity of BSC_1.0 was 227.27 mg/g and the specific surface area was 556.36 m^2/g.

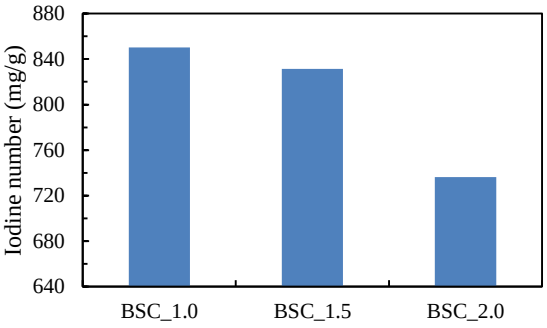


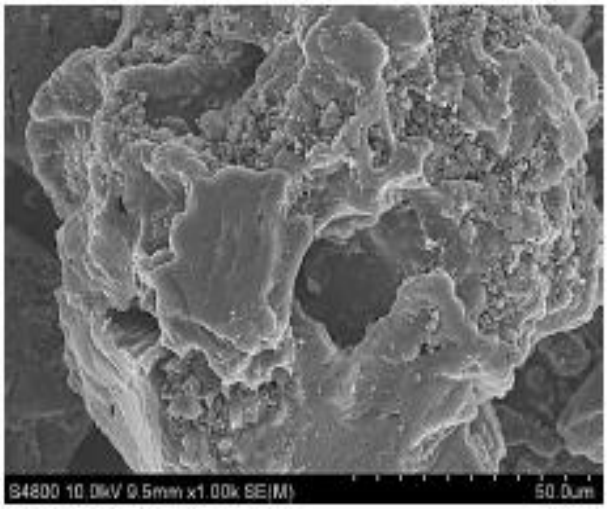
Figure 2: Iodine number of bel shell-derived activated carbons at different impregnation ratios.

The surface morphology i.e., the presence of pores with irregular surface patterns is one of the parameters which plays a significant role in the adsorption capacities of

the adsorbents[27]. The surface morphology of BSC_1.0 was studied using scanning electron microscopy (SEM). SEM scans the high-resolution imaging of macro and mesopores on the surface. The SEM images (Fig 3) of BSC_1.0 taken at different resolutions show the amorphous nature of activated carbons with irregular shapes and sizes. Fig 3 shows that the activation followed by carbonisation induced different-sized and shaped pores on the surface of bel shell powder. The nanopores observed in the high-resolution images suggested that as suggested by methylene blue and iodine numbers, phosphoric acid successfully developed pores on the surface of the activated carbon. The presence of nanopores of different sizes (macro, meso, and micropores) with charged surfaces will efficiently adsorb contaminants from the heavily polluted river water.

In addition to pore structures, the surface functional groups and surface charge also play a significant role in adsorption. The surface characterisation i.e., the functional group present on the surface of a precursor is essential to understanding the adsorption efficiency.

The presence of functional groups such as carboxylic, lactonic and phenolic on the surface of activated carbon calculated from Bohem titration was tabulated in Table 1[24]. Table 1 shows that the 1.323 m mole carboxylic, 0.676 m mole lactonic and 0.125 m mole phenolic groups were found on the surface of 1 g of BSC_1.0. The total surface acidic group was nearly 4 times higher



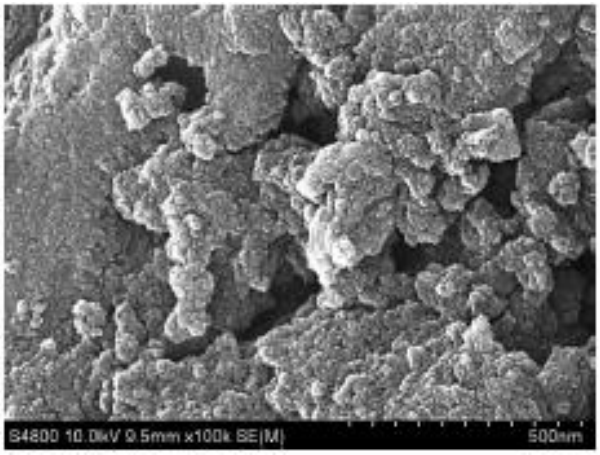


Figure 3: Scanning Electron microscopy (SEM) images of BSC_1.0 at different resolutions

than the total basic group. The observed pH_{pzc} value of BSC_1.0 was 6.45. Hence, it is considered that when the solution is lower than 6.45 the surface of activated carbon is positively charged and negatively charged when the pH is higher than 6.45[23-30]. At low pH, the surface of activated carbon will protonate and electrostatically adsorb negatively charged anions, at high pH, the surface becomes negatively charged and adsorbs cations.

Table 1: Functional groups and point of zero charge present on the surface of BSC_1.0.

Functional group	(mmole/g)
Carboxylic	1.323
Lactonic	0.676
Phenolic	0.125
Total acidic	2.124
Total basic	0.568
pH_{pzc}	6.45

The functional groups of BSC_1.0 were qualitatively analyzed using Fourier Transform Infrared (FTIR) Spectroscopy (Fig. 4a). The spectrum shows a broad peak at 3532 cm^{-1} indicates the O-H stretching vibrations of the alcoholic or phenolic groups and the peaks at 2966 and 2795 cm^{-1} indicate the C-H stretching vibration the alkyl group[12-27]. The distinct peak observed at 1761 cm^{-1} represents the stretching vibration of C=O

from the acetyl group of lignin, cellulose, and hemicellulose. The band at 1532 cm^{-1} represent C=C stretching of lignin. The bands at 1117 and 950 cm correspond to C-O stretching vibration of the carboxylate groups including >C=O, COOH, OH and

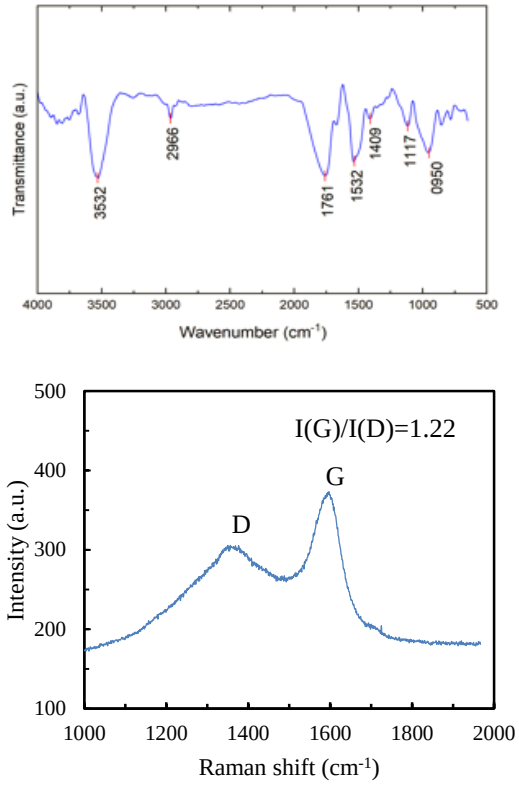


Figure 4: (a) FTIR spectrum (b) Raman spectrum of bel shell-derived activated carbon (BSC_1.0).

lactones. Both the Boehm titration and FTIR spectrum suggested that the surface of BSC_1.0 consists of carboxylic and hydroxyl groups which will interact and remove contaminants from water. The amorphous carbon usually shows a broad band at about 1550 cm^{-1} overlapping with a wider band at 1400 cm^{-1}. These spectra of Raman scattering can be used to define the G- and D-bands, which are two peaks. In general, the intensity ratio (I_G/I_D) is a useful indicator of the ratio of sp^3 to sp^2 bonding in hydrogenated amorphous carbons or the size of sp^2 clusters[27]. The Raman spectra of BSC_1.0 (Fig 4b) showed the D and G bands at 1344 and 1591 cm^{-1}, respectively. The defects are associated with the intensity and the disordered carbon structure is related to the D band. The peaks of the G and D bands in the BSC_1.0 suggest the graphitic structure with defects in the activated carbon[27]. The I(G)/I(D) ratio (1.22) in Raman spectra of BSC_1.0 attributes defects produced during the activation process.

Adsorption isotherms

The adsorption efficiency of BSC_1.0 was evaluated using Langmuir and the Freundlich adsorption model for methylene blue adsorption. The linearized curves of Langmuir and Freundlich isotherms were plotted in Fig 5a and 5b, respectively. Figure 5a shows that the relation between C_e and C_e/q_e is linear with a coefficient of determination, R^2 of 1.0 (Table 2). The Langmuir constant and maximum adsorption capacity calculated from the Langmuir model were 1.07 and 227.27 mg/g, respectively (Table 2). The excellent correlation between the parameters is attributed to homogeneous monolayer adsorption of methylene blue on the surface of BSC_1.0. The linear relationship between log q_e and log C_e and a high (0.8829) coefficient of determination corresponds to the well-fitted Freundlich adsorption model (Fig. 5b, Table 2). This value is lower than that for the Langmuir model, which suggests that the BSC_1.0 activated carbon was more suitable for the Langmuir isotherm model i.e., there was the presence of ionic bonding between the negatively charged activated carbon and positively charged methylene blue[6].

Remediation of Bagmati River water

Bagmati River passes through the core city of the Kathmandu Valley. The previous studies concluded that river water is excessively polluted and observed water quality parameters exceeded the WHO limit for drinking water[18,20,31]. In this study, phosphoric acid-activated bel shell powder (BSC_1.0) was deployed to treat heavily polluted Bagmati River water. The values of observed water quality parameters of Bagmati River water collected from Balkhu, Kathmandu in the winter season were presented in Table 3. As shown in Fig 6a, the river water is completely black. Hardness, alkalinity, phosphate, ammonium ions and chlorine demand were very high and exceeded the limits of the WHO standard[19]. The observed water quality parameter suggested that Bagmati River water cannot be used for

any purposes without treatment. Excitingly, treatment with BSC_1.0 excessively decreased the pollutants and turned black water into clear water (Fig 6b). The pH of the river water was slightly alkaline (7.4) and slightly increased to pH 7.69 after treatment. The slight increase in pH after treatment may be because of ion exchange interactions with surface functional groups which

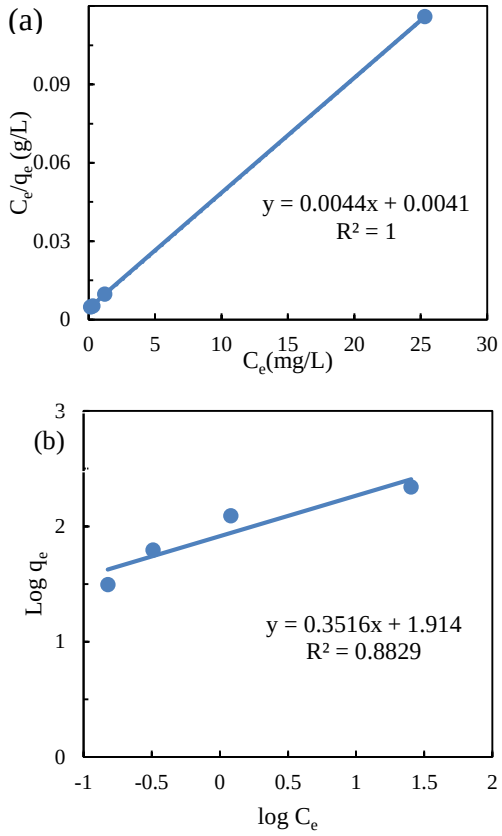


Figure 5: (a) Langmuir adsorption isotherm and (b) Freundlich adsorption isotherm.

Table 2: Langmuir and Freundlich adsorption parameters.

Langmuir parameters	Freundlich parameters
K_L = 1.07 L/gm	K_F = 82.04
q_{max} = 227.27 mg/g	n = 2.84
R^2 = 1.0	$1/n$ = 0.35
	R^2 = 0.8829

generate hydroxide (OH^-) ions and increase pH. The conductivity and total dissolved solids (TDS) were 823 µS/cm and 411 ppm, respectively before treatment and reduced by 5 % after treatment. The alkalinity and total hardness were 360.0 and 910 ppm, respectively. Both parameters were nearly 2 times higher than that recommended by WHO (Table 4). After treatment, the alkalinity and total hardness were reduced by 89.58 and 96.61 %, respectively following the WHO limits[32,33] (Table 3). This massive reduction in the concentration suggested that bel shell-derived activated carbon can efficiently remove pollutants.

The anions such as chloride, phosphate, sulphate, and nitrate ions are the pollution-indicating parameters. The observed concentrations of nitrate and phosphate exceeded the WHO limits though the sulfate and

(a) (b)

Figure 6 : Bagmati River water (a) before and (b) after treatment with BSC_1.0.

chloride ion concentrations were within the WHO limit. After treatment 91.61, 87.58, 73.96 and 62.61 % of sulphate, phosphate, chloride, and nitrate ions were removed respectively[34]. Similarly, BSC-1.0 reduced chromium and iron from 1.716 and 1.149 ppm to 0.116 and 0.0026 ppm, respectively. As indicated by Boehm titration the surface of activated carbon consists of acidic and basic functional groups that efficiently adsorbed oppositely charged ions from heavily polluted river water[18,32-34]. The reduction of more than 80% of all observed parameters suggested that bel shell-derived activated carbon efficiently reduced pollutants to the WHO recommended value indicating that heavily polluted river recommended value indicating that heavily polluted river water can be used for agricultural, industrial, irrigation and livestock drinking purposes after treatment provided that all other pollutants of concern treatment are also within the recommended value.

Conclusions

The bel shell powder was treated with phosphoric acid and carbonized in a tube furnace at 400℃ for three

hours in the inert atmosphere of N_2 gas. The activated carbons treated with different ratios of phosphoric acid were characterized by iodine adsorption. The iodine number (a maximum of 850.36 mg/g) indicated that the 1:1 ratio of precursor and phosphoric acid was better for developing pores on the surface of bel shell-activated carbon. Further surface characteristics of the bel shell-derived activated carbon (BSC_1.0) was determined by using Scanning Electron Microscopy, Boehm's titration, Fourier-transform Infrared Spectroscopy (FTIR), Raman spectroscopy, and point of zero charge (pH_{pzc}). The SEM images suggested that the surface amorphous activated carbon consists of macro, meso and micropores for the adsorption of foreign particles.

The Boehm titration and FTIR spectrum show that acidic and basic functional groups are available on the surface of activated carbon with the point of zero charges 6.45. It was observed that both the Langmuir and Freundlich models fitted well in methylene blue adsorption isotherm though, the Langmuir model is more suitable than the Freundlich model suggesting surface charges are more active to remove charged particles from aqueous solution.

The observed physico-chemical parameters of Bagmati River water suggested that the river water is excessively contaminated by ions and heavy metals turning to black water. The alkalinity and hardness of river water were nearly 2 times higher than the WHO-recommended value. A very low value of ORP (-214 mV) and black colour of river water indicated that the river water is contaminated by domestic effluent. Treatment with BSC_1.0 removed more than 80% of contaminants and changed the black colour of river water to clear water. Most of the measured parameters fall on the WHO limit after the treatment. It is concluded that bel shell-derived activated carbon is an excellent adsorbent for efficiently removing contaminants from extremely polluted Bagmati River water by reducing water quality parameters to within the WHO limit.

Acknowledgement

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Table 3: Physico-chemical parameters of river water before and after treatment with activated carbon.

Measured Parameters	Before treatment	After treatment	WHO limits[19]	Removal (%)
Color	black	Colorless	Colorless	
pH	7.4	7.69	6.5-8.5	-3.92
Conductivity (µS/cm)	823	781	1500	5.1
Turbidity (FNU)	333	29.9	<4	91.02
ORP (mV)	-214	91	-	-142.52
TDS (ppm)	411	369		5.11
Acidity (ppm)	112.5	18	500	84
Alkalinity (ppm)	360	37.5	200	89.58
Hardness (ppm)	910	28	500	96.92
Sulphate (ppm)	35.04	2.94	300	91.61
Phosphate (ppm)	13.465	1.67	0.1	87.58
Nitrate ions (ppm)	112.34	42	50	62.61
Chloride ions (ppm)	188.15	49	<5	73.96
Iron (ppm)	1.149	0.0026	0.3	99.77
Chromium (ppm)	1.716	0.116	0.05	93.27

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Data availability statement

The data presented in this study are available on request from the corresponding author upon reasonable request.

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