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Triphala Seed Stones-Derived Nanoporous Activated Carbon Materials with Excellent Adsorption Applications

Chhabi Lal Gnawali [a], Rinita Rajbhandari, Bhadra Prasad Pokharel

Department of Applied Science and Chemical Engineering, Pulchowk Campus, Institute of Engineering, Tribhuvan University, Nepal

Corresponding Author: [a]chhabig123@ioe.edu.np

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Abstract:
Biomass-derived activated carbon materials with nanoporous structure exhibit high surface area due to well-defined pore structure as a result of which the activated carbon from biomass could be suitable materials for high performance separation, purification technologies and energy storage application as efficient adsorbent. Here, the novel nano-porous activated carbon from Triphala (Harro, Barro and Amala) seed stones by chemical activation using Zinc Chloride as an activating agent is presented. The prepared activated carbon is characterized by iodine and methylene blue adsorption properties and scanning electron microscopy (SEM). Keeping other parameter constant, the effect of carbonization temperature of the activated carbon was studied. The experimental result indicates that Triphala seed stones and Zinc Chloride at mixing ratio1:1 by weight, carbonized for 4 hours at 500 ^{0}C shows high value of iodine number and methylene blue number as 856.8 mgg^{-1} and 373.5 mgg^{-1} respectively. The surface area, micropore volume and the total pore volume of the sample using quadratic model was found to be 908.9 m^{2}g^{-1}, 0.827 cm^{3}g$^-$ and 0.932 cm^{3}g^{-1} respectively. The prepared activated carbon possesses well-defined pore-size distribution with nanoporous structure These results show that Triphala seed stones derived nanoporous activated carbon will be a suitable material for water purification as well as energy applications.

Keywords: Triphala, Microporous activated carbon, Carbonization, Chemical activation, Quadratic model

1.Introduction

In the recent time, because of energy crisis, the developments in nanoscience and nanotechnology are expected to produce the advanced materials with smart properties which can be utilized for the production of efficient solar cell and the energy storage devices [1]. The excessive use of fossil fuels predicts the great threat to global economics mainly in the energy crisis and the environmental pollution. Thus, it is essential to search and develop the sustainable biowaste material that will solve the issue related to the environmental pollution and the energy supply. Recent trends show that the biowaste materials are used for the production of activated carbon to prepare the suitable material for the separation and energy applications.

The activated carbon (AC) is carbonaceous materials having well developed pores, internal surface area and high mechanical strength. Because of large surface area and well-developed porosity, the nanoporous AC will be suitable for the adsorption phenomena and the energy storage devices [2-4]. On the basis of pore size distribution, the AC are of microporous (pore size below 2 nm), mesoporous (2 < pore size <50 nm), and macroprous (pore size > 50 nm) structure. The AC with multiplicity of micropores and mesoporous are used for the adsorption of small molecules as well for the various applications related to separation and energy. The various agricultural waste biomass such as bamboo, coconut husk, wood, sugarcane, argan seed, date seeds, peanut shell and etc. [5-13] are used as a raw material to prepare activated carbon that can be used as the waste water treatment as well as the electrode material for supercapacitors. The agricultural waste biomass is used as the precursor because they are

renewable, locally available resources in large quantities and cost efficient.

Basically, there are two methods for the preparation of activated carbon which are physical or chemical activation. The physical activation can be carried out by direct carbonization of precursor at different temperatures, ranging from 800 ^{0}C to 1100 ^{0}C with constant flow of steam, carbon dioxide, nitrogen or air [14]. While, the chemical activation is carried out by the carbonization of the mixture of precursors with different activating agents like Zinc Chloride ($ZnCl_2$), Potassium Hydroxide (KOH), Phosphoric acid (H_3PO_4), Sodium hydroxide (NaOH), Sulphuric acid (H_2SO_4) etc. at temperature ranging from 400 to 900 ^{0}C under the inert atmosphere of nitrogen or argon gas. The activated carbon materials prepared from physical activation method have low specific surface area (500-1000 m^2g^{-1}) which are not suitable for separation as well as energy storage applications [15-17], while the activated carbon prepared from chemical activation results with very high surface areas (1000-2500 m^2g^{-1}) comparable with commercially available carbon materials [18-20]. Therefore, the chemical activation method has several advantages over the physical activation method because of high surface area, higher yield, lower activation temperature and can be performed in a single step.

Triphala comprises Harro (*Terminalia Chebula*), Barro (*Terminalia bellirica*), and Amala (*Phyllanthus emblica*), have found the formula to be potentially effective for several clinical uses such as appetite stimulation, reduction of hyperacidity, antioxidant, anti-inflammatory, antibacterial and etc. Triphala is an ayurvedic formulation with anticancer activities, so can be used for treating and preventing from cancer too [21-23]. However, no research work observed on activated carbon derived from Triphala seed stones so far but the supercapacitor applications of hierarchically porous carbon materials from *Phyllanthus emblica* is recently reported [24]. Therefore, there is particular interest towards the study of preparing a nanoporous carbon material from Triphala seed stones which are locally available in different regions of Nepal.

In this paper, the preparation of AC from Triphala seed stones (mixture of Harro Barro and Amala precursor in 1:1 weight ratio) by chemical activation method using Zinc Chloride as an activating agent is presented. Triphala seed stones powder was mixed with Zinc Chloride in 1:1 wt. ratio and carbonized at different temperatures (400, 500, 600 and 700 ^{0}C) for 4 h under the constant flow of ultrapure nitrogen gas. The prepared AC were named as MxC-Z400, MxC-Z500, MxC-Z600 and MxC-Z700 on the basis of carbonization temperature. The control sample by direct carbonization at 500 ^{0}C was also prepared and named as MxP-500. The Iodine number, Methylene blue number, surface area, the pore volume and SEM of the prepared activated carbon were recorded.

2. Material and Methods

2.1 Material

Triphala (Harro, Barro and Amala) were collected from local market of Kathmandu, Nepal. Zinc Chloride of FIZMERK, methylene blue of HiMedia were used. All the solutions were prepared in distilled water. During carbonization, ultra-high pure (UHP) nitrogen gas was used for the inert atmosphere.

2.2 Preparation of Nanoporous Activated Carbon

Triphala (Harro, Barro and Amala) were collected from local markets were peeled and the seed stones were separated. The obtained seed stones were washed for several times with distilled water and dried in oven at 50 ℃ for 24 hrs. Then the seed stones were crushed by using a grinder crusher and sieved through a mesh size of 300μm for the precursor. Directly carbonized carbon was prepared in tube furnace at inert atmosphere of nitrogen gas flow for 4 hrs. at 500 ℃ and labeled as MxP-500.

The activated carbons from Triphala were prepared by chemical activation method using $ZnCl_2$ as an activating agent. The precursors of particle size $300\mu m$ were chemically activated with $ZnCl_2$ in the ratio of 1:1 and left for 24 hours at 30 ^{0}C. The mixture was heated in the hot plate till the dry mass was obtained. The dry mass was transferred to a quartz tube, the carbonization was carried out in tube furnace (Accumax India) under the continuous flow

of nitrogen gas and the effect of carbonization temperature 400, 500, 600 and 700℃. The prepared activated carbons were cooled to room temperature, then it was treated with 5% HCl and then with distilled water for several times until the supernatant liquid attained the pH of 7 and then dried at 100℃ for 3 hrs. The prepared activated carbons prepared at 400, 500, 600 and 700℃ were labelled as MxC-Z400, MxC-Z500, MxC-Z600 and MxC-Z700. The effect of carbonization temperatures on iodine number, methylene blue number and surface morphology of the prepared activated carbons were studied.

2.3 Yield of Prepared Activated Carbon

The yield of prepared activated carbon was calculated on a chemical-free basis and regarded as an indicator of the process, efficiency for the chemical activation process. The yield of activated carbon was calculated by using the following equation.

$$\text{Yield } (\%) = \frac{\text{Weight of Activated Carbon after carbonization}}{\text{Weight of seed powder precursor}} 100\% \quad \ldots(1)$$

2.4 Characterization

The Iodine number of prepared activated carbon was calculated by using American Society for Testing Materials (ASTM) D46707-94 method [25]. Methylene blue number of prepared activated carbon was calculated by using standard method [26], single point adsorption isotherm studies. The concentration of MB was analyzed by using UV-visible spectrophotometer (CECIL-CE-1020) at absorbance 664 nm. The scanning electron microscopy was carried out by using SEM: S-4800, Hitachi Co., Ltd. Tokyo, Japan operated at 10 kV.

2.5 Iodine Number

Iodine number (I_N) indicates the micro-porosity of activated carbon materials and is defined as the milligram of iodine adsorbed per gram of carbon [27]. Iodine number was determined by following the standard method [25]. To determine the iodine number, 5 mL of 5% HCl solution was added to 100 mg activated carbon and boiled for 30 mins. After cooling the solution, 10 mL of 0.1 N iodine solution was added and shaken vigorously for 1min, filtered and washed with distilled water. The whole filtrate was titrated against 0.1 N sodium thiosulphate solution using starch as indicator. The iodine number was calculated as:

$$\text{IN} = \frac{\text{weight of iodine adsorbed on carbon (mg)}}{\text{weight of carbon (g)}} \quad \ldots(2)$$

2.6 Methylene Blue Number

Methylene blue number (MBN) indicates the meso-porosity of activated carbon materials and is defined as the maximum amount of dye in mg adsorbed per gram of adsorbent. MBN of activated carbon materials was determined by single point adsorption isotherm studies [26]. In the single-point adsorption isotherm method, 0.1 g of AC was mixed with 75 mL of 1000 ppm MB solution each separately. The suspension was equilibrated in an electric shaker at 200 rpm for 3hrs at room temperature (25 ℃). After that the solution was filtered and the remaining concentration of MB was analyzed using UV-visible spectrophotometer (CECIL-CE-1020) at absorbance 664 nm. The MBN was calculated as:

$$\text{MBN} = \frac{(\text{Co} - \text{Ce}) \times \text{V}}{\text{M}} \quad \ldots(3)$$

Where C_o = Initial concentration of MB solution (mgL^{-1}), C_e = concentration of MB solution at equilibrium time (mgL^{-1}), V = volume of solution (L) and M = mass of activated carbon added (g)

2.7 Surface area, micro and total pore volume

The surface area (S) micropore volume (V_m) and total pore volume (V_t) of the prepared activated carbons are the function of Iodine number and Methylene Blue number. The surface area and the micropore volume of activated carbon using quadratic model [27] while the total volume by using linear model [27], as given by the following equations.

$$S\,(m^2g^{-1}) = 2.28 \times 10^{2} - 1.01 \times 10^{-1}\,MBN + 3.00 \times 10^{-1}\,IN + 1.05 \times 10^{-4}\,MBN^2 + 2.00 \times 10^{-4}\,IN^2 + 9.38 \times 10^{-4}\,MBN\,IN \quad (4)$$

$$V_m(cm^3g^{-1}) = 5.56 \times 10^{-2} - 1.00 \times 10^{-3}\,MBN + 1.55 \times 10^{-4}\,IN + 7.00 \times 10^{-6}\,MBN^2 + 1.00 \times 10^{-7}\,IN^2 - 1.18 \times 10^{-7}\,MBN\,IN \quad (5)$$

$$V_t\,(cm^3g^{-1}) = 1.39 \times 10^{-1} + 1.90 \times 10^{-3}\,MBN + 1.00 \times 10^{-4}\,IN \quad (6)$$

3. Result and Discussion

The nanoporous activated carbons by chemical activation with Zinc Chloride from Triphala seed stones were prepared and characterized by Iodine number, Methylene blue number, surface area, pore volume and scanning electron microscopy.

3.1 Iodine and Methylene Blue Number

The variation of Iodine number (IN) and the methylene blue number (MBN) as a function of carbonization temperature are presented keeping the ratio and carbonization time constant.

Iodine number (IN) indicates the microporosity of prepared carbon materials. The variation of IN with carbonization temperature is shown in figure 1. Iodine number (IN) of the control sample by direct carbonization at 500 ^{0}C MxP-500 possess the low IN whereas AC with $ZnCl_2$ shows the significant increase of IN because this activating agent $ZnCl_2$ enters the interior of the particles whereby it acts as a template for the creation of the micropores. Increase in carbonization temperature has led to increase in microporosity as indicated by iodine number and then decreased after specific temperature. The increase in micropores may be due to the opening of inaccessible pores and expanding of former pore structures. The decrease in micropores after 500 °C may be due to the degeneration of micropores into mesopores or macropores structure.

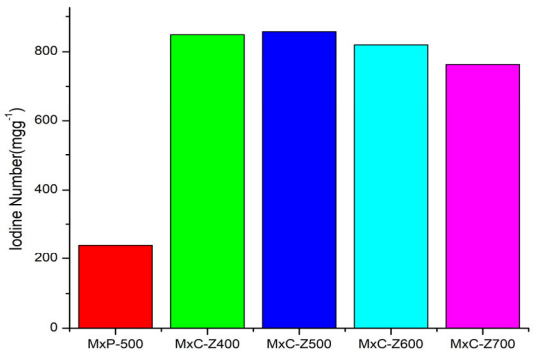

Figure 1: Variation of Iodine number with temperature

Methylene blue number indicates the mesoporosity of prepared carbon materials. As shown in figure 2, the MBN increased with the increase in carbonization temperature up to 500 ^{0}C and then decreased. The decrease in MBN after a specific temperature may be due to the rupture of pore walls to form macropores or due to destruction of mesopores forming macropores.

The low value of IN and MBN for directly carbonized sample (MxP-500) indicates the lack of porosity

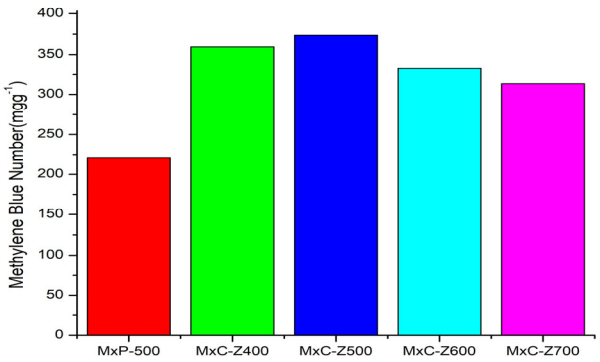

Figure 2: Variation of Methylene blue number with temperature

3.2 Surface area, micropore volume and total pore volume

The surface area and the total pore volume of the prepared activated carbon as the function of Iodine Number and the Methylene Blue Number, can be calculated by using quadratic model [27]. The

surface area, micropore volume and total pore volume of prepared activated carbon as a function of carbonization temperature is shown in figure 3, figure 4 and figure 5. respectively. These figures show that the surface area, micropore volume and total pore volume of prepared activated carbon ranges from 343.4 to 908.9 m^2g^{-1}, 0.214 to 0.827 cm^3g^{-1} and from 0.580 to 0.932 cm^3g^{-1} respectively.

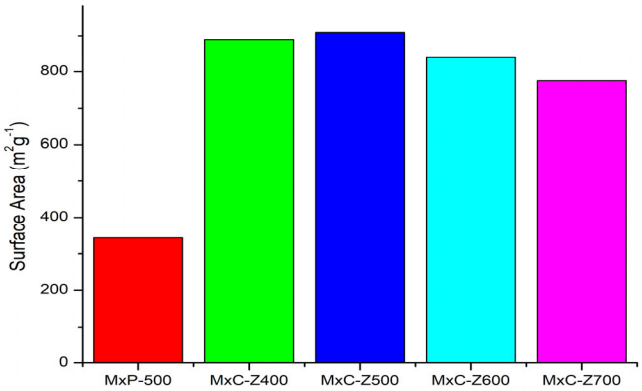

Figure 3: Variation of surface area with temperature

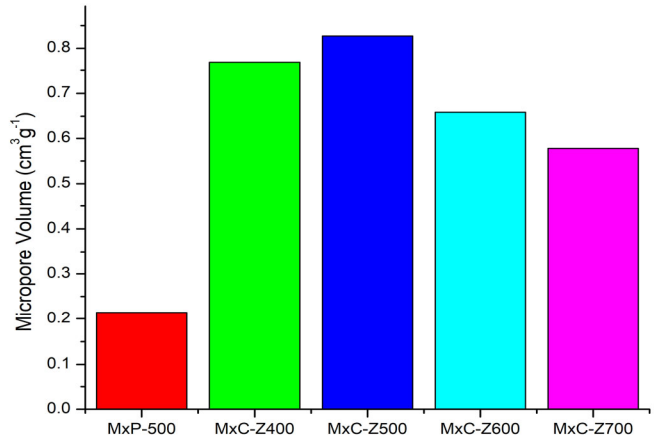

Figure 4: Variation of micropore volume with temperature

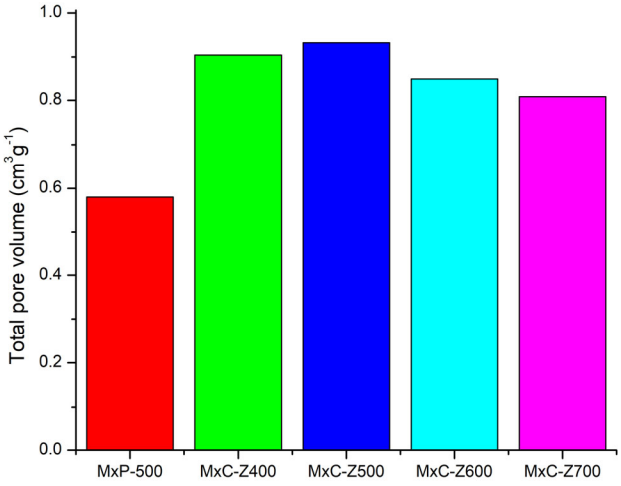

Figure 5: Variation of total pore volume with temperature

3.3 Yield

The yield (%) of activated carbon materials were studied by changing the carbonization temperature as it is an essential parameter in the preparation of nanoporous activated carbon materials in terms of industrial cost production Figure 5. shows the yield vs. carbonization temperature. From figure it is observed that the yield of activated carbon gradually decreases with the increase in carbonization temperature, this may be due to release of gaseous products, enhancement of carbon burn-off and may be due to the release of volatile matters [28].

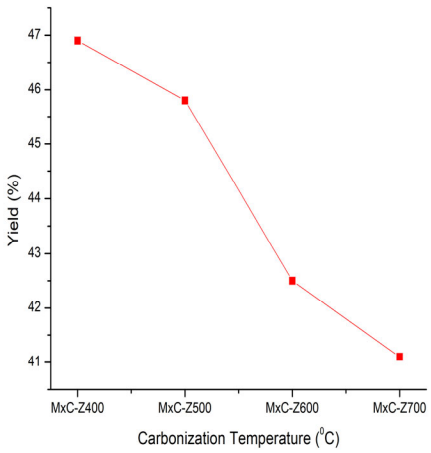

Figure 6: The effect of carbonization temperature on yield (%)

3.4 Scanning electron microscope SEM images

The surface morphology and the porous structure of the prepared nanoporous activated carbon from Triphala seed stones was studied by using SEM imaging. The SEM images reveal that the surface porosity of directly carbonized sample is low (figures 7a, b), this may be due to low adsorption of nitrogen. The SEM images of the activated samples (figure 7c, d-MxC-Z400 and 7e, f-MxC-Z500), shows the carbon particles with inhomogeneous shape and size with macropores at low resolution while at high resolution we can observed the nanoporous structure. Thus, we observe that the activating agent increases the surface porosity of the activated carbon this is due to the nitrogen adsorption phenomena [24,29].

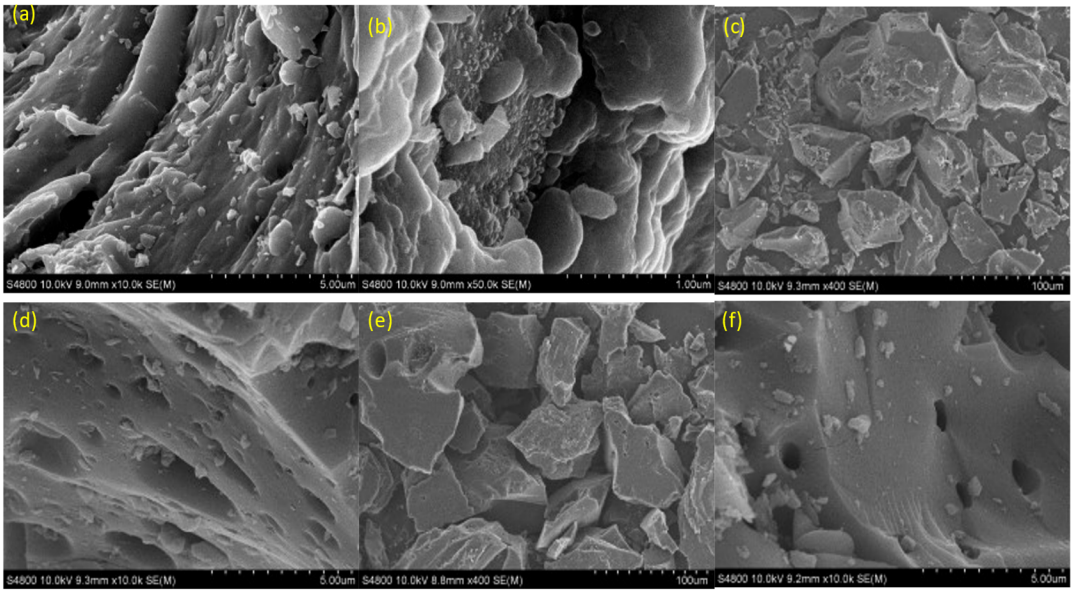

Figure 7: Scanning electron microscope (SEM) images of activated carbon materials. (a,b) MxP-500, (c,d) MxC-Z400 and (e,f) MxC-Z500

5. Conclusions

In conclusion, we have successfully prepared nanoporous activated carbon materials from novel precursor, Triphala seed stones by chemical activation with zinc chloride by varying the carbonization temperature and their adsorption performance for the iodine and methylene blue was studied. The iodine number and the methylene blue numbers of the prepared nanoporous activated carbon ranges from 762.0 to 856.8 mgg^{-1} and from 313.4 to 373.5 mgg^{-1} respectively. The prepared carbon materials have high surface area ranges from775.3 to 908.9 m^2g^{-1}, large micropore volume ranges from 0.578 to 0.827 cm^3g^{-1} and large pore volume ranges from 0.808 to 0.932 cm^3g^{-1} by using quadratic model. The Zinc Chloride activated carbon materials from Triphala possessing micro and mesoporous structure. From these results we conclude that the bio-waste material like Triphala seed stones could be the potential source for the preparation of nanoporous activated carbon that will be suitable for the preparation of electrode material for high performance supercapacitors as well for the waste water treatment process.

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