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Preparation of Waste Betel Nut based Activated Carbon (AC) to Test its Storage Performance

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Abstract

Energy storage is the main focus of today's world and in the scientific community.Due to the limited of the fossil fuels and the energy storage issues in the electrostatic field, a very high power density fast charging supercapacitor has emerged as the best potentials in energy storage development. Activated carbon (AC) due to its many fascinating properties like high porosity,low cost and easy processing, chemical and thermal stability, AC has become one of the major candidate electrode materials for the capacitor. Different agro-waste based AC carbons are on investigations for the applications in the fabrication of supercapacitor and solid state hydrogen stores. In this study, waste betel nut based AC was prepared by chemical activation with H_3PO_4 as an activating by mixing finely crushed waste betel nut powder and H_3PO_4 in ratio 1:1 (by weight) followed by carbonization at 500℃ for 3 h in nitrogen gas environment. Characterization of the resultant AC were performed by idoine number (I_N), methylene blue (MB_N), surface area, X-ray diffraction (XRD), Fourier infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Raman Spectroscopy.The methylene blue, iodine number and surface are of the resultant AC ware found to be 365 mgg^{-1},882 mgg^{-1} and $927m^2g^{-1}$. The SEM image signifies that there is presence of mesopores on the surface of the resultant AC which means it has high adsorptive efficiency favourable for many applications like energy storage, dye removal and decontamination.

Keywords

Betel nut, activated carbon, energy storage, chemical activation, supercapacitor

1. Introduction

Activated carbon (AC) is microcrystalline, amorphous, isotropic, complex carbonaceous,non-graphitisable, non-graphitic [1] with highly disordered microstructured compound composed of carbon (80-90 % by weight), water, ash, volatile matter and impurities . AC is a type of carbon species categories that is processed and prepared from carbon-rich coal or lignocellulosic feedstock to have high porosity and higher surface area. It has been used in many research field such as adsorption of heavy metals, energy storage, water purification, cosmetics, etc [2].ACs may be called active carbons due to their highly developed porosity and internal surface area. The large surface area of the ACs indicates, it has high adsorbing capacity which can be beneficial for the removal or absorption of unwanted chemicals from fluids, gases or any sample whose surface zone ranging from 800 to 1500 m^2g^{-1} as determined by the adsorption of the nitrogen gas .Differences in pore sizes influences the adsorption capacity for molecules of various shapes and measures, ant it is one of the criteria by which carbons are chosen for an explicit application . Pores of ACs as classified by IUPAC are of three groups namely micropores having pore diameter less than 2nm, mesopores ranging from 2 nm to 50 nm and macropores having pore diameter greater than 50 nm [3]. Mircroporous abundant ACs are suitable for absorption of small molecules and highly developed mesoporous ACs are suitable for the large molecules. ACs are generally dark in color and has amorphous structure. It is tasteless non-toxic adsorbent with larger surface area having three class of pore size distribution after a sequential processing of the carbon-rich material by physical and chemical processes including carbonization in inert atmosphere, activation of carbonized product, acid/base cleaned and further washing. Carbonization produces the fixed carbon characterized by rudimentary porosity and the activation enlarges the pore diameter and creates new pores. ACs can be utilized for both physical and chemical adsorption of small and macro molecules in gas and liquid phase. It can also be applied to do many functions like purification, refining, decontamination, deodorization/ decolorization [4, 5] and sterization [6]. In industrial application, due to its suitable adsorptive property to both liquid to gaseous phase and its low cost, AC is one of the most widely applied adsorbents and has become most essential adsorbent components in various industries for pharmaceuticals, foods, chemicals, environment-protection, water treatment, power generation and agricultural industry [7]. AC is employed in purification of gases, water and gold. ACs are also in used in gas masks and respirators as air filters. AC is important component for metal cutting, metal forming and metal finishing process. Application of tool materials varies with variations in carbon concentration in the tool materials. It can also be used for removing organic impurities from bright nickel-plating solutions. Smoothness, brightness, ductility and deposit qualities of electroplating solutions are improved by adding different organic chemicals. The impurities in the compounds are removed by activated carbon treatment to restore plating performance to the desired level [8] In environment field, due to its excellent absorptive property, ACs can be employed for numerous applications such as spill clean-up, drinking water filtration, air purification, dye removal, volatile organic compound adsorption, dry cleaning and other processes [9]. Arsenic contamination in ground water has become alarming issues in the areas where the population density is high . In case of Nepal, it has become an issue of growing concern, particularly in Terai region . So, removal of Arsenic from ground water is very important for the good quality drinking water which can be carried by the AC by using it as the adsorbent.Different researches are going on for the production low cost AC in Nepal world by utilizing carbonaceous agro-wastes products [10]. Activated carbon fiber filters are used for the indoor air purification and different physico-chemical and biological technique from color removal from textile wastewaters mainly rely on activated carbons due to its AC-enhanced coagulation and membrane filtration process, catalytic property in advanced oxidation process and dye reduction process and its oxidizing agents production in dye oxidation [11]. In medical applications, activated charcoal is used to treat poisonings and overdoses following oral ingestion and is considered universal antidote for many poisonings [12]. AC binds to poison and prevent its absorption by the gastrointestinal tract. The rate of diffusion of Granular activated carbon (GAC) is faster and is considered suitable for gases and vapour adsorption. Activated charcoal is also used as sorbents for treating patients with chronic kidney disease. Different investigations suggest that, dialysis process is improved by application of activated charcoal which removes urinal toxins, urea, indoxyl sulfates and is considered one of the strong sorbents [13] .For various medical and industrial applications, pellets of activated carbons are produced for vapour adsorption. Water filtration in fish aquariums also utilizes activated carbons namely called aquarium charcoal. Arsenic is a crystalline metalloid with atomic number 33 and is one of the 20^{th} abundant element in the earth's crust also known as King of Poisons [14]. The most urgent danger concern of arsenic is drinking water along with air and food. And, in some places, the risk of Arsenic contamination is particularly high in water resources [15]. In Nepal, arsenic contamination is major issue of current drinking water systems using groundwater and is one of the major environmental health management issues especially in Terai districts, where the population density is comparatively high. The level of arsenic allowed in drinking water has been set at $0.01mgl^{-1}$ by World Health Organization (2011) and Nepal has also set drinking water quality standard of $0.05mgl^{-1}$. National Sanitation Steering Committee (NSSC) collaborated with many other organizations and investigated arsenic blanket test by analyzing 737,009 groundwater samples in 25 districts of Nepal.From the results, it is found that in the 89.8% of cases arsenic concentration lower than 10 ppb in the 7.9% of cases concentrations between 10 and 50 ppb, and larger than 50 ppb in the 2.3% of samples. These numbers indicate a serious mass poisoning, considering the severe consequences of chronic arsenic contamination caused by drinking water. Taking into consideration the problem of the highly toxic arsenic content in the drinking water, different researches are going on for the production of agro-waste based AC in Nepal and in many parts of the world for the arsenic removal from contaminated water. Moreover, for increment of the adsorption efficiency of the resultant AC, magnetic nanocomposites of the ACs can be fabricated which even can be regenerated for the reuse due to the magnetic characteristic of the so prepared nanocomposite. The agro-wastes are the pollutants themselves and preparation of the AC from the agro-wastes for the pollution control is very beneficial since one pollutant can be utilized for the adsorption of another pollutant.

2. Experimental Methods

2.1 Materials used and preparation method

Waste betel nuts were purchased from local market, Kalimati, Kathmandu at low price. It was washed repeatedly with tap water and then with distilled water several times and dried in oven for about 24 hrs at 110 °C, then, broken into small pieces with traditional machine Khal and grinded into powder with electric grinder and mortar. The powder was sieved to get fine powder of particle size 312 μm.

2.2 Preparation of activated carbon

AC can be prepared either by physical method or chemical method of activation.A physical method of activation is sequential two stage process involving carbonization of the carbonaceous precursor in an inert environment and activation of the resultant char by carbon controlled gasification by suitable carbondioxide presence, steam flow,heating rate and temperature [16]. A chemical activation is single step process where precursor is mixed with activating different suitable activating agents like $ZnCl_2$,H_3PO_4,NaOH,KOH,$FeCl_3$,H_2SO_4 etc and carbonized followed by activation in an inert environment in presence of nitrogen [17]. Chemical method of activation is preferred over physical method of activation since it is faster process and can be activated at lower temperature. The resultant AC obtained from the chemical method of activation has advantages of high surface area and pore development and high carbon yield, however,it needs a lot of washing for the removal of the inorganic residuals which is pollutant to the environment [18]. In this study, 20 gm of the nut powder were mixed with H_3PO_4 in the ratio of 1:1 by weight and stirred with magnetic stirrer at 70 °C until party dried. Then, the resulting mixture was dried in an air oven at 100 °C for 24 h. For carbonization, the dried sample was taken out from the oven and grinded gently on crucible furnace.The sample was transferred into in quartz tube which can withstand very high temperature of around 2000°C. The tube containing the mixture was placed inside an electric horizontal tube furnace and the carbonization of the activated carbon was carried out in an inert environment by continuous flow of Nitrogen gas at the rate of 75 $mlmin^{-}1$ maintaining temperature of furnace at 500°C for 3 h. After 3 h the sample was taken out and kept at oven maintaining the temperature of 100°C for 24 h. The resultant AC was treated with 1% $NaHCO_3$ followed by repeated washed by warm distilled to neutralize the as prepared AC. The resultant AC sample was then dried in an air oven at 110 °C for 24 h and sieved to get the particle of a size 106 μm.

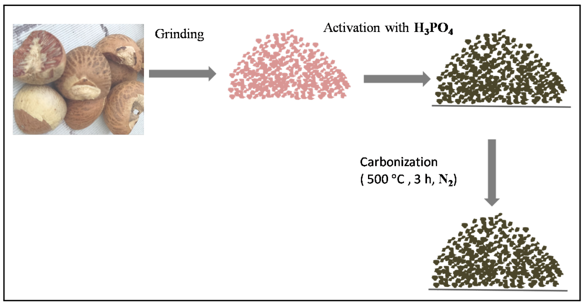

Figure 1: Preparation of AC from waste betel nut

2.3 Characterization

The resultant AC was first characterized by determining the iodine number, methylene blue and surface area .The iodine number was determined by ASTMD4607 standard [19]but the presence of volatiles, sulfur, water might effect the iodine number. The methylene blue was determined by single point isotherm studies method.X-ray (XRD) analysis and Raman Spectroscopy were performed to determine its structural property, the presence of functional group was determined by using Fourier transform infrared(FTIR) spectra, nature of the pores determined from the Scanning electron microscopy (SEM) image by its observing surface topography.

3. Results and Discussion

3.1 Iodine number, methylene blue and surface area

The iodine number was determined by ASTMD4607 standard, the methylene blue was determined by single point isotherm studies method and surface area by multiple regression. The methylene blue, iodine number and surface are of the resultant AC ware found to be 365 $mgg^{-}1$,882 $mgg^{-}1$ and 927$m^2g^{-}1$. [20, 21].

3.2 XRD analysis

XRD analysis is done to identity the structural property of the AC.The XRD of the resultant AC shows broad peak at 2θ of about 24.3°which corresponds to the (002) plane of carbon. Another weak peak at $2\theta$of about 43.8 was observed which corresponds to the (100) plane of carbon. This plane suggests the graphitic nature of carbon. Graphitic carbon is conductive and very useful for its application as energy storage material [22]

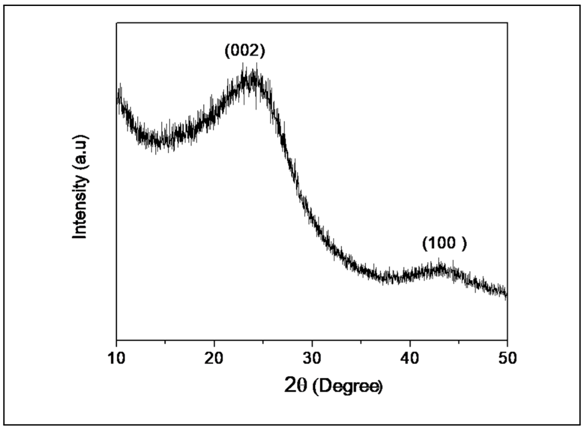

Figure 2: XRD pattern of waste betel nut based AC

3.3 FTIR Analysis

The FTIR pattern of the resultant AC shows that the first broad peak was observed at 3422 Cm^{-1} which represent the presence of -OH functional groups. Another peaks were observed at3034 Cm^{-1}, 1707 Cm^{-1}, 1585Cm^{-1}, 1434 Cm^{-1}, 1244 Cm^{-1}, 883 Cm^{-1} and 723 Cm^{-1} which represents the presence of different functional groups like -OH, CO, COOH.

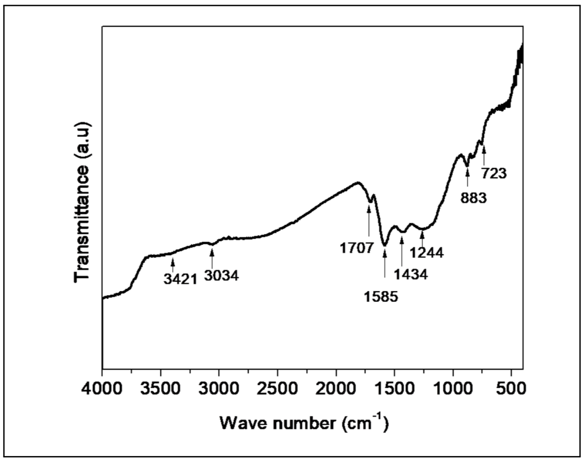

Figure 3: FTIR pattern of waste betel nut based AC

3.4 Raman Spectroscopy

Raman spectrocopy was performed to identify and compare structure as determined by the XRD analysis.The Raman Spectra pattern of the the resultant AC shows D band and G band at 1348.9 $cm^{-}1$ and 1588.9 $cm^{-}1$.The corresponding intensities of the D band and G band are I_D=267.9 and I_G=317.6 respectively.The intensity ratio in the spectra of the D band and G band gives the structure of the carbon material.The intensity ratio was(I_D/I_G) was calculated to be 0.84 which shows the resultant AC is graphitic in nature [23, 24] .

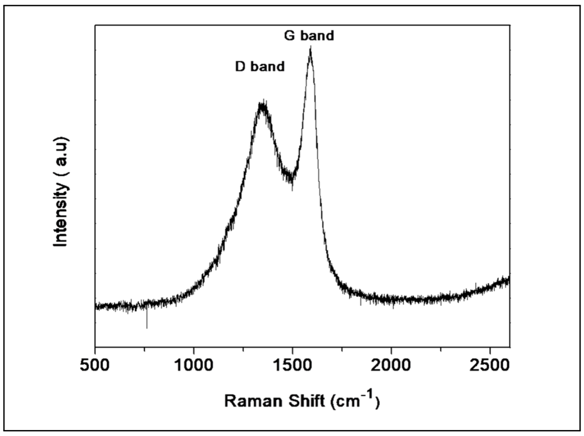

Figure 4: Raman spectroscopy of waste betel nut based AC

3.5 SEM image

The SEM image analysis was performed to observe the physical surface morphology and porosity of the activated carbon. The SEM image of the resultant AC as prepared from the waste betel nut analyzed. The presence of mesopores on the surface were obsereved which means it has good adsorptive property favorable for various application like purification, absorption electrode material of energy storage device [25].

To test the electrochemical performance of as prepared AC for supercapacitor, working electrode will be prepared. For this, nickel foam will be kept in 5% HCl in a clean beaker and sonicated for 10 minutes. It will be washed with distilled water repeatedly and dried in oven for about 6 h.4 g of AC will be mixed with 10 % polyvinylin difluoride (PVDF) and 10 % acetylene black grinded with piston by adding N-methyl-2-pyrrolidone (NMP) to get a slurry. It will be deposited on 1×1 cm^2 clean nickel foam using micropippete. It will be dried in oven at 80 °C for 6h. As prepared AC carbon based electrode will be used as working electrode. Plantinum (Pt) wire will be used as counter electrode and Ag/AgCl will be used as reference electrode. 3 M aqueous KOH will be used as electrolyte. The test will be carried out in electrochemical work station and analyse with the help of Cyclic voltametry curves and Galvanostatic charge discharge (GCD) curves.

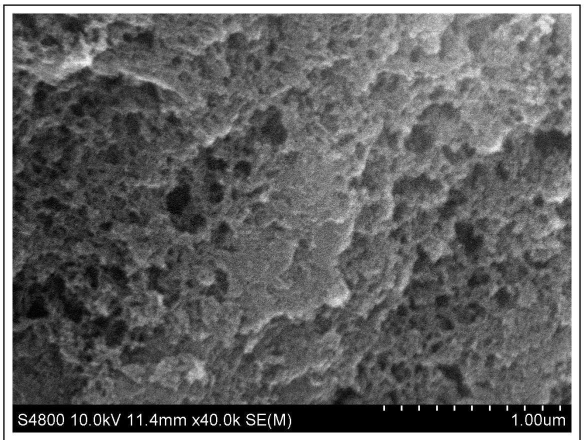

Figure 5: SEM image of waste betel nut based AC

4. Conclusion

The preparation of the betel nut waste AC was prepared from the waste betel nut by the chemical activation method. Characterization by iodine number, methylene blue and estimation of surface area directs the resultant AC to have high porosity.Further, analysis the resultant AC by SEM image shows the presence of mesopores on the surface. XRD and Raman Spetroscopy analysis were compared and concluded the graphitic nature of the resultant AC. FTIR analysis show the presence of different oxygenated functional group.As prepared AC concluded its good absorptive property preferable for many applications like purificationa and charge storage. As prepared AC will be further carried for charge storage application in supercapacitor.

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