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Preparation of Waste Betel Nut Based Activated Carbon and Test its Energy Storage Performance

By

Ram Bahadur Ale

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The undersigned certify that they have read, and recommended to the Institute of Engineering for acceptance, a thesis entitled "Preparation of Waste Betel Nut Based Activated Carbon and Test its Energy Storage Performance" submitted by Ram Bahadur Ale in partial fulfillment of the requirements for the degree of Masters in Material Science and Engineering.

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ABSTRACT

In this study, waste betel nut based AC was prepared by chemical method of activation of finely crushed and shieved powder of waste betel nut with H_3PO_4 in the ratio 1:1 (by weight) followed by carbonization at temperature of 500 °C. Characterization of the as prepared AC was performed by determination of iodine number (I_N), methylene blue (MB_N) and estimating its surface area followed by analysis of Scanning electron microscopy (SEM) image, X-ray diffraction (XRD) pattern, Raman spectroscopy pattern and Fourier transform infra red (FTIR) pattern. The MB_N, I_N and surface area of the resultant AC was determined to be 365 mg g^{-1},882 mg g^{-1} and 927 m^2 g^{-1}. These analyses suggested the formation of AC with high specific surface area and high porosity from waste betel nut, successfully. The cyclic (CV) curve galvanostatic charge discharge curves shows that as prepared working electrode fabricated by using as waste betel nut based AC, exhibits specific capacitance increases to 154 F g^{-1} at current density of 0.5 Ag^{-1}. This suggests a probable use of as prepared AC as supercapacitor electrode material. Moreover, preparation of AC from waste betel nut is a simple way of bio-waste management.

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

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List of abbreviations

AC	Activated carbon
BET	Brunner –Emmett-Treller
BJH	Barrett-Joyner-Halenda
CAC	Commercial activated carbon
EDLC	Electrical double layer capacitor
FTIR	Fourier transform infra red
GAC	Granular activated carbon
FESEM	Field emission scanning microscopy
IUPAC	International union of pure and applied chemistry
NSSC	National Sanitation Steering Committee
PAC	Powdered activated carbon
PCB	Printed Circuit Board
pHpzc	Point of zero charge
Redox	Reduction and oxidation
SEM	Scanning electron microscopy
TC	Total carbon
TMCs	Transition metal compounds
VOC	Volatile organic compounds
WHO	World health organization
XRD	X-ray diffraction

List of symbols

a.u	Arbitrary unit
$^{\circ}C$	Degree Celsius
g	Grams
h	Hours
mg g^{-1}	Milligrams per grams
m^2 g^{-1}	Square meter per grams
I_N	Iodine number
MB_N	Methylene Blue
$CaCl_2$	Calcium chloride
CO_2	Carbon dioxide
CoO	Cobalt oxide
Co_3O_4	Tricobalt tetroxide
$Co(OH)_2$	Cobalt hydroxide
Co_2P	Cobalt phosphide
CoS	Cobalt sulfide
$FeCl_3$	Ferric chloride
H_2SO_4	Sulfuric acid
H_3PO_4	Phosphoric acid
IrO_2	Iridium oxide
KCNs	Potassium thiocyanates
K_2CO_3	Potassium carbonate
KOH	Potassium hydroxide
MoS	Molydenum sulfide

MnO_2	Manganese dioxide
NaOH	Sodium hydroxide
NiO_2	Nicke dioxide
$Ni(OH)_2$	Nickel hydroxide
Ni_2P	Nickel phosphide
NMP	N-methyl-2-pyrrolidone
PVDF	Polyvinylin difluoride
RuO_2	Ruthenium oxide
V_2O_5	Vanadium pentoxide
$ZnCl_2$	Zinc chloride
ZnO	Zinc oxide

CHAPTER ONE: INTRODUCTION

1.1 Background

The increase in carbon emissions due to usage of fossil fuels and its depletion have become the main concern of the today's global community. The population growth, industrialization and the invention and the development of the new devices and appliances have highly increased the energy consumption of the globe. World Energy Council has expected energy consumption/demand to be twice the today's energy demand by 2050 (Dubey & Guruviah, 2019). So, researchers are focused on the development of high efficient, environmentally friendly and low cost energy production and storage devices like batteries, supercapacitors, fuel cells to fulfill the present and future estimated energy demand and consumption (Sevilla & Mokaya, 2014). In electrochemical double layer capacitor (EDLC) type supercapacitor, the performance of energy storage devices highly depends on electrochemical properties of electrode material. AC (Activated carbon) being low-cost highly porous, thermally stable and reliable material, many researchers are attracted to AC in its application as an electrode material for supercapacitors and solid state hydrogen store (Reddy, Gowda, Shaijumon, & Ajayan, 2012). The AC has easy and established methods of preparation and the AC based electrode materials for energy storage devices can be fabricated as per the requirement of the specific application by controlling porosity and texture (J. Gamby, P. Taberna, P. Simon, J. Fauvarque, & M. Chesneau, 2001). For the reasons, ACs are considered one of the appropriate electrode candidate materials in energy storage applications and are being commonly used (Ngai, 2022; Sevilla & Mokaya, 2014). In the past, AC has very common application as an adsorbent for purification, decontamination, filtration and dye removal but the more useful application as energy storage material has increased the usefulness of the AC (Sevilla & Mokaya, 2014). Different agro-waste based ACs are on investigation for the application as an electrode material due to its fascinating properties like porosity, chemical stability, thermal stability, low-cost and easy processing. The development and advancement in the carbon nanomaterials may have considerable benefits in the development of the energy storage devices to enhance the performance of the energy storage devices. The AC suitable for the application in the energy storage should have very high specific surface area, controllable microtexture permitting the

electrolyte access to the carbon electrode interface and low internal resistivity (J. Gamby, P. L. Taberna, P. Simon, J. F. Fauvarque, & M. Chesneau, 2001) . But, due to the low energy density of the capacity of the carbon based electrode, the focus is centered on the application of hybrid functional group nanostructured materials as an electrode in energy storage device (Reddy et al., 2012).

In this study, the waste betel nut based activated carbon synthesized was by chemical activation at 500 °C, by mixing waste betel nut powder and H_3PO_4 in ratio 1:1 in nitrogen environment. The tests and characterization of the resultant activated carbon (AC) showed the suitable characteristics as the electrode material for the supercapacitor. Then, the betel nut based AC was fabricated and its electrochemical tests were performed which showed that waste betel nut based AC can be one of the strong candidates for the electrode materials in supercapacitor.

1.2 Activated carbon (AC)

Activated carbon (AC) is microcrystalline, amorphous, isotropic, complex carbonaceous (Jones et al., 2021), non-graphitisable, non-graphitic (Yahya et al., 2018a) with highly disordered microstructured compound composed of carbon (80-90 % by weight) ,water, ash, volatile matter and impurities (Tennison, 2008). It is a type of carbon species categories that is processed and prepared from carbon-rich coal or lignocellosic feedstock (Jones et al., 2021) 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 (Foo & Hameed, 2009; Hammani et al., 2019). Due to highly developed porosity and internal surface area, ACs are also called active carbons. 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 (Heidarinejad et al., 2020). The most generally utilized commercial carbons have a particular surface zone ranging from 800 to 1500 $m^2 g^{-1}$ 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 (Saleem, Shahid, Hijab, Mackey, & McKay, 2019). Pores of ACs as classified by IUPAC are of three groups namely micropores having pore diameter less than 2 nm, mesopores ranging from 2 nm to 50 nm and macropores having pore

diameter greater than 50 nm (Sahira Joshi, 2017). Mircroporous ACs are suitable for absorption of small molecules and highly developed mesoporous ACs are suitable for the large molecules (Sahira Joshi, 2017). 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 (Ioannidou & Zabaniotou, 2007). 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 (Reza et al., 2020), deodorization/ decolorization (Lee & Shoda, 1989), and sterilization(Liang et al., 2022).

In industrial application, due to its fascinating adsorptive property to both liquid and 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 (Tennison, 2008) and agricultural industry (Jones et al., 2021). AC is employed in purification of gases, water and gold. ACs are also in used in gas masks and respirators for air filter applications. 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 (Sivakumar, Nouri, Modhini, & Deepalakshmi, 2018).

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. Arsenic contamination in ground water has become alarming issues in the areas where the population density is high (Mondal & Garg, 2017). 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 (Joshi, Sharma, Kumari, Shrestha, & Shrestha, 2019). Different researches are going on for the production low cost AC in Nepal world by utilizing carbonaceous agro-wastes products. Activated carbon fiber filters are used for the indoor air purification (Ao & Lee, 2005) 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 (Mezohegyi, van der Zee, Font, Fortuny, & Fabregat, 2012).

In medical applications, Activated charcoal is used to treat poisonings and overdoses following oral ingestion and is considered universal antidote for many poisonings (Heidarinejad et al., 2020) . 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 (Winchester & Ronco, 2009) and is considered one of the strong sorbents (Olson, 2010). 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.

AC is non-toxic carbonaceous amorphous, homogenous material characterized by its high microporous structure and surface area which may be as large as 1500 $m^2 g^{-1}$ and even as high as 3000 $m^2 g^{-1}$ (Saleem et al., 2019). Its availability and simplicity in methods of preparation with unique ability of absorption contributes to its low cost and higher efficiency (Baker, Miller, Repik, & Tolles, 2000) . For this reason, it is widely used and is considered one of the best adsorbents known. Mostly, ACs in granular forms are employed in the removal of organic and inorganic compounds for water purification. This also removes the odor, taste and color of the water due to contamination and improves the quality of the water. But, it cannot disinfect the microorganisms (Marsh & Reinoso, 2006) (Backer, 2017).

1.3 Raw materials used in the production of AC

AC is produced from variety carbon-rich raw materials such as wood, pineapple waste, sludge, sugarcane bagasse, waste tea, cherry stones (George, 2015), seed stones, husks, nut shells (Muzarpar, Leman, Rahman, Shayfull, & Irfan, 2020), bones peat, peels and plastics (D. Zhang et al., 2020) by both the physical and chemical activation process (Yeganeh, Kaghazchi, & Soleimani, 2006). New materials are currently under investigation as sources for AC for low cost carbon production (Raut, Thakur, & Chaudhari, 2021). Any carbonaceous organic compounds with high carbon concentration can be carbonized to get fixed carbon and activate to enlarge porous structure and hence enhance the adsorptive property (Du, Wang, Fu, Chen, & Wang, 2013). The carbonaceous material with minimum amount of organic compound can produce low cost activated carbon with high quality. But such raw materials have limited supply due to their explicit usage. As a result, wood is by far the most common source of activated carbon, followed closely by coal; coconut shell. Peat coals are also used in large quantities but they are expensive and not easily available. Different research shows that the porosity and absorption property of the ACs are greatly affected by the property of the precursor employed followed by activating agents and carbonization conditions (carbonization time, carbonization temperature, impregnation ratio) (Joshi & Homagai, 2017). So, ACs produced from different raw materials has different adsorbent quality even if the carbonization conditions and activation conditions are maintained at the similar condition (Yahya, Al-Qodah, & Ngah, 2015). During the selection of precursor/raw material for the production of the AC, availability, cost and carbon concentration are considered followed by amount of fixed carbon that can be extracted. The quality of the resultant AC is also of the one of the most important factor for the precursor selection.

1.4 Betel nut (*Areca catechu*)

The areca nut which is correctly called as betel nut is seed of *Areca catechu* fruit having size of about the plum stone (Arjungi, 1976). Betel nuts are generally hard kernel and are oval in shape and brown in color. Betel nuts are the core constituent of a variety of raw and purified products. Betel nut constitutes various chemicals such as tannins, alkaloids, fatty acids, flavonoids, triterpenoids, chrysophonic, physcione and other chemicals (Salehi et al., 2020). Alkaloid is one of the major and most active constituent toxic component of the betel nut, whereas, tannins is another major

constituent which make the betel nut astringent and bitter (X. Chen, He, & Deng, 2021) . It is used most commonly within the Indian sub-continent (India, Nepal, Pakistan, and Bangladesh), but is also prevalent in Taiwan and Southeast Asia which includes Indonesia, Thailand, Philippines, Cambodia, Loas, Guam, Malaysia among others, the Pacific rim and southern China. Data shows that, the largest betel nut production countries in the world includes Bangladesh, India and Malasiya (X. Chen et al., 2021). It is known by name Supari in India and Nepal, Daka in Papua and Guan in Thailand (Winstock, 2011). The nut is a hard brown oval kernel, about the size of a plum stone. Besides consumptions of nut in different forms, its husk can be used as raw material for insulating wool and paper manufacturing. So, its husk is also the reason of the cultivation of the areca palm. It grows in much of the tropical part of the Pacific Southeast and South Asia and regions of east Africa. In south countries, it is often wrapped inside betel leaves (paan) or with tobacco (betel quid) for their consumption, the composition of which varies in different populations and countries. It's consumption as addictive substances in the world is very high ranking the 4th after nicotine, ethanol and caffeine, and is consumed by approximately 10 % of the world's population. The consumption of betel nut is free and people starts its chewing at the very young age (Apurva Garg, 2014). Medical use of betel nut is very limited but many scientific research shows that it has various health hazards. Medical researches proved that the regular consumptions of betel nut may causes systemic illnesses, cancers, and various other diseases. It consumption is also related to toxicities and drug interactions. But, the greater hazard linked to its consumption is and malignancy and submucous fibrosis (Winstock, 2012).

1.5 Betel nut based activated carbons

The primary application of betel nut is chewing in different forms by boiling, with or without tobacco and wrapping in betel leaves, whereas the betel nut husks can be used as raw material for the paper and insulating wood manufacturing. However, the main concern is the value addition to the betel nut/ betel nut-husks product and also the utilization of the betel nut/betel nut-husks which are not suitable for direct consumption in any form (Bardhan, Novera, Tabassum, Islam, & Jawad, 2020). So, different researches are going on in Nepal and in different countries for the preparation and application of activated carbon from the waste betel nut. The waste betel nut based activated carbon can be prepared by the both the physical and

chemical method of activation (Joshi & Homagai, 2017). Investigation shows ,besides, the type of precursors, the properties of the activated carbon are greatly influenced by the activating agents like $ZnCl_2$, NaOH, H_3PO_4, $FeCl_3$ and carbonization conditions like carbonization temperature, carbonization time and the mixing ratio of carbonaceous powder and the activating agent (Joshi & Homagai, 2017). So, the waste betel nut based ACs of different properties can be prepared by altering the method of activation, controlling the carbonization conditions then the resultant AC can be employed for the large variety of the applications for the decontamination and adsorption (Baker et al., 2000).

1.6 Supercapacitor

Supercapacitor or ultracapacitor is an intermediate energy storage device which bridge the gaps between dielectric capacitors which has very high power density and electrochemical batteries which has low power density (Iro, Subramani, & Dash, 2016) . This means, supercapacitors combines the energy storage capacity and power delivering ability of electrochemical batteries and dielectric capacitors (Moftah & Al Shetiti, 2019). The supercapacitor is an electrochemical charge storage device having low internal resistance and high power density when compared to betteries (Frackowiak, 2007). In EDLC, the supercapacitors consist of an electrolyte in which two electrodes are separated electrically by a separator and involve the charge separation between the electrode/electrolyte interface (Devillers, Jemei, Péra, Bienaimé, & Gustin, 2014). In EDLC, the energy storage capacity of the capacitor depends on the separation of the electrode and electrolyte interface (Vangari, Pryor, & Jiang, 2013). Due to reduced separation distance of the electrode/electrolyte interface, supercapacitos can store energy of about 3-5 Wh kg^{-1} which is higher than the conventional dielectric capacitors (S. Liu, Wei, & Wang, 2020). And charge storing mechanism involving in supercapacitor led to higher power densities and more reversibility and higher life cycles compared to batteries (Sevilla & Mokaya, 2014). The most important component of the supercapacitor is the electrode material, however, electrochemical performance are also influenced by wetting of electrolyte, surface area, electrical resistivity, distance between electrode/electrolyte interface and the permeability of electrolyte solution (Iro et al., 2016). The faster charge discharge applications prefer supercapacitors while the slower one prefers batteries. Supercapacitors are used in fuel cell vehicles and hybrid low emission vehicles.

Supercapacitors have several advantageous properties over other charge storage devices which includes long life cycle, higher power density, environmentally friendly, wide thermal range from - 40 °C to 70 °C, high conductivity, portability and low weight (Iro et al., 2016). Based on the energy storage mechanism, supercapcitors are categorized in into three groups which are electrical double layer capacitor (EDLC), pseudocapacitor and hybrid capacitor (Dubey & Guruviah, 2019). In EDLC, the capacitive nature depends on the accumulation of the different pure electrostatic charges in electrode/electrolyte interface which is highly influenced by the surface area of the electrode material (Libich, Máca, Vondrák, Čech, & Sedlaříková, 2018). In pseudocapacitor, capacitive nature is due to fast and reversible redox process by the electro active species. The hybrid capacitance combines the capacitive nature of EDLC and pseudocapacitors (Poonam, Sharma, Arora, & Tripathi, 2019). Low resistivity is desirable property of the electrode materials for the supercapacitors (Moftah & Al Shetiti, 2019). Also, in EDLC type supercapacitor the surface area of the electrode material is the most importance factors determining its capacitive nature (Libich et al., 2018). So, the selection of electrode material is one of the crucial parts of the supercapacitor fabrication (Moftah & Al Shetiti, 2019).

Researchers are focused on the development of the new electrode materials to increase the energy density and reduce high self discharge to overcome the challenges faced by the supercapacitors (Poonam et al., 2019). Different materials like carbon materials, metal oxides, conducting polymers are used as the electrode materials (Poonam et al., 2019).

The operation of supercapacitor is based on the energy storage and ions of electrolyte accessible to surface area of the electrode. Based on the energy storage mechanism, supercapacitors are grouped into EDLC, pseudocapacitors and hybrid supercapacitors.

1.6.1 Electrical double layer capacitor (EDLC)

EDLC type of supercapacitor is a type of supercapacitor in which two porous electrodes are isolated by a separator in an electrolyte (Conway, 2013). The electrode materials used, in general, are conducting polymers, metal oxides and carbon materials which have high capacitance and high surface area (Kiamahalleh, Zein, Najafpour, SATA, & Buniran, 2012). The electrolyte is aqueous solution or organic solvent in which separator isolates two electrodes allowing transfer of ions

(Jayalakshmi & Balasubramanian, 2008). In EDLC type of supercapacitor, energy is stored due to the accumulation of non-faradic/electrostatic charge on the high surface area electrode/elctrolyte interface (Halper & Ellenbogen, 2006). When voltage is applied across the electrode, the ions on the electrolyte are accumulated/diffused to the porous electrode forming electrical double layer where the separation between electrode surface and centre of ion layer is extremely less (Iro et al., 2016). The less separation distance between electrical double layer and the high surface area of the electrode contributes to the high capacitance of the supercapacitor. The charging and discharging of the EDLC type supercapacitor is high due to storage mechanism and it has low energy density (Obreja, 2008). The involvement of electrostatic charge in the energy storage mechanism supports its greater life cycle compared to Li-ion batteries. However, today's research on EDLC is focused on the improvement of the energy density. Different nano structured materials are on investigation in the application of supercapacitors due to their high surface area, low resistivity, good mechanical behavior and stability (Obreja, 2008) (Frackowiak & Beguin, 2001). AC is one the most commonly used electrode materials in EDLC. Some of the drawbacks of the EDLC type supercapacitor are; limited lifespan of the electrolyte, low energy density, high internal resistance, limited temperature due to organic solvents etc (Mensah-Darkwa, Zequine, Kahol, & Gupta, 2019).

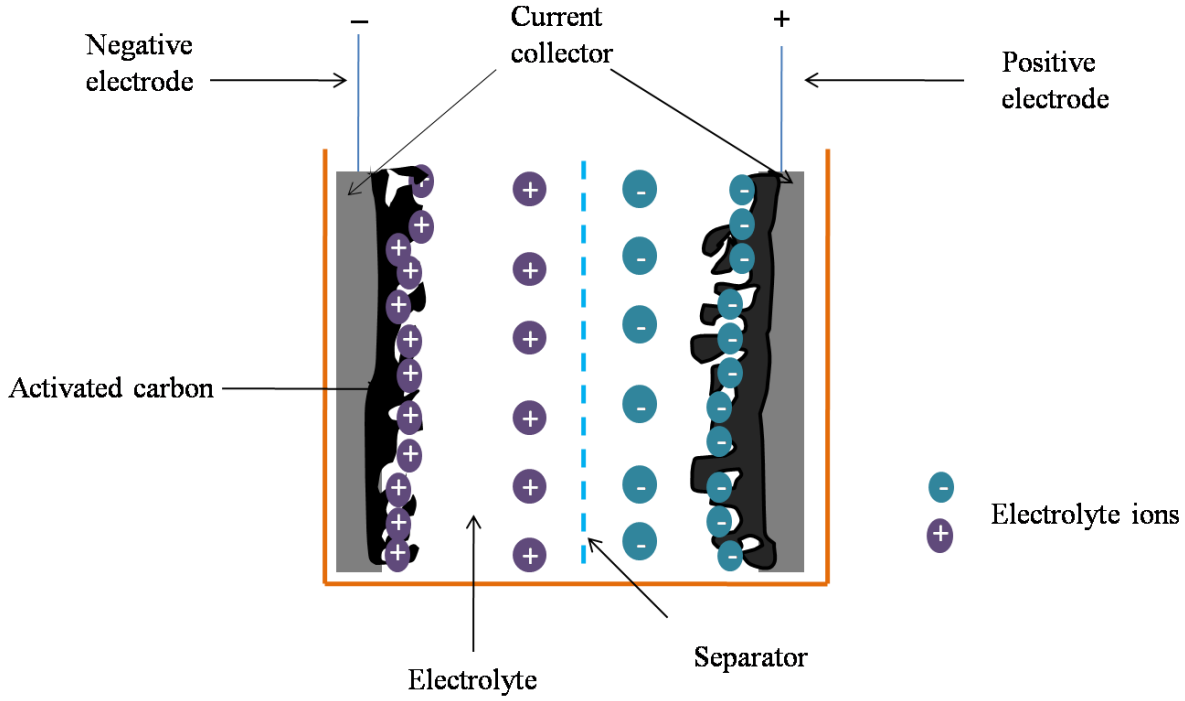

Figure 1 Schematic of an EDLC type supercapacitor

1.6.2 Pseudocapacitor

Pseudocapactor is the of type super capacitor which is intermediate between the battery and EDLC type supercapacitors. It consists of two redox-active materials and electrolyte. Due to use of redox reactive electrode materials, it has lesser life cycle as compared to EDLC (Mohapatra, Acharya, & Roy, 2012). It stores the energy by faradic process in which transfer of charge between electrode and electrolyte occurs. Reversible redox reaction is occurred on the electrode when the external potential is applied involving the transfer of charges between electrolyte and electrode (S.-M. Chen, Ramachandran, Mani, & Saraswathi, 2014). The energy density is higher than EDLC but they have lesser life cycle. Materials like conducting polymers, metal oxides are used as the electrode materials in pseudocapacitors, but they have less stability due to the redox reaction involved in the energy storage mechanism (Beidaghi & Wang, 2012).

1.6.3 Hybrid capacitor

Hybrid capacitor combines the nature of EDLC which has high power density and pseudocapacitor which has higher energy density (Vlad et al., 2014). The combination of power source electrode similar to electrode material in battery and energy source from the electrode materials similar to that battery capacitor makes the hybrid super

capacitor intermediate supercapacitor between pseudo capacitor and EDCL (Burke, Liu, & Zhao, 2014). Different combinations of electrodes are tested to improve the energy and power densities. The application of the composite electrode improves the energy density but the cyclic stability is reduced (Naoi & Simon, 2008). Different types of combination of hybrid capacitors are battery type, asymmetric and composite type (Burke, 2007) (Muzaffar, Ahamed, Deshmukh, & Thirumalai, 2019).

In composite type of hybrid supercapacitor, two or three different types of electrode materials are combined to form a single electrode. Generally, carbon based electrode material are combined with conducting polymers or metal oxides so that high surface area of the carbon based materials increases the intersurface contact between metal/conducting polymer based electrodes in which faradic process is involved (Mastragostino, Arbizzani, & Soavi, 2002). The metal oxides and conducting polymer improves the capacitance of the composite electrode. This type of hybrids involves both physical and chemical charge storage mechanism (Halper & Ellenbogen, 2006).

In asymmetric hybrids, conducting polymer or metal oxides are used as positive electrode and the carbon based materials are used as the negative, whereas, in battery type of hybrids, battery electrode and supercapacitor electrodes are combined to develop the hybrid properties of battery and supercapacitor within a single charge storage device (Halper & Ellenbogen, 2006).

1.6.4 Electrode materials

Electrodes in supercapacitor are the thin coatings of the material (generally, carbon in EDLC type supercapacitor) on inert electrode surface connected electrically by conductive metallic current collector. The electrode material used in the supercapacitors should be chemically and thermally stable for longer cyclic life of the supercapacitor. The electrode materials should have high porosity and high surface area for the high energy density supercapacitor (G. Wang, Zhang, & Zhang, 2012). The electrode materials also should have low internal resistance, low cost, environmentally friendly, easy availability and easy processing. Generally, carbon materials and its composite haves attracted the researches in the development of electrode materials for the suspercapacitors (G. Wang et al., 2012).

The energy storage performance and capacitive nature of the capacitance depends on the electrode material used in the fabrication of the supercapacitors. However, the

main issue in the supercapacitor is regarding the low energy density and high self discharge rate (G. Wang et al., 2012) (Iro et al., 2016). Many researches are directed towards the development of the new electrode materials to increase the energy density and reduce the energy discharge rates of supercapacitor. Various materials like AC, carbon, grapheme, conducting polymers, metal oxides are used as the electrode materials (Forouzandeh, Kumaravel, & Pillai, 2020).

1.6.5 Transition metal compounds

Transition metal compounds (TMCs) are characterized by low resistance and high specific capacitance (R. Liu et al., 2021). For this reason, transition metal compounds are useful in the fabrication of the supercapacitors with high energy and power. Various metal oxides like ruthenium dioxide (RuO_2), iridium oxide (IrO_2), manganese oxide (MnO_2), nickel oxide (NiO_2) are commonly used as the electrode materials the supercapacitors (Q. Li, Zheng, Xu, Xue, & Pang, 2018) (D.-Q. Liu, Yu, Son, & Joo, 2008). They are found to give highly reversible redox reaction, high specific capacitance, high conductivity, and longer life cycle (D.-Q. Liu et al., 2008). Milder electrolytes can be used for the metal oxide electrodes and their cost of production is also low, so the metal oxides are one of the suitable candidates for electrolyte materials. RuO_2 has fascinating properties in catalytic action, conductivity, redox-reactive nature and high chemical and thermal stability which attracts the researchers in its application in supercapacitor besides its various applications in integrated circuits, resistors and electronic appliances (C. Zhao & Zheng, 2015).

Various oxides such as CoO (Zhang, Yan, Li, Ma, & Ng, 2017), Co_3O_4 (Xu, Gao, Cao, Wang, & Chen, 2010), V_2O_5 (Shao, Jeon, & Lutkenhaus, 2012) are on investigation and widely studied to improve the stability and energy storage performance of the supercapacitor. Hydroxides like $Co(OH)_2$ (C. Zhao & Zheng, 2015)and $Ni(OH)_2$ (Fu et al., 2009) are also used as the electrode materials. Various sulfides like CuS, MoS_2 and CoS (Van Nguyen et al., 2020) are also used as electrode materials (Samdhyan, Chand, Anand, & Saini, 2022). Phosphides like Ni_2P (Y. Zhao et al., 2019), Co_2P (X. Zhang et al., 2020) are used as electrode materials (X. Li, Elshahawy, Guan, & Wang, 2017). TMCs have high capacitance due to charge storage mechanism by faradic process but they have low cyclic stability (R. Liu et al., 2021).

1.6.6 Conducting polymers

Conduction polymers are low cost and easy production compounds. So, many researches are focused on the conducting polymers for its application as supercapacitor electrode materials. Conducting polymers have high conductivity but comparable equivalent series resistance to the carbon based electrode. However, the redox process involved in the charge storage mechanism of the conducting polymer based supercapacitor causes the low stability of the supercapacitor cycle (Mastragostino et al., 2002). Many conducting polymers are utilized as the electrode materials for the charge storage mechanisms. They include polyaniline (PANI) (Eftekhari, Li, & Yang, 2017), polypyrrole (PPy) (Fan & Maier, 2006), polythiophene (PTh) (L. Li, Meng, Zhang, Liu, & Zhang, 2022). Amongst them, PANI is widely used due to its low cost, high conductivity, easy processing and high energy density (Cheng, Tang, Shinya, & Qin, 2013). But, it has low cyclic stability. Investigations shows that PANI combined with carbon materials improved the stability and charge storage of supercapacitor (Eftekhari et al., 2017).

1.6.7 Carbon nanomaterials

In the past, carbon materials like AC were widely as adsorbent materials. But, due to attractive properties like stability, high porosity and availability, easy processing, light weight, environment friendly and low cost have increased its application in the supercapacitors. Different carbon materials that are used as electrode material include AC (Kubozono et al., 2016), grapheme (Szczęśniak, Durajski, & Szczęśniak, 2014), carbon nanotubes (Cleuziou, Wernsdorfer, Bouchiat, Ondarçuhu, & Monthioux, 2006)and carbon aerogels (Azwar et al., 2018; Bokhari et al., 2020; Reynolds, Fung, Wang, Dresselhaus, & Pekala, 1995; Z. Yang et al., 2019). Generally they are as used as electrode materials in EDLC type of supercapacitor but they have low energy density (Kubozono et al., 2016) . Carbon materials are combined with electrode materials like metal oxides and conducting polymer to improve the energy storage capacity of the supercapacitors (Kar, 2020).

1.7 Statement of problem

Energy storage is the main focus of today's world and of the scientific community. Due to depletion of sources of fossil fuels, increase of population and the energy storage issues in the electrostatic field, a very high power density fast charging

supercapacitor has emerged as the best potentials in the 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 are on investigations for the applications in the fabrication of supercapacitor and solid state hydrogen stores. However, the main challenges with the AC based electrode materials are its low energy density and very high self discharge rate. To improve the capacitive performance of the supercapacitor, different alternative electrode materials based on the carbon rich precursors are widely studied (Sundriyal et al., 2021). Due to many attractive properties of the AC, the focus is on the development of the new carbon based nanocomposite electrodes for the supercapacitors.

Different carbon rich precursors like agro-waste, nuts, peels, husks, bagasse, etc are gone waste and are causing the environment pollution despite the agro-waste based AC having desirable properties for energy storage and purification. So, the locally available agro-based waste can be best utilized to prepare AC for the application as the adsorbents and electrode materials. Its application as the electrode materials may reduce the consumption of the fossil fuels and carbon emissions. For this reason, preparation of the AC from agro-waste carbon rich precursors could be a very fruitful to environment pollution control in Nepal and around the globe. Studies regarding modifications and development of the carbon based electrode materials to improve its capacitive performance could be one of the leading research fields for the fulfillment of the future energy demand and carbon emission control.

1.8 Objectives

The general objective and specific objectives for the proposed research are as follows:

General objective

To prepare waste betel nut based activated carbon and test its electrochemical performance for energy storage.

Specific objectives

i. To prepare waste betel nut based activated carbon.
ii. To characterize the as-prepared activated carbon by iodine number, Methylene blue number, Fourier transform infrared (FTIR) spectroscopy, X-ray

diffraction (XRD), Scanning electron microscopy (SEM) and Raman spectroscopy.

iii. To test electrochemical performance of activated carbon.

1.9 Limitations

i. Preparation of the AC was only limited to chemical method of preparation with H_3PO_4 as an activating agent, carbonization temperature of 500 °C and 1:1 mixing ratio of betel nut powder and H_3PO_4.

ii. Surface morphology of as prepared AC could not be visualize properly due to limitation of high resolution Field emission scanning electron microscope (FESEM)

iii. Brunauer-Emmet-Treller (BET) and Barrett-Joyner-Halenda (BJH) give specific surface area, pore size and pore volume. These data can help for proper explaining the electrochemical charge storage. This could not be done.

iv. AC couldn't be prepared at high temperature due to working limitations of furnace.

CHAPTER TWO: LITERATURE REVIEW

2.1 Overview

Activated carbon (AC) has been widely used as the adsorbent and electrode materials from the very first (Xie & Zhou, 2019). Its' useful absorption property was identified since the ancient time. Charcoals were used for the water purification in the past in the ancient hindu society (Bansal & Goyal, 2005). In the recent past, the focus is on the utilization of the carbon based AC as the electrode materials in high power density supercapacitors (Miller, Hua, & Tezel, 2018). Different agro-waste like lapsi seed stone (Sahira, Mandira, Prasad, & Ram, 2013) sugarcane baggase (Qureshi, Bhatti, Kazi, & Ansari, 2008), orange peels (Foo & Hameed, 2012), etc have been prepared at various carbonization temperature and impregnation ratio and there is increasing focus and interest on utilization of the bio-waste based AC for the energy storage application (Mensah-Darkwa et al., 2019). Several types of electrode materials have been tried so far but the most common electrode materials used in the EDLC type supercapacitors are carbon based electrode and organic electrolyte due to its easy fabrication (Mensah-Darkwa et al., 2019). ACs from different bio-waste precursors like almond shell (González, Román, Encinar, & Martínez, 2009), apricot stone (Özçimen & Ersoy-Meriçboyu, 2010), bagasse, coconut shell, jute, peanut hull tea, walnut shell etc. have been prepared either by physical or chemical activation methods (Zequine et al., 2017) (Mensah-Darkwa et al., 2019).

Preparation of AC using waste betel nut as the precursor by using different activating agents like H_3PO_4, $ZnCl_2$, NaOH (Bardhan et al., 2020), HCl etc. at different carbonization temperature and impregnation ratio have been carried by researcherszz. H_3PO_4 treated AC have found to have better porosity (Bardhan et al., 2020; Qureshi et al., 2008) as compared to other activating agent. The porous structure is very desirable for the purification. According to research carried by (Sahira et al., 2013), betel nut based activated carbon treated with H_3PO_4 in the ratio 1:1 by weight with betel nut powder and carbonized at 400 ° C was found to highly porous with presence of mesopore and micropores on the surface. The result also showed that betel nut based AC was graphitic in nature which is suitable for EDLC type supercapacitor electrode (Najib & Erdem, 2019). The experimental results indicate the potential use of waste betel as a precursor material for the preparation of high surface area nanoporous AC

for its application as the electrode materials EDLC type supercapacitor(Rahmah et al., 2017) .

In a research conducted by Shrestha et al., (2012) activated carbon was prepared from Lapsi seed stone by chemical activation with phosphoric acid at 400 °C. pH of point of zero charge (pHpzc), iodine number, proximate analysis and concentration of surface oxygen functional groups of activated carbon was determined by Boehm titration. The adsorption of methylene blue by thus prepared AC was analyzed by the Langmuir and Freundlich adsorption isotherms. The data fitted well to the Langmuir isotherm with monolayer adsorption capacity 277 mg g^{-1}. Thermogravimetric analysis and proximate analysis of Lapsi seed stone were also carried out. The analysis showed that the activated carbon derived from Lapsi seed stone activated with phosphoric acid was comparable with commercial activated carbon and could be used as a potential adsorbent and can also be utilized for EDLC type supercapacitor.

In the similar research conducted by (Joshi et al., 2015) AC was prepared from Lapsi seed stone by chemical activation with ptassium hydroxide at 400 °C. The AC was characterized by pH, moisture content, Fourier transform-infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), iodine number (I_N), methylen blue (MB_N), FTIR spectra indicated the presence of various oxygen containing functional groups on the surface of AC. SEM images showed the highly porous characteristics of AC with full of cavities. The Iodine number of AC revealed that the AC was found to be highly micro-porous. Also, research carried by (Joshi, 2017) showed the comparison between betel nut based AC and Lapsi seed stone based AC. Both the precursors were treated with H_3PO_4 in the impregnation ratio and 1:1 and chemically activated at 400 °C at 3h in nitrogen environment. Betel nut based AC was found to have high porosity as determined by iodine number, methylene blue and surface area.

The bio-waste based ACs are commonly used as electrode materials in EDLC type of supercapacitor with organic electrolyte. Research by (Jain & Tripathi, 2014) , prepared coconut shell based AC by chemical activation treating with KOH and tested its performance by using the resultant AC as the electrode material in EDLC type supercapator. The power density and energy density were respectively determined to be 1.6 Kw kg^{-1} and 88.8 Wh kg^{-1}. Another research which was carried by (Du et al.,

2013), prepared porous carbon using porous starch as the precursor and carbonized and activated by treating with KOH. The BET surface area was estimated to be 3251 $m^2 g^{-1}$. The electrochemical test were performed and result showed that the specific capacitances at current densities of 0.05 A g^{-1} and 180 A g^{-1} respectively 304 F g^{-1} and 197 $^{-1}$ F g^{-1} exhibiting retention of 98 % over 100 cycles in 6 M KOH.

In another research performed by (Qu, Xu, Lu, Zhang, & Li, 2015), porous carbon was prepared from corncob residue at 850 °C and further acid washing The BET surface area of the porous carbon was obtained to be 1210 $m^2 g^{-1}$ with percentage yield of 23.2 % by weight. Further, the obtained porous carbon was used to fabricate the working electrode supercapacitor and tested which showed that the capacitance at scan rate 5 mV s^{-1} and capacitance retention was 82 %. Various researches conducted by the researchers showed that the surface area and porosity of the AC are mainly affected by the precursors used, carbonization temperature and impregnation ratio. And, the capacitance of the supercapacitor depends on the electrode materials (Barbieri, Hahn, Herzog, & Kötz, 2005).

2.2 Research gap

Energy storage is one of major focus of the global scientific community due to the rapid increasing energy consumption demand of the world. Many researches on the energy storage device like battery, fuel cells and supercapacitors and electrode materials are on going on. But, in context of Nepal, many researches based on the synthesis of ACs were already performed. But, very few researches regarding energy storage application have been done so far. The locally available bio-waste products can be utilized for the preparation of the AC and AC based electrode materials can be fabricated for the application in the supercapacitos. This could be one the attracting field of researches in Nepal for the solution of energy demand problem and environment pollution control.

2.3 Methods of activation of carbon

Generally, the activation of a carbonaceous precursor can be performed through physical (steam, air or CO_2) or chemical activation (activators such as $ZnCl_2$, KOH, etc.) or a combination of both (Namasivayam & Kadirvelu, 1997). The chemical activation method is low temperature and faster method when compared to physical method of carbon preparation. Also, the ACs prepared by chemical method of

activation also have high surface area, high porosity and gives high percentage carbon yield (Barbieri et al., 2005). So, chemical method of activation is preferred over physical method of activation for the production low cost activated carbon

2.3.1 Physical method of activation

Physical activation is two-step carbon activation process, namely carbonization of the carbonaceous precursor in an inert atmosphere followed by subsequent activation of the resulting char in the presence of carbon gasification reactants (gaseous) such as carbon dioxide, steam or air or a suitable combination of the above mentioned gaseous activating agents (Joshi & Homagai, 2017). In the method of physical activation, the reaction involved is between carbon atom and the oxidizing gas. Some parts of the char structure are more reactive than the other parts. This reason of the development of the porous structure in the physical activation method (Heidarinejad et al., 2020). The physical method of activation utilize gaseous activating agent which doesn't produce waste water. So, physical method of activation is environmentally friendly. However, it time consuming method of AC preparation and consume more energy (Kwiatkowski & Broniek, 2017).

Also another inherent drawback of this method is that large amount of internal carbon mass is eliminated to obtain well developed pore structure. And thus one has to satisfy himself with limited carbon yields if one were to go in this route (T. Yang & Lua, 2003).

2.3.2 Chemical method of activation

On the other hand, chemical activation is a single step process of the AC preparation where carbonization of organic precursor is done by treating with chemical activating agents (Viswanathan, Neel, & Varadarajan, 2009). In this method, the activating agents employed for treating with precursor are solid containing alkali and alkaline earth metal elements which highly influence the surface area and porosity of the AC (Togibasa, Mumfaijah, Allo, Dahlan, & Ansanay, 2021). The activating agents in this method are responsible for dehydrating and enhancing carbon percentage yield (Shamsuddin, Yusoff, & Sulaiman, 2016) (Ahmadpour & Do, 1997). The temperatures used in chemical activation are lower than that used in the physical activation process (Heidarinejad et al., 2020). As a result the development of a porous structure is better in the case of chemical activation method. But, the product obtained

need treating and repeated washing for the neutralization and removal of organic impurities which cause serious pollution (Ahmadpour & Do, 1997) (González-García, 2018).

2.3.2.1 Activating agents in the methods of chemical activation

Chemical methods of activation utilize the micro-explosion behaviour of chemical agents. Carbon nanomaterials have very fascinating electrical and structural properties which make them very attractive and indispensable in energy conversion and energy storage devices like supercapactors (as electrode material), catalytic processes (as support material) and purification technologies (sorbents). One of the main factors limiting their applicability is their low specific surface area (in the range of 200 – 300 $m^2\ g^{-1}$) (C.-M. Wang et al., 2015). Many ways to increase the surface area of the carbon nano materials are on investigation (T. Chen & Dai, 2013) .

In chemical activation processes, well-known chemical agents, i.e., $ZnCl_2$, HPO_4, H_2SO_4, K_2S, KCNS, HNO_3, H_2O_2, $KMnO_4$, $(NH_4)_2S_2O_8$, NaOH, KOH and K_2CO_3 are used to activate carbons, resulting in a high surface area and appropriate porous structure (Yahya et al.2015) (Gao, Yue, Gao, & Li, 2020).

2.3.2.2 KOH as activating agent

Among the alkali metal salts, KOH is the most effective activating agent in producing activated carbon materials.

Besides the quality of porosity and surface area, the toxicity and the effects of the activating agents on the environment and human health are also taken into consideration. The boiling point of KOH is 1327 °C which is generally greater than the activation temperature during chemical activation. So, some of the KOH are not vaporize (Hui & Zaini, 2015). These are then expected to released to the environment when washed to neutralize the activated carbon (Hui & Zaini, 2015).

Potassium hydroxide (KOH) has been widely used as an activator for preparing activated carbon. But, the carbon percentage of the KOH based activated carbon is low also due to highly micropores, it may not be suitable for the removal of micro-pollutants in the water and air. Its effect on human health and environment and its toxicity are the concerns of the KOH activating agent (Hui & Zaini, 2015). Such drawbacks should be taken into account in the activated carbon preparation. Also, it is

of utmost importance to establish alternative activators in the synthesis of excellent activated carbon (Dıaz-Terán, Nevskaia, Fierro, López-Peinado, & Jerez, 2003) (Mohanty, Das, & Biswas, 2006).

2.3.2.3 H_3PO_4 as activating agent

Phosphoric acid (H_3PO_4) is an activating agent to open the pores and enlarge the surface area of activated carbon so that the adsorption power will increase (Mohanty et al., 2006). In this study, H_3PO_4 based activated carbon was prepared from the waste betel nut. During carbonization process of waste betel nut impurities like ketones, alcohols, acids, aldehydes are removed (Joshi & Homagai, 2017; Safitri, Pangestika, Fauziah, Wahyuningrum, & Astuti, 2017) Moreover, H_3PO_4 form phosphate bonds which connects the carbon layer structure that enlarges the pore size and increase the surface area of the AC (Safitri et al., 2017) (Mohanty et al., 2006) .

2.3.2.4 $ZnCl_2$ as activating agent

Zinc chloride is a chemical compounds having formula $ZnCl_2$ and its hydrates. The aim of this study was to optimize the chemical activation process using $ZnCl_2$ in order to produce highly mesoporous activated carbon with a pore size distribution suitable for use as a catalyst support and adsorption of larger organic molecules (Mohanty et al., 2006). The effect of the activation temperature and different impregnation ratios of precursor and $ZnCl_2$ on the specific surface area, pore size distribution, yield, total carbon (TC), and adsorption abilities of the activated carbon will be investigated (Nayak, Bhushan, Gupta, & Sharma, 2017) (Mohanty et al., 2006).

2.3.3 Impregnation ratio

The term “impregnation ratio” describes the ratio of mass of the activating agents to the dry mass of the precursor (Yahya et al., 2018b). It is true that, more than temperature or any other experimental parameter, the porous texture obtained after heat treatment of the impregnated material depends mainly on the amount of activating agent per unit weight of precursor (Mohanty et al., 2006). In other words, impregnation ratio has a major effect. A small increase in the ratio causes an increase in the total pore volume of the AC thereby increasing in the surface area. However, high ratios lead to a reduction in pore volume and surface area due to a collapse of micropores caused by weak pore walls after intensive dehydration (Mohanty et al., 2006).

2.3.4 Carbonization time and temperature

Generally, increase of the carbonization temperature would increase surface area and decrease yield of AC productions due to the volatilization process. Minimum carbonization temperature of 400 °C is required for the transformation of the lingocellosic precursor into grapheme structure (Daud, Ali, & Sulaiman, 2000).

The duration of carbonization/carbonization time should be enough to allow the evolution of volatiles from the precursor in order to develop of porosity in AC. However, too long carbonization time brings about the enlargement of the pores, causing a decrease in the surface area. This parameter must be optimized in order to obtain the maximum porosity development, to maximize the yield and to reduce the fuel/energy consumption (Üner & Bayrak, 2018).

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CHAPTER THREE: RESEARCH METHODOLOGY

3.1 Methodology

This work was focused the experimental tests and analysis to validate the quality of the sample fabricated in our laboratory. To complete the research work, background research were done and problem regarding the work were stated. Identification of the methods of preparation of the sample and previous and current works related to this research work were identified through literature review. Then, the waste betel nut were collected and waste betel nut based AC was prepared in the laboratory. The characterization of the resultant AC and validated by comparing its values with commercial AC. After validation, electrode was based on waste betel nut based AC was fabricated and its electrochemical tests were performed. The results were analysis and discussed.

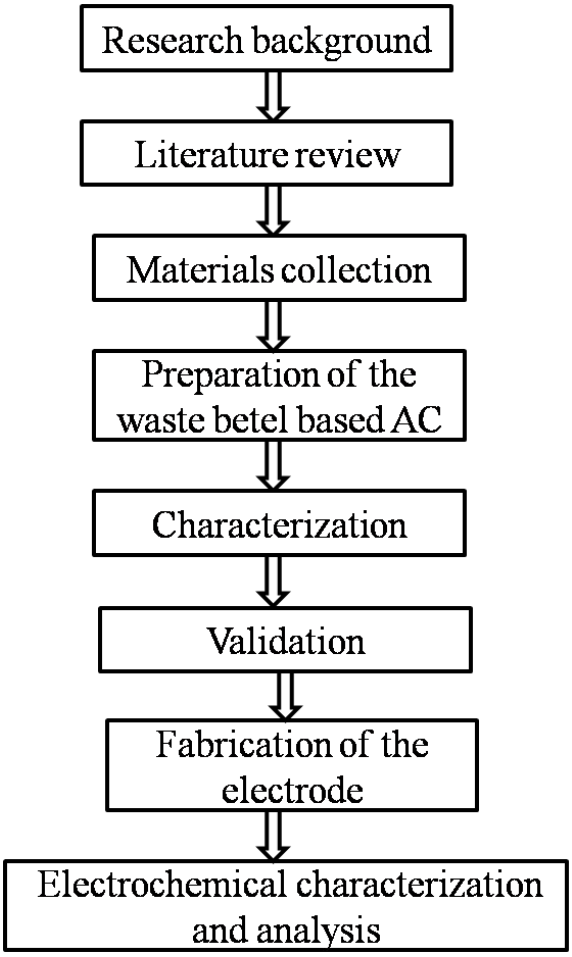

Figure 2 Flow chart of the methodology

3.2 Preparation of waste betel nut based AC and working electrode ;

1. Powder preparation
2. Activation
3. Carbonization
4. Washing
5. Resultant AC
6. Grinding by polyvinylin difluoride (PVDF), acetylene black grinded and N-methyl-2-pyrrolidone (NMP)
7. Coating on nickel foam
8. Pressing
9. Resultant carbon based working electrode

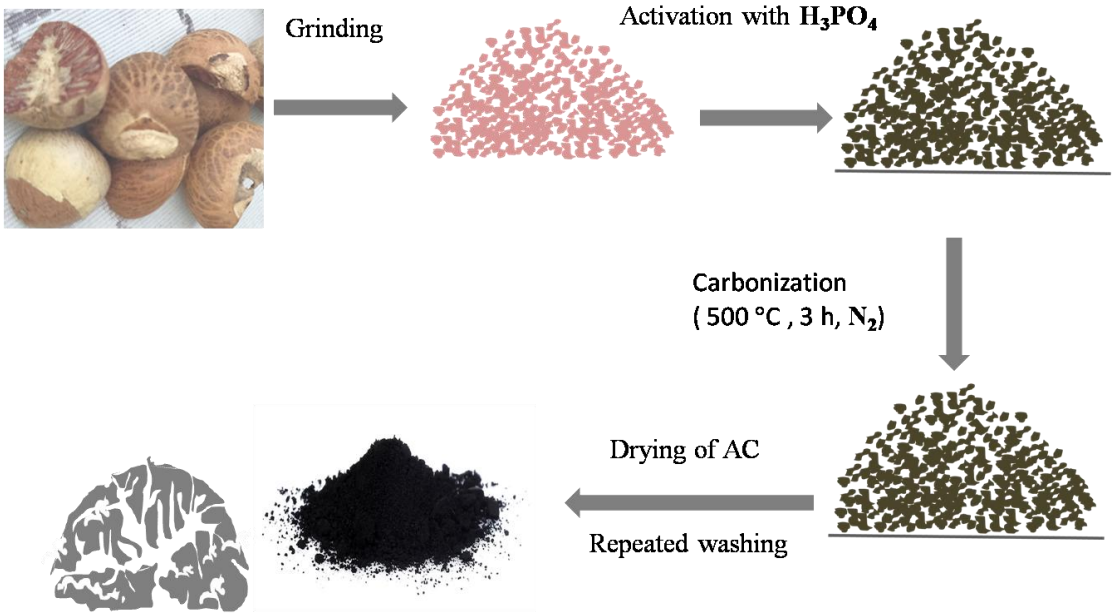

Figure 3 Schematic for preparation of activated carbon

3.3.1 Powder preparation

Waste betel nuts were purchased from local market, Kalimati, Kathmandu. It was washed repeatedly with tap water and then with ethyl alocohol and finally with distilled water several times. They were dried in oven for about 24 h 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.

3.3.2 Activation with H_3PO_4 as the activating agent

With reference to literature review, the waste betel nut based ACs prepared with H_3PO_4 as an activating agent was found to have presence of large amount of micropores and mesopores, so, H_3PO_4 was selected as the activating agent.

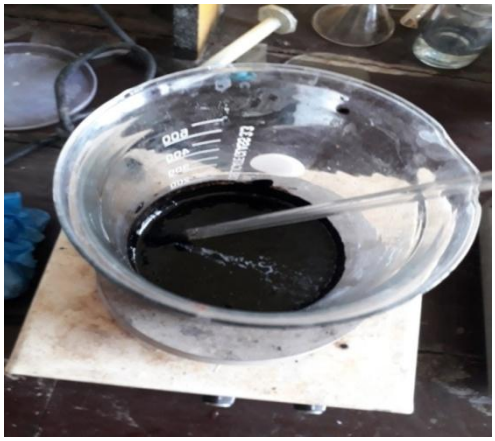

Figure 4 Chemical activation process

After the powder preparation, 20 g 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.

3.3.3 Carbonization

Carbonization is the process of converting carbon- rich material to pure carbon through pyrolysis process. For carbonization the dried sample was taken out from the oven and grinded gently on crucible furnace. Then, the sample was transferred into in quartz tube. The tube with the mixture was placed inside an electric horizontal tube furnace and the carbonization of the AC was carried out in an inert environment by continuous flow of nitrogen gas at the rate of 75 ml min^{-1} maintaining temperature of furnace at 500 °C for 3 h. After 3 h, the sample allowed to cool and taken out and kept at oven maintaining the temperature of 100 °C for 24 h.

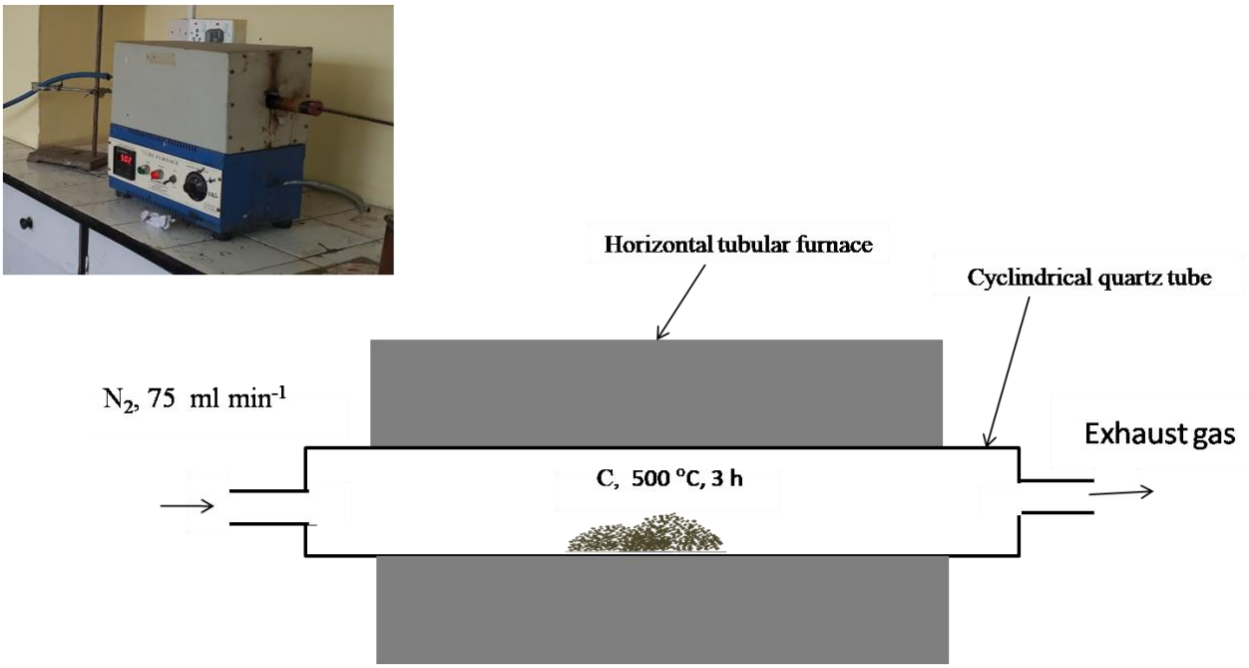

Figure 5 Schematic of carbonization process in tube furnace

3.3.4 Washing and drying

The carbonized sample was washed to remove the impurities and neutralize the sample. The prepared AC was washed with 1% $NaHCO_3$, repeatedly washed with warm water until it became neutral. In this process, distilled water was heated to warm inside the beaker then the warm water was poured to the beaker where the carbonized sample was kept and stirred with magnet on magnetic stirrer for about 30 minutes for each time. Then, the stirred sample was kept still for the sedimentation of the product to ease the filtration process. The process was carried out repeatedly until the pH value came to around neutral point. The AC sample was dried in an air oven at 110 ^{o}C for 24 h and sieved to get the particle of a size 106 µm.

3.4 Preparation of electrode

To test the electrochemical performance of as prepared AC for supercapacitor, working electrode was prepared. For this, nickel foam was kept in 5% HCl in a clean beaker and sonicated for 10 minutes. It was washed with distilled water repeatedly and dried in oven for about 6 h. 4 g of AC was 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 was deposited on 1×1 cm^2 clean nickel foam using micropippete. It was dried in oven at 80 °C for 6 h.

As prepared AC carbon based electrode was used as working electrode. Plantinum (Pt) wire was used as counter electrode and Ag/AgCl was used as reference electrode. 3 M aqueous KOH was used as electrolyte. The test was carried out in versa Stat electrochemical work station.

Carbon materials generally show EDLC type capacitance. Specific capacitance of EDLC type material was calculated by using the equation (I)(Mukhiya et al., 2019):

$$C = \frac{I \times t}{m \times V} \ldots\ldots\ldots\ldots\ldots\ldots\ldots (I)$$

where, C is specific capacitance (F g^{-1}),

I is current (A)

t is discharge time (s)

m is the mass of the active electrode material (AC)

V is voltage difference (V).

3.5 Materials characterization

FTIR spectra of the resultant C was recorded on the Thermo Electro Corporation, Nicolet 4700 Japan at room temperature. Spectra were recorded at 25 °C in the transmission mode over 500-4000 cm^{-1} range.

The XRD pattern of the resultant AC was recorded on the Rigaku X-ray diffractormeter, RINT, Japan. The instrument was operated at 40 kV and 40 mV with Cu-Ka radiation at room temperature.

SEM image was recorded at 10 kV on U-800 Hitachi Co. Ltd scanning electron Microscope Japan.

Raman scattering of the resultant AC was taken using Raman spectrometer Jobin-Yvon T6400, Japan. AC samples were spread over clean glass substrate. The AC sample was excited using green laser; 514.5 nm and 0.5 mW power.

The working electrode fabricated from resultant AC was tested using Gamry electrochemical workstation at 3 M aqueous KOH electrolyte in which Ag/AgCl was reference electrode, platinum wire was counter electrode and the fabricated electrode was working electrode.

CHAPTER FOUR: RESULTS AND DISCUSSION

4.1 Physicochemical test

For the confirmation of the removal of the impurities, pH test was carried out with the universal indicator for all the AC which was activated with H_3PO_4. After neutral indication was observed from the universal indication, the pH value was checked in the pH meter .The pH value H_3PO_4 activated carbon and char were, 7.2, 7.3 ,7.1& 7.1 respectively which was in the neutral range (7 to 7.5). Therefore, the samples of activated carbon were concluded to be neutral.

AC was characterized by using the iodine number test (I_N), methylene blue (MB_N) test and surface area. The MB_N, I_N and surface area of the resultant AC was determined to be 365 mg g^{-1},882 mg g^{-1} and 927 m^2 g^{-1}. T blue was determined using ASTM D4607-94 method (Gao et al., 2020). The methylene blue was determined by single point adsoption isotherm (Dollimore & Spooner, 1974). The surface area was estimated with the methylene blue and iodine number by multiple regression (Nunes & Guerreiro, 2011)

Scanning electron microscopy (SEM) image is used to observe the surface topography and morphology of AC which could also characterize the nature of the pores present in the AC. Figure 6 is the SEM image of the waste betel nut based activated carbon carbonized at 500 °C which showed that the highly developed micropores are on present on the surface of the activated carbon with mesopores on the surface of the AC (Achaw, 2012) (Achaw, 2012).

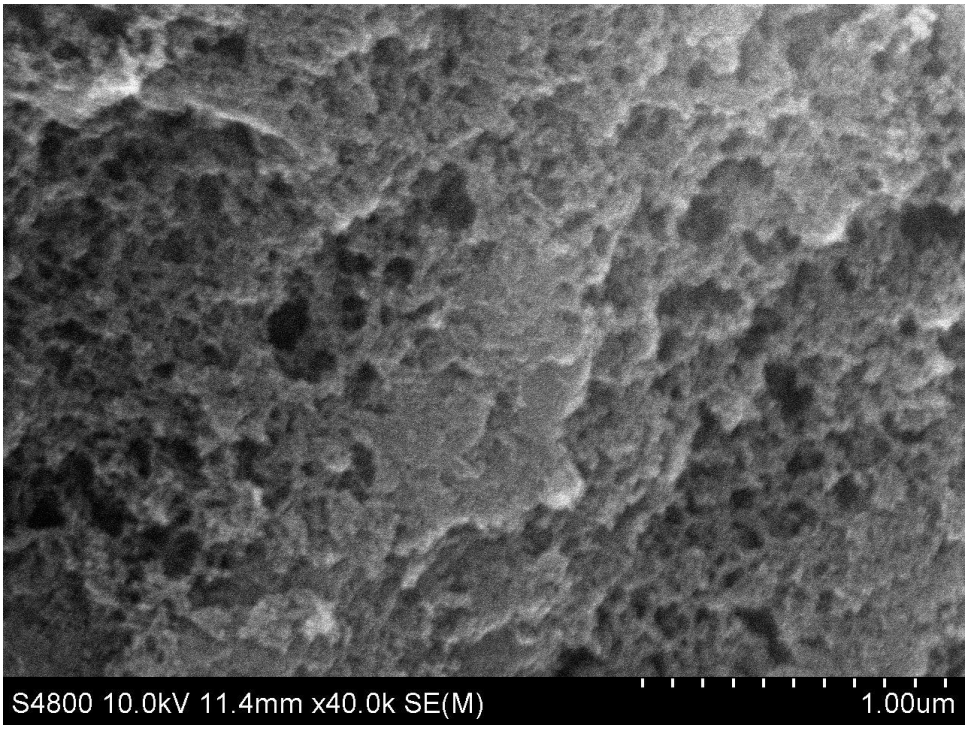

Figure 6 SEM image of waste betel nut based activated carbon

XRD is the basic technique for crystallography which helps to determine the structure of the material. The XRD of waste betel based AC in figure 7 shows broad peak at 2θ of 24.3° which corresponds to the (002) plane of carbon. Another weak peak at 2θ of about 43.8° was observed which corresponds to the (100) plane of carbon. These planes are the planes of graphitic cluster. This suggests the graphitic nature of carbon. Graphitic carbon is conductive and very useful for its application as energy storage material (Mukhiya et al., 2020; Wibawa, Nur, Asy'ari, & Nur, 2020).

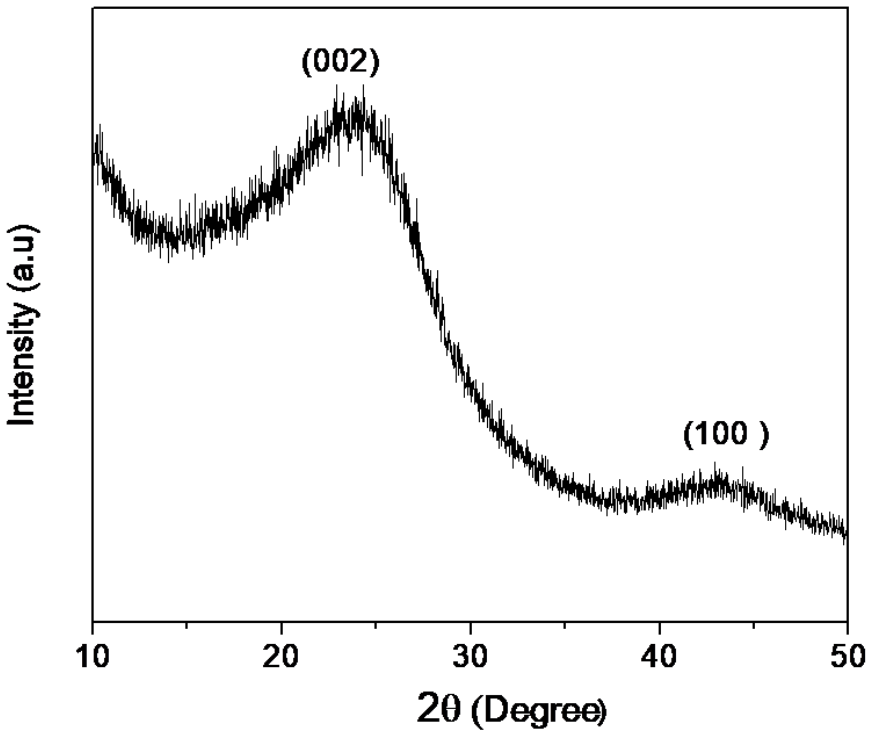

Figure 7 XRD pattern of betel nut based activated carbon

Raman spectrocopy was performed to identify and compare structure as determined by the XRD analysis. The Raman spectra pattern of the resultant AC sample as seen in figure 8 shows D band and G band at 1348.9 cm^{-1} and 1588.9 cm^{-1} respectively. 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 (I_D/I_G) was calculated to be 0.84 which shows the resultant AC is graphitic in nature (Amaral et al., 2017) (Roh, 2008) (Afseth, Segtnan, & Wold, 2006).

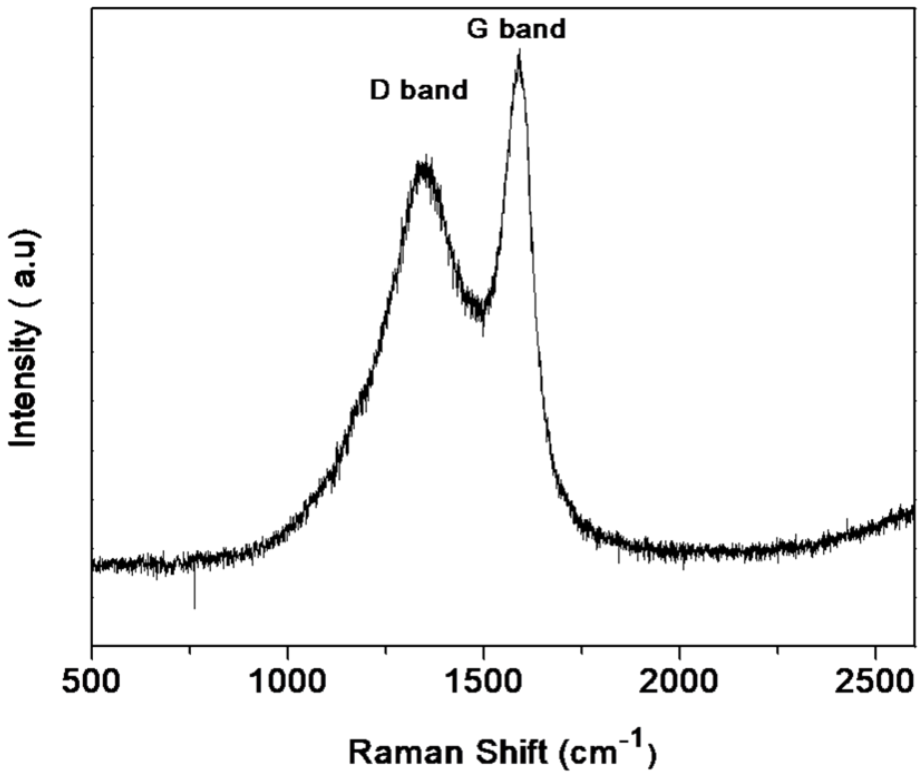

Figure 8 Raman spectroscopy pattern of waste betel nut based activated carbon

The FTIR pattern of the resultant AC is shown in figure 9 which shows different absorption peaks at different wave number range. The sample shows that the first broad absorption peak was observed at 3422 cm^{-1} due to stretching mode of hydrogen-bonded OH functional groups of carboxylic acid, phenols and alcohol. This represents the presence of water absorbing -OH functional groups. Another peaks were observed at 3034 cm^{-1}which represents the presence of C=C unsaturated alkenes. Another absorption peak at 1707 cm^{-1} represents the presence of C=O of carboxyls, aldehydes, ketones, and lactones. Another peaks ranging from 1585cm^{-1}-1434 cm^{-1} represents the presence of C=C bond in aromatic rings of lignin structure. Also, the absorption peaks as 1244 cm^{-1} represents the presence of C-O bonds of phenols, lactones and ethers. Other weak bands ranging from 883 cm^{-1} to 723 cm^{-1} represent the out-of-plane deformation in aromatic rings. So, it was found theat sample showed the presence of different functional groups like -OH, CO, COOH (Cuhadaroglu & Uygun, 2008) (Wazir, Haq, Manan, & Khan, 2022).

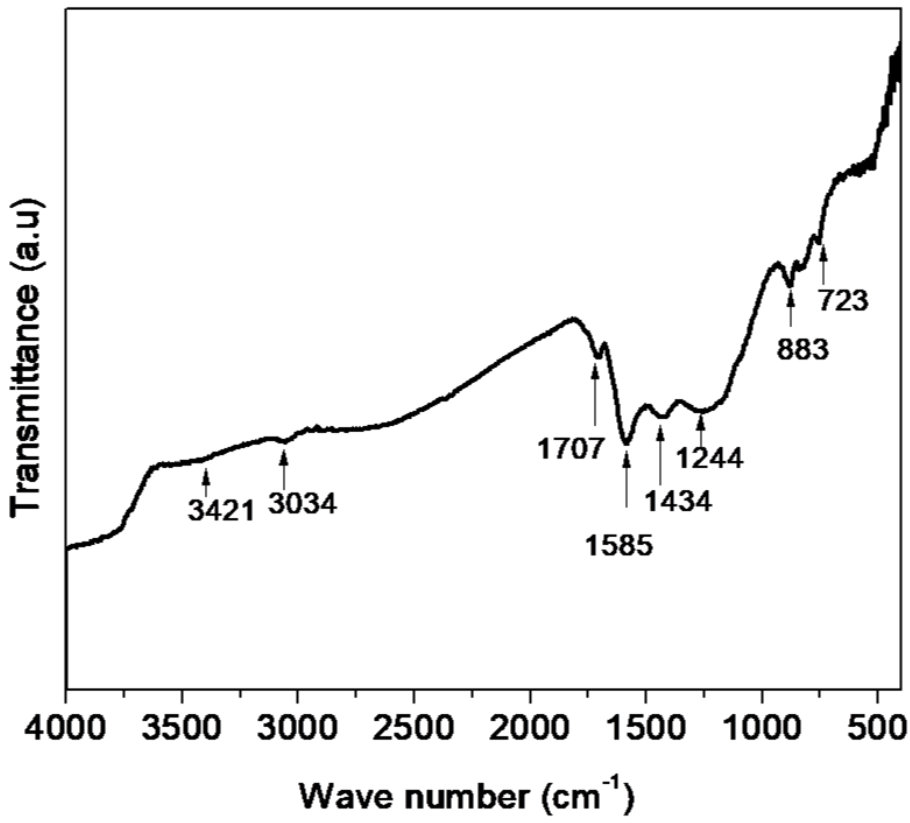

Figure 9 FTIR pattern of betel nut based activated carbon

4.2 Electrochemical test

To test the electrochemical performance of as prepared AC for supercapacitor, as prepared AC deposited on nickel foam was used as working electrode. Plantinum (Pt) wire was used as counter electrode and Ag/AgCl was used as reference electrode. 3 M aqueous KOH was used as electrolyte. The test was carried out in electrochemical work station. Cyclic voltametry (CV) was carried out 0.0-1V, at different scan rates (Wazir et al., 2022) .

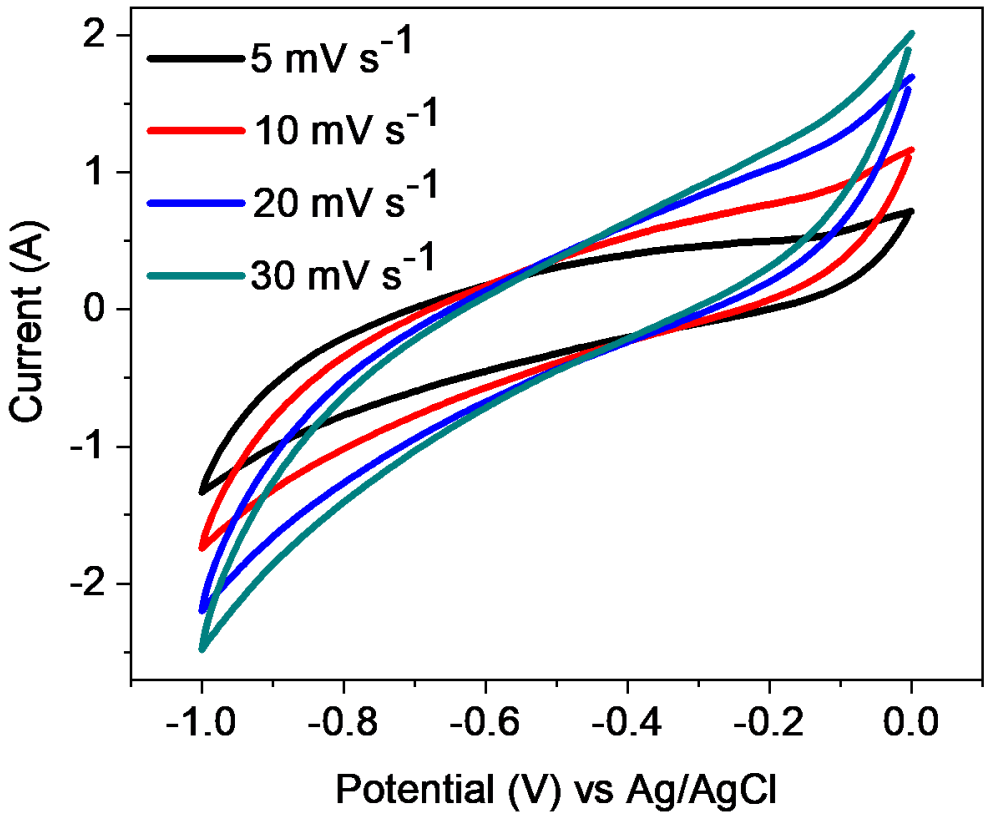

Figure 10 CV curves of the electrode fabricated from waste betel nut based AC

The nature of CV curve (Figure 10) shows that the charge storage in the as prepared AC is electrical double layer capacitor (EDLC) type. The high surface area and high porosity of as prepared AC offers effective surface for the formation of electrical double layer. The current increases with increase in scan rate.

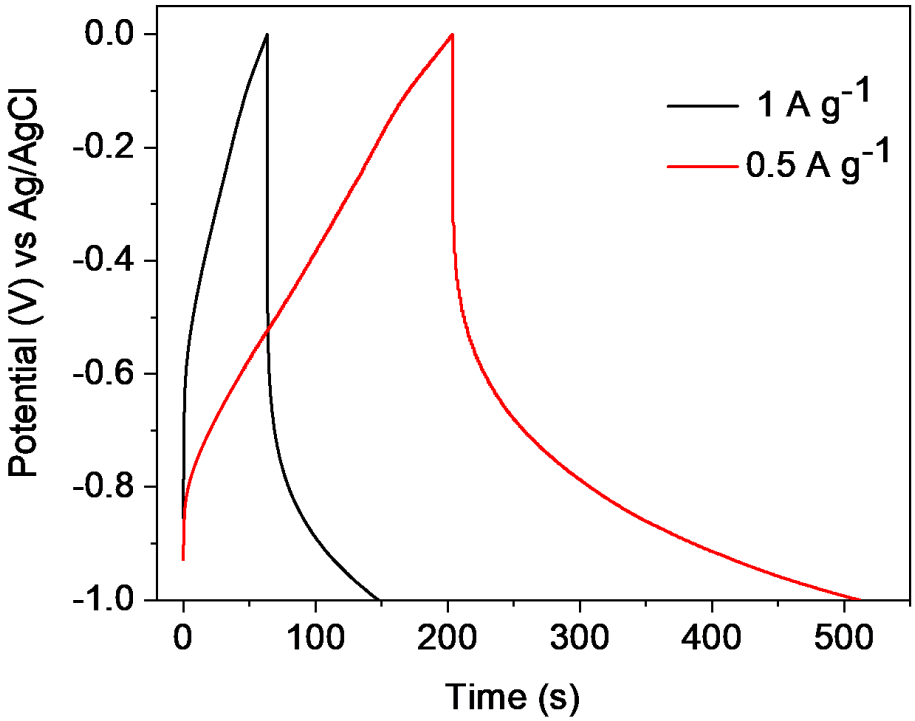

Figure 11 GCD curves of the electrode fabricated from waste betel nut based AC

Galvanostatic charge discharge (GCD) test was carried out in 0 to -1 V at different current densities of 0.5 A g^{-1} and 1 A g^{-1}. The nature of the GCD curve (Figure 11) is in agreement with their CV curves. This supports the charge storage in the as prepared AC is EDLC type. The material shows a specific capacitance of 86 F g^{-1} at current density of 1 A g^{-1}. At a current density of 0.5 A g^{-1}, the specific capacitance increases to 154 F g^{-1}. This material show 44.15 % capacitance retention when energy density is doubled. (Du et al., 2013).

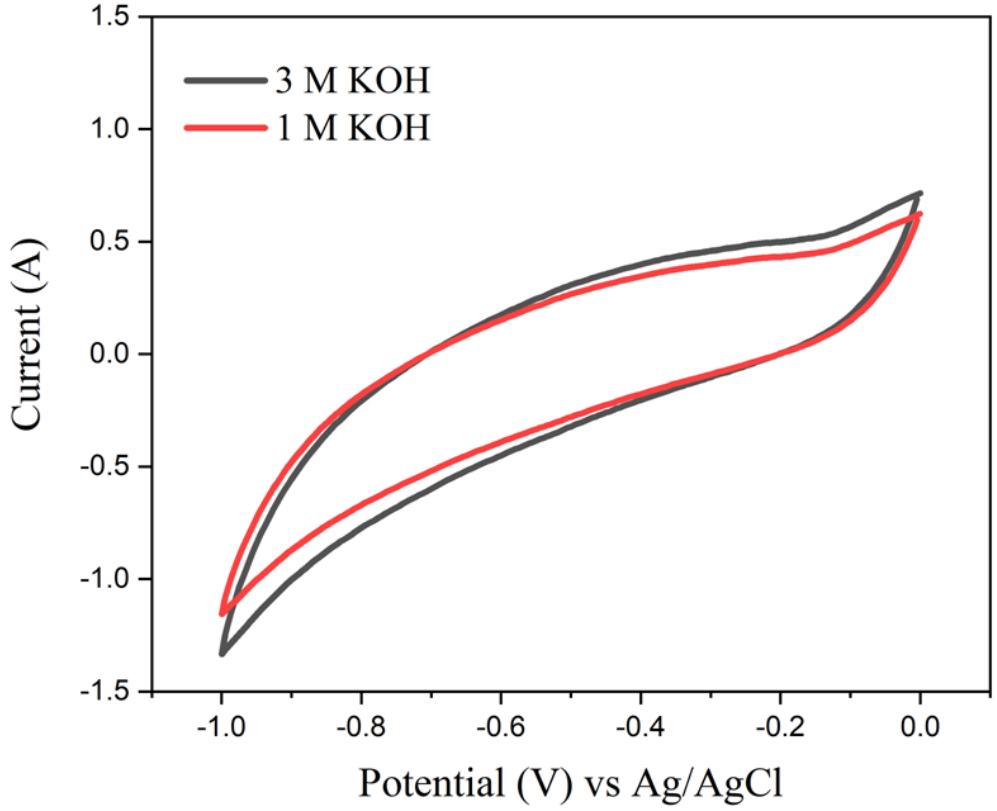

Figure 12 Comparison of CV curves at different concentrations of electrolyte

For comparison, CV test of the prepared electrode material was also carried out in 1 M aqueous KOH. However, CV curves at 3 M aqueous KOH were found to be larger in size than those in 1 M aqueous KOH. In figure 12, CV curves of the electrode material at scan rate of 10 mV s^{-1} has been compared for different concentrations of electrolyte. The larger area of CV curves indicates the higher capacitance of the electrode material. This is in agreement with the larger ionic concentration in 3 M aqueous KOH than 1 M aqueous KOH. These findings show as prepared AC can be a possible low cost electrode material for supercapacitor.

CHAPTER FIVE: CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In summary, activated carbon (AC) was successfully prepared from the waste betel nut by chemical activation method at 500 °C for 3 h by mixing waste betel nut powder and H_3PO_4 in the ration 1:1 in N_2 environment. Initial quality comparison with commercial AC was performed by determining iodine number (I_N), methylene blue (MB_N) and estimation of surface area (A). The I_N, MB_N and A values are respectively found to be 882 mg g^{-1}, 365 mg g^{-1}, and 927 m^2 g^{-1} which are comparable to the commercial AC. The values directed the resultant AC to have high porosity. Then, further analysis and characterization were performed. SEM image of as prepared AC showed the presence of mesopores on the surface. XRD and Raman spectroscopy analysis were compared and concluded that the resultant AC was graphitic in nature desirable for supercapacitor electrode material. FTIR analysis showed the presence of different oxygenated functional group. As prepared AC was concluded to have good absorptive property preferable for many applications like purification and charge storage.

Then, as prepared AC was utilized to fabricate the working electrode for the capacitor by depositing slurry obtained by mixing as prepared AC with 10 % polyvinylin difluoride (PVDF) and 10 % acetylene black grinded with piston by adding N-methyl-2-pyrrolidone (NMP). Then, electrochemical test was performed which showed the current increases with increase with scan rate according CV analysis and galvanostatic charge-discharge (GCD) showed the retention of capacitance 44.15 % . From the results, it can concluded be waste that betel nut based AC can be used as the electrode material for EDLC type supercapacitor.

5.2 Recommendation

The study covers the preparation of the waste betel nut based AC and test its storage performance. The research was conducted within the given time-frame with limited resources and materials available in the laboratory. So, following points are for the future works;

i) It is recommended to prepare and characterize the AC samples at different carbonization temperature, impregnation ratio and with various activating agents so that even better porosity and surface area could be detected.

ii) The fabrication of working electrode materials and its test to higher number of cycles could be performed which was not possible in our laboratory due to limited range of the device available in our laboratory.

iii) Waste betel nut based nanocomposite electrode materials can be fabricated and test its energy storage performance.

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To Whom It May Concern

This is to confirm that the paper titled "*Preparation of Waste Betel Nut based Activated Carbon (AC) to Test its Storage Performance*" submitted by **Ram Bahadur Ale** with Conference ID **12375** has been accepted for presentation at the 12th IOE Graduate Conference being held in October 19 – 22, 2022 at Thapathali Campus, Kathmandu.

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