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Activated carbon derived from non-metallic printed circuit board waste for supercapacitor application



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ABSTRACT

Activated carbons (ACs) have been synthesized by using waste PCBs via physical activation subsequent to pyrolysis processes. The physical and chemical properties of the produced activated carbons were studied using nitrogen adsorption, FT-IR spectroscopy, RAMAN spectroscopy, X-ray diffraction, X-ray photoelectron spectroscopy and scanning electron microscopy techniques. Among the synthesized ACs, AC with the highest surface area of $700 \, \text{m}^2 \, \text{g}^{-1}$ produced at $850 \, ^{\circ}\text{C}$ for a time interval of 5 h was subjected to electrochemical studies. Capacitance behaviour of the obtained AC sample has been evaluated using cyclic voltammetry (CV), galvanostatic charge–discharge (GC-D) measurements and electrochemical impedance spectroscopy (EIS) technique. Specific capacitance (C_{spec}) values vary from 220, 185 and $156 \, \text{Fg}^{-1}$ for corresponding scan rate of 30, 50 and $100 \, \text{mV s}^{-1}$ respectively. The well-developed surface area properties and good capacitance values associated with nitrogen functionalities indicates the AC developed is a good and suitable candidate for the supercapacitor fabrication.

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1. Introduction

Supercapacitors is one of the energy storage devices which is considered to be important due to its high power density, longer robustness, rapid charging ability and better efficiency over the rechargeable batteries [1,2]. Supercapacitors are classified into two types, based on energy storage principle; electric double layer capacitors (EDLCs) and pseudo capacitors [3]. EDLCs capacitance has its charge stored electrostatically at the electrode/electrolyte interface which depends significantly on the surface area of electrodes, which is in contact with electrolyte ions. In case of pseudocapacitors, the capacitance stores its charge depending on electron transfer between the electrolyte ion and the surfaces of the electrodes, i.e., electrodes are exposed to the fast and reversible faradaic redox reactions [4]. Recently various carbon based materials are used as electrodes for supercapacitors, which include activated carbons, mesoporous carbons, carbon nanotubes and graphene [5-7]. Carbon nanotubes and graphenes though extensively studied for supercapacitor applications has its drawback for being highly priced and tedious process involved in their

production. [8]. Activated carbon is considered as the most beneficial source of supercapacitor electrode material compared to the other materials due to its following advantages; effective electrical conductivity, high surface area, longer cycling life, relatively cost effective, high capacitance, good porosity, electrochemical stability and most importantly environment friendly nature [9-11]. Activated carbon has its precursors from various resources such as carbonaceous, agricultural and industrial materials (few examples are coal, coke, peat, petroleum, wood or coconut oil palm shells, banana fibers, corn grains, lapsi seed, needle cokes, seaweeds, sunflower shells, etc.) [12-14]. Out of above mentioned examples coal, coke and petroleum sources are expensive and rapidly exhaustible with high energy consumption. Hence agriculture-industry based activated carbon attracts more attention than the expensive activated carbon produced through coal. Henceforth finding an alternative waste source apart from agro-based wastes or non-biodegradable pollutant waste as resource will provide a potential opportunity for the synthesis of activated carbon and for the application in producing electrodes for supercapacitors.

Electronic wastes (e-wastes), one of the rapidly growing waste generates around 20–50 million tonnes annually. It continues to increase at a rapid rate of around 4–5 percent per year [15,16].

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Technological innovations and the rapid production of electronics have led to an associated increase in use of electronic devices resulting in significant generation of e-waste. Utilising this e-waste as a resource will provide an effective alternative to use of conventional raw materials. In particular printed circuit boards (PCBs) are prevalent in almost every electrical and electronic gadget. PCB is a complex structure to assemble and also to dismantle, as it is built by using both metals and non-metals. PCBs are basically classified into single/double sided and multi-layered PCB. The configurational structural arrangement of parts is more complex in multilayered PCBs than in the single/double sided PCB [17-20]. PCBs in computers and communication equipment are made from glass fibre reinforced epoxy resin (referred to commercially as FR-4 type) but televisions, monitors and home electronics predominantly use PCB's made of phenolic based polymeric material (FR-2 type). Generally in PCBs the arrangement of polymers, ceramics and metals assembled and manufactured in one substrate. In recent days the fabrication of PCBs are becoming more complex with further reduction in size therefore leading to extremely tedious to recycle or to recover the materials for further usage. Pyrolysis is one of the effective ways of recovering valuable materials from e-waste compared to other conventional physicochemical processes. Pyrolysis process involves heating the material in an inert atmosphere [21]. It is widely practiced for its ability to disintegrate the matrices of metal and non-metals in the complex designed PCB. The most common by-products produced during pyrolysis process are gas and residue [22]. Most of the research works were focussed on recovering valuable metals present in waste PCB but recycling non-metallic fractions (NMF) are neglected and landfilled again as secondary waste. Waste PCB of monitor from an End of life computer, which is considered to be polymer rich based material (FR-2) produces carbon rich nonmetallic residue after pyrolysis process. Therefore, this alternative secondary waste NMF can be considered for replacing the conventional sources to produce activated carbon which helps in maintaining the much needed sustainability. This further largely helps in bringing down the environmental concerns caused through e-waste and also as an electrode material for supercapacitor.

In this present study we have devised a novel approach by utilising the NMF of PCBs as the source for activated carbon. A detailed investigation has been performed to study the morphology and composition of the activated carbon using various analytical techniques. In addition, the synthesised AC from waste PCB was used as supercapacitor electrodes. The electrochemical properties of the prepared AC were analysed using Galvanostatic charge-discharge measurements; cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were used to study the electrochemical properties of the supercapacitor electrodes. This study can be demonstrated as a simple approach to synthesize AC. Further this approach will have a major effect in lessening the waste PCBs overall.

2. Experimental

2.1. Materials and methods

Random PCB of End of life monitor (polymer base material, FR-2 type) of a computer was picked up from the recycling unit of UNSW Australia, Sydney for this study. The multifarious PCB was physically dismantled from its units with the help of mechanical devices. Majority of transistors, capacitors, semiconductor circuits, battery, ceramic projections and microchips were also removed. The dismantled PCB from monitors was further truncated into possible smaller sizes (~1 cm) and then used accordingly. Homogenisation of the truncated PCB was achieved by converting

them into fine powders by using CryoMill (Mixer Mill—Retsch®) grinding technique under the Liquid Nitrogen (Dewar LN2 Auto fill 501—Retsch®) atmosphere.

Raw waste PCB powders and their non-metallic residue obtained after pyrolysis were examined using PANalytical PW2400 Sequential Wavelength Dispersive X-Ray Fluorescence (XRF) spectrometry to determine the composition of various elements.

2.2. Pyrolytic approach

Pyrolysis of PCB was performed under controlled conditions of atmospheric pressure in a horizontal tube furnace in an inert (argon) atmosphere. The furnace is programmed to a predetermined temperature of 800 °C which is controlled by a thermocouple. A measured quantity of waste PCB sample was kept in an alumina crucible and then placed on to a sample holder made of graphite. Before exposing the sample to hot zone of the furnace the sample holder along with the sample is placed in the cold zone for 5 minutes to avoid thermal shock. After 20 minutes in the hot zone the pyrolysed WPCB samples were taken out and allowed to cool down in argon atmosphere to room temperature. In the obtained pyrolysed residue, the metallic fractions (MF) were easily separated manually from the non-metallic fractions. The gases generated during the pyrolytic experiments were not recovered for analysis but allowed to escape the furnace using multi point exhaust system. The obtained non-metallic fraction residues were further characterised and used to prepare activated carbon.

2.3. Preparation of activation carbon

The activation of the obtained PCB non-metallic char is achieved by using physical or chemical method. CO_2 gas was used for the activation process mainly for its purity and easy to handle properties [23]. The weighed non-metallic waste PCB char was placed in the tubular furnace and then inert atmosphere was switched to CO_2 atmosphere. It was noted prior to the experiments that CO_2 gas leakage has been checked thoroughly to avoid failure in activation. The samples were activated at 3 different temperatures, $(650\,^{\circ}\text{C}, 750\,^{\circ}\text{C} \text{ and } 850\,^{\circ}\text{C})$ time intervals $(3\,\text{h} \text{ and } 5\,\text{h})$ and later cooled down in an inert atmosphere. The resulting activated carbon was further for characterised.

2.4. Characterisation of activated carbon

Nitrogen adsorption isotherms were analysed by means of nitrogen adsorption at 77 K using a TriStar 3000 V6.08 A. Samples were degassed at 150 °C under vacuum for atleast 3 h prior to measurement. The multipoint Brunauer-Emmette-Teller (BET) method was used to determine the total surface area. For the mesopore surface area, pore volume and pore diameter, the Barrette-Joynere-Halenda (BJH) method was used. Micropore surface area and pore volume were determined using the tmethod, and the micropore diameter was determined using the Dubinine-Astakhov (DA) method. The external surface area $S_{\rm ext}$ of the sample can be determined from the slope (s) of the t-plot between the relative pressure and volume adsorbed. Micropore surface area $(S_{\rm micro})$ can be calculated from the relation $S_{\rm micro}$ = $S_{\rm BET} - S_{\rm ext}$ (m²/g) Where, $S_{\rm ext}$ is calculated using the relation $S_{\rm ext}$ = $s \times 15.47$ (m²/g).

Scanning Electron Microscope (SEM) was used to describe the physical structure of the produced activated carbon. The samples were then examined and imaged using a TM 3000 Hitachi Scanning Electron Microscope (SEM).

XPS analysis was carried out on produced activated carbon obtained by using Thermo ESCALAB250i X-ray Photoelectron

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