SYNTHESIS OF NANO POROUS CARBON FIBERS FROM PLANT WASTE AND ITS **ANALYSIS**

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ABSTRACT: We reported here simple method of preparation of nano porous Carbon fibers (CFs) by thermal decomposition. Carbonization of waste plant material was carried out in single zone furnace. Scanning electron microscope (SEM) image revels that the Carbon fibers (CFs) formed around 200 ~ 500 nm in width. For the synthesis of Carbon fibers (CFs) processing on raw fibers is important step, for that naturally dried waste plant material was washed with distilled water and soaked in 20% NaOH solution for three days for the complete removal of alkaline material. These raw fibers then washed properly with distilled water and dried in oven at 120°C. Synthesis of carbon fibers (CFs) was carried out by using Carbonization method at 850°C. Hydrogen gas was used as carrier gas. The morphological structural properties of Carbon fibers (CFs) were investigated by scanning electron microscopy (SEM), EDX and XRD. The synthesized Carbon fibers (CFs) have a crystalline size of 200~ 500 nm. Surface of fibers is highly porous in nature with presence of small size pores. The pore cavities are present all over the surface. The side walls of each pore ranges in between 10~15 nm and inner diameter of pore cavities is in the range of 20~50 nm confirmed from SEM images. The crystalline nature of fibers are observed with help of XRD. Synthesised Carbon fibers (CFs) can be used in different fields like electrochemical sensors, Supercapacitors, fuel cell etc.

Keywords- Carbon fibers (CFs), carbonization, NaOH, porous, crystalline

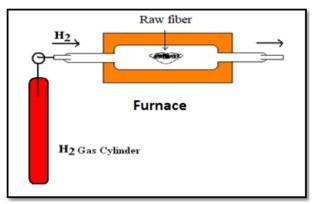
I. INTRODUCTION

Several scientists have studied nanoparticles and nano fibers of carbon in the last few decades. Nanoparticles are getting continuous importance for various applications such as catalysts supercapacitors, gas sensors, electrochemical devices, magnetic materials, rechargeable batteries, solar energy, absorber, pigment for ceramics biosensors, supercapacitors, fuel cell etc. [1-4]. Generally, Carbon fibers are prepared by using conventional source like petroleum and fossil fuel which are less cost effective. Therefore, we have to find alternate precursor for the preparation of Carbon fibers. Current report is synthesis of carbon fibers from plant waste by thermal decomposition in H₂ atmosphere. The prepared CFs underwent with various treatment like washing with distilled water, acid treatment and heating at 400°C. Several methods are reported for the preparation of carbon nano fibers like, arc discharge, laser ablation, thermal decomposition, chemical vapour deposition etc. In present work Carbonization of waste plant material was carried out in single zone furnace and synthesized carbon fibers (CFs) were characterized by XRD, SEM and EDX.

II. MATERIALS AND METHODS

Synthesis of Nano porous carbon fibers (CFs)

Firstly, collected waste fibrous plant material was dried naturally to remove moisture in it. This fibrous plant material was then washed with distilled water multiple times to remove dust and other impurities present on surface. Plant material is then soaked in 20% NaOH to remove other waste material than fibers. Plant material is soaked for three days with stirring in between for the complete removal of unwanted material, after that raw fibers obtained are scratched and washed with distilled water to remove NaOH. Raw fibers then dried at room temperature for one day and then in an oven at 125°C. The processed raw fibers became very clean and shiny. Carbon fibers (CFs) were prepared from raw fibers by carbonization in a single zone furnace (Fig.1) [5-8]. In furnace, unit weighed raw fibers were taken in quartz boat and kept at centre of furnace. Carrier gas H₂ was allowed to flow into the quartz tube with a fixed flow rate (6ml/min) to make oxygen free atmosphere. After 15 min of flow, furnace was switched on to reach the desired temperature 850°C. Around 400°C, decomposition process started which was observed by collection of carbon soot at opposite end of furnace. At the end of the desired time (1 hour) the furnace was switched off and allowed to cool at room temperature. Carbon fibers (CFs) formed inside the quartz boat was collected (Fig.2). Weighed CFs were washed with distilled water followed by treatment with 2N HCl till neutral pH and then dried and heated in muffle furnace at 400°C for 20 min to remove amorphous carbon. Prepared Carbon fibers were characterized by XRD, SEM and EDX [9,10].



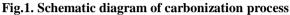


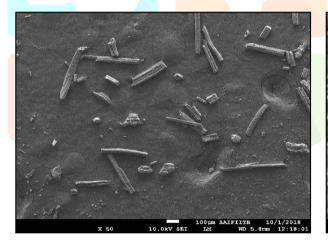


Fig.2. Photograph of Carbon Fibers (CFs) formed after carbonization

III. RESULTS AND DISCUSSION

SEM Study of Carbon Fibers

Under low resolution, SEM images (Fig.3) confirmed that carbon fibers of around 200-500 nm width are formed. A SEM image under high magnification shows surface of fibers is highly porous in nature with presence of small size porous cavities. The pore cavities are present all over the surface. The side walls of each pore ranges in between 10~15 nm and inner diameter of pore cavities is in the range of 20~50 nm confirmed from SEM images. To get better separation, Carbon fibers (CFs) were sonicated in ethyl alcohol for 30 minutes and dried under IR lamp. These highly porous carbon fibers expected to have high surface area [11-13].



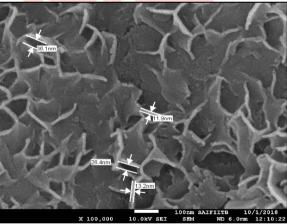


Fig.3. SEM Images for carbon fibers at low and high resolution

XRD Study of carbon fibers

XRD peak at $2\theta = 26.21$, 42.36, 51.71 corresponding to Carbon fibers (CFs) are shown in (fig.4). The crystallite size 'D' was obtained by the measurement of broadening of diffraction lines and application of the Debye-Sheerer equation, $D = 0.94 \lambda/\beta \frac{1}{2}COS\theta$. Where λ is wavelength of XRD radiation, β is the full width at half maximum of the peak corresponding to the plane. θ is the angle obtained from 2θ value corresponding to the XRD pattern. From the XRD graph of Carbon fibers (CFs) it is found that they are crystalline in nature.

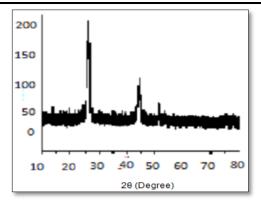


Fig. 4. XRD pattern of Carbon fibers (CFs)

EDX Study of Carbon fibers (CFs)

In order to confirm the carbon content EDX analysis was performed. During the EDX measurement different areas were focused and the spectrum was obtained for the type of element present. Atomic and weight % of elements were also recorded by EDX analysis which are listed in Table 1.

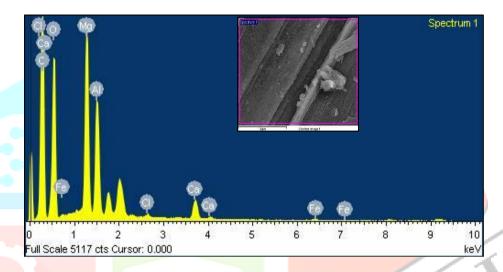


Fig.5. EDX Spectrum of Carbon fibers (CFs)

Table 1. Elemental Composition of Carbon Fibers (CFs)

Element	C K	ОК	Mg K	Al K	Cl K	Ca K	Fe K	Total
Weight %	44.15	35.90	10.08	6.71	0.23	2.40	0.53	100
Atomic %	55.20	33.70	6.22	3.74	0.10	0.90	0.14	

From Energy Dispersive X-Ray Analysis (EDX) study it is observed that fibers mainly contain carbon. Oxygen is detected due to the porous nature of carbon material. Around 20 % impurities of Mg, Al, Cl, Ca and Fe are present.

Carbon fibers heated to temperature 400°C to remove amorphous carbon. Amorphous carbon and matrix residue is also removed by treatment with 50% HCl to further increases in active surface area [14-20].

IV. CONCLUSION

By the use of waste plant material; nano porous carbon fibers (CFs) were successfully synthesized by carbonization method. The synthesized carbon fibers (CFs) are stack like, agglomerated, homogeneous in size and ranging in between 200-500 nm dimension. Surface of fibers is highly porous in nature with presence of small size porous groves. Fibers produced with this method are crystalline and adequate carbon content. Such highly porous carbon nano material like CFs will be suitable for potential application in supercapacitors, biosensors, electrochromic devices, etc. In further investigation, we are using synthesized carbon fibers (CFs) for the supercapacitor application.

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