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Novel Method to Prepare Activated Carbon Nanomaterials from Defunct Leaves

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Abstract

In this work, we collected the dead *Eucalyptus tereticornis* (Forest red gum) and *Azadirachta indica* (Neem) leaves, then washed, dried, powdered, mixed in equal proportionate and fired in high-temperature muffle furnace up to 750°C for 6hrs. The resultant mesoporus nano-carbon powders are then characterized using Scanning Electron Microscopy (SEM), Energy Dispersive Analysis X-Ray Spectroscopy (EDAX) and X-Ray Diffraction (XRD) method. The structural morphologies of the various samples were studied using SEM. The calcium content increased as inferred from the EDAX analysis, as the temperature of the firing increased to 750°C. The prepared activated carbon nanoparticles were confirmed with XRD to be the carbon-based nanocomposites such as calcium carbonate ($CaCO_3$), magnesium carbonate ($MgCO_3$) and non-carbon-based nanocomposites such as enstatite ($MgSiO_3$) and diopside ($CaMgSi_2O_6$). These results were promising for the supercapacitors in powering bio-medical device applications.

Keywords

Neem, Eucalyptus, SEM, EDAX, XRD, Supercapacitor

1. Introduction

The research gap bridging between electrolytic capacitors and rechargeable batteries are the supercapacitors. Researches in supercapacitors from eco-friendly contribution are promising especially in medical electronics device applications. A supercapacitor (often called an ultracapacitor) differs from an ordinary capacitor in two important ways: its plates effectively have a much bigger area and the distance between them is much smaller, because the separator between them works in a different way to a conventional dielectric. Like an ordinary capacitor, a supercapacitor has two plates that are separated. The plates are made from metal coated with a porous substance such as powdery, activated charcoal, which effectively gives them a bigger area for storing much more charge.

Activated carbon also called activated charcoal or activated coal, is a form of carbon processed to have small, low-volume pores that increase the surface area available for adsorption or chemical reactions. Activated is sometimes substituted with active. Due to its high degree of micro porosity, just one gram of activated carbon has a surface area in excess of 750 m^2, as determined by gas adsorption. An activation level sufficient for useful application may be attained solely from high surface area; however, further chemical treatment often enhances adsorption properties. Activated carbon is usually derived from charcoal and, increasingly, high-porosity bio char. Activated carbon is used in gas purification, decaffeination, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and respirators, filters in compressed air and many other applications. [1, 2]

It is none other than the carbon produced from carbonaceous source materials such as nutshells, coconut husk, peat, wood, coir, lignite, coal, and petroleum pitch. It can be produced by one of the following processes:

The source material is developed into activated carbons using hot gases. Air is then introduced to burn out the gasses, creating a graded, screened and de-dusted form of activated carbon. [3] This is generally done by using one or a combination of the following processes:

1. Carbonization: Material with carbon content is pyrolyzed at temperatures in the range 600–900°C, usually in inert atmosphere with gases like argon or nitrogen.
2. Activation/Oxidation: Raw material or carbonized material is exposed to oxidizing atmospheres (oxygen or steam) at temperatures above 250°C, usually in the temperature range of 600–1200°C.

Then the chemical activation of carbon is carried out by the following process: Initially, prior to carbonization, the raw material is impregnated with certain chemicals. The chemical is typically an acid, strong base, or a salt (phosphoric acid, potassium hydroxide, sodium hydroxide, calcium chloride, and zinc chloride 25%). Then, the raw material is carbonized at lower temperatures (450–900°C). It is believed that the carbonization / activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material. [4]

The present study is aimed at preparing activated carbon nanoparticles and nanocomposites from neem, eucalyptus and mixture of both by chemical activation procedure and will be tested using SEM, EDAX and XRD to study the carbon and composite nanomaterials characteristics of the converted material. Supercapacitor from mango leaves, neem leaves and other leaves were contributed by various researchers are the true motivation of this work. [5], [6]. No one reported with mixture of neem and eucalyptus leaves, which is innovative and new contribution to the research gap in the eco-friendly synthesis of activated carbon nanostructures and nanocomposites for the supercapacitor applications.

2. Experimental Methodology

First, the waste, defunct and dead neem and eucalyptus leaves from Karunya Institute of Technology and Sciences on the foothills of Western Ghats Hills, located near Coimbatore, in Southern India were collected, dried for 6 to 7 days and washed to remove the impurities present in them and again dried for 2 to 3 days. Then using the blender, the dried leaves are powdered. Now, the powdered samples were fired in the furnace for 300°C and 750°C for 6 hours and cooled overnight for the complete conversion of activated carbon. Finally, the characterization using SEM, EDAX and XRD carried out to understand the potential of the prepared carbon for the application of supercapacitors in medical device applications such as powering the device. The proposed work flowchart is shown in Figure 1.

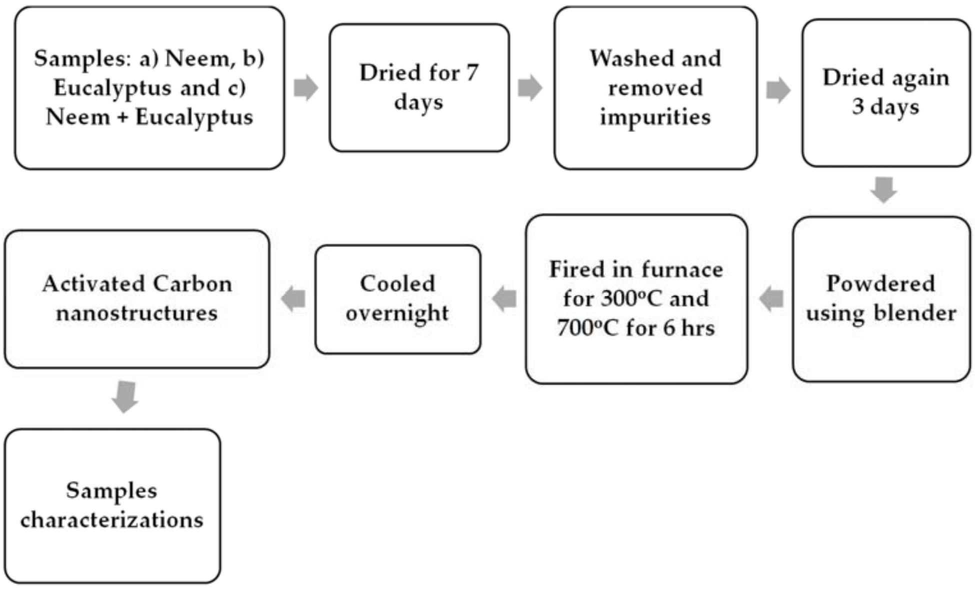

Figure 1. *Flowchart of the proposed work.*

3. Results and Discussions

3.1. Structural Morphology

The Scanning electron microscopy (SEM) is a best technique for probing plant surfaces at high resolution. Plant tissues are ought to be sealed by dehydration for observation in an electron microscope because the coating system and the microscopes operate under high vacuum and most specimens cannot withstand water removal by the vacuum system without distortion.

Figure 2 (a)-(i) shows the SEM morphology images of the various samples. They are: Figure 2 (a): neem powder, Figure 2 (b): eucalyptus powder, Figure 2 (c): the mixed powders of neem and eucalyptus. From figure (a)-(c) it is inferred that the structures are micro and mesoporous particles. A little shrinkage of the epidermal cells was observed as expected. Figure 2 (d) neem powder after heat treatment of 300°C for 6hrs and cooled overnight Figure 2 (e) eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight Figure 2 (f) the mixed neem and eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight Figure 2 (g) the neem powder after heat treatment of 750°C for 6hrs and cooled overnight Figure 2 (h) the eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight and Figure 2 (i) the mixed Neem and Eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight.

As inferred from the SEM images of the various samples as shown in Figure 2, figure 2 (a) – figure 2 (f), the magnification shown is X10,000 and for the figure 2 (g) – figure 2 (i), the magnification is X55,000.

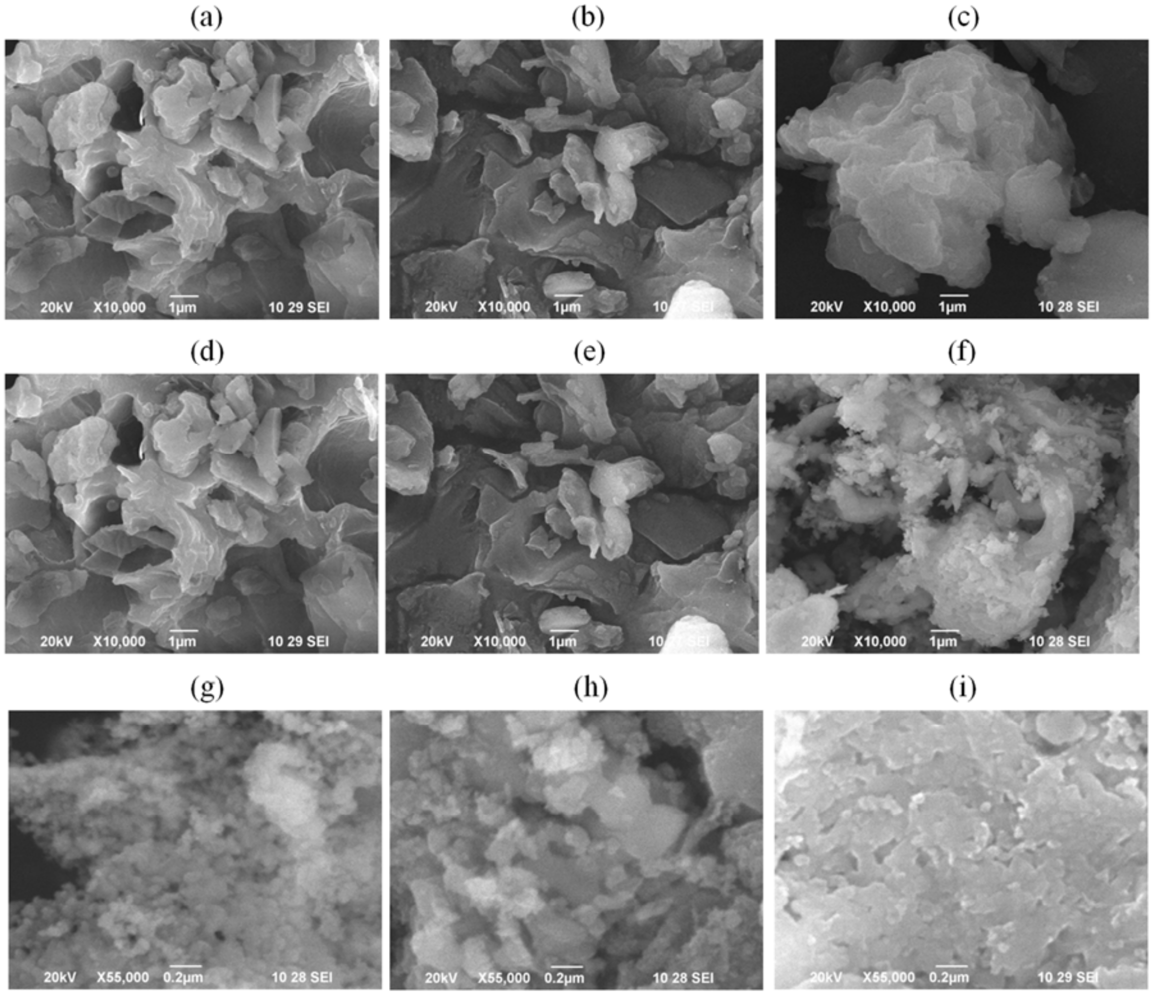

Figure 2. *SEM images of the following samples: (a) neem powder (b) eucalyptus powder (c) mixed powders of neem and eucalyptus (d) neem powder after heat treatment of 300°C for 6hrs and cooled overnight (e) eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight (f) the mixed neem and eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight (g) the neem powder after heat treatment of 750°C for 6hrs and cooled overnight (h) the eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight and (i) the mixed Neem and Eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight*

3.2. EDAX Analysis

The elemental composition is carried out using Energy Dispersive Spectroscopy (EDAX) Analysis as shown in Figure 2 (a)-(i). It includes mainly carbon (C), calcium (Ca), potassium (K), magnesium (Mg), oxygen (O), and residues of silicon (Si), aluminium (Al), iron (Fe), chlorine (Cl), Sodium (Na) and sulphur (S). Table 1 shows the elemental composition of the various samples. Kumar et. al. and Singh et. al. confirmed the presence of Calcium from Carbon prepared from Neem leaves [7], [8].

The elemental composition of the various samples with respect to the elements weight percent in a pictorial chart are shown in Figure 4. It helps easily to interpret the presence of each element in the particular sample. Sulphur (S) is the only element seen in the sample of eucalyptus powder subjected to 300°C (E300) and vice-versa.

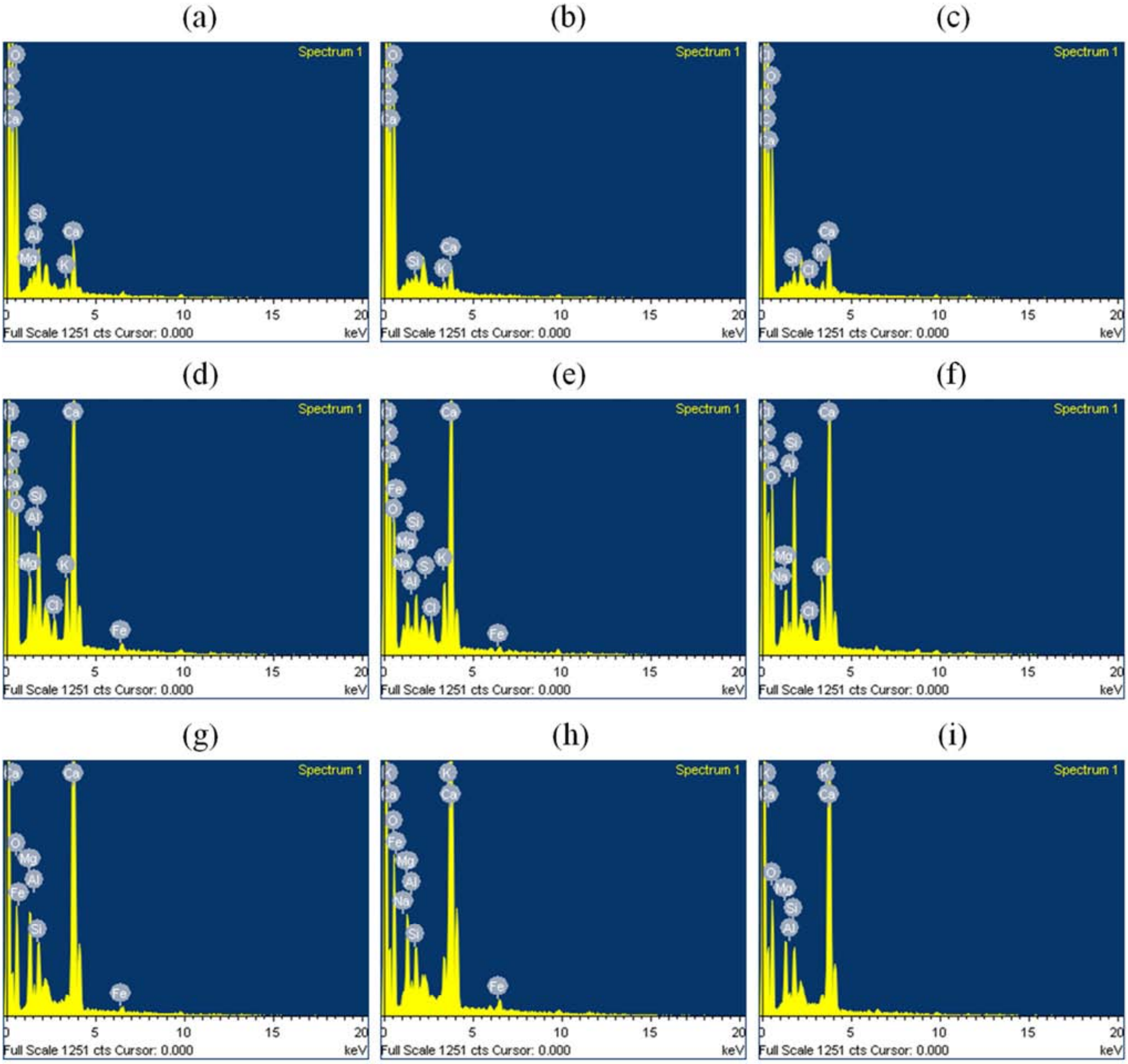

Figure 3. *EDAX spectrum of various samples: (a) neem powder (b) eucalyptus powder (c) mixed powders of neem and eucalyptus (d) neem powder after heat treatment of 300°C for 6hrs and cooled overnight (e) eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight (f) the mixed neem and eucalyptus powder after heat treatment of 300°C for 6hrs and cooled overnight (g) the neem powder after heat treatment of 750°C for 6hrs and cooled overnight (h) the eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight and (i) the mixed Neem and Eucalyptus powder after heat treatment of 750°C for 6hrs and cooled overnight*

Table 1. *Elemental composition of various samples.*

Samples Code	Elements weight%										
	C	**O**	**Ca**	**Mg**	**K**	**Na**	**Fe**	**Cl**	**S**	**Al**	**Si**
N	53.1	42.31	2.19	0.37	0.40	-	-	-	-	0.45	1.17
E	57.55	40.86	1.11	-	0.20	-	-	-	-	-	0.28
N+E	55.92	40.38	2.60	-	0.32	-	-	0.29	-	-	0.50
N300	*	60.01	22.89	4.28	3.24	-	1.13	1.27	-	1.73	5.44
E300	*	58.83	25.73	3.37	3.42	2.40	1.05	1.46	0.37	0.89	2.47
N+E300	*	58.93	21.45	3.81	3.37	1.76	-	1.01	-	0.92	8.74
N750	*	51.08	36.67	6.99	-	-	0.95	-	-	0.92	3.38
E750	*	51.54	37.63	4.75	1.06	1.32	0.88			0.77	2.04
N+E750	*	56.85	32.89	5.57	0.20	-	-	-	-	0.88	3.61

* - Not recorded for Carbon, as the samples are placed on a Carbon tape.

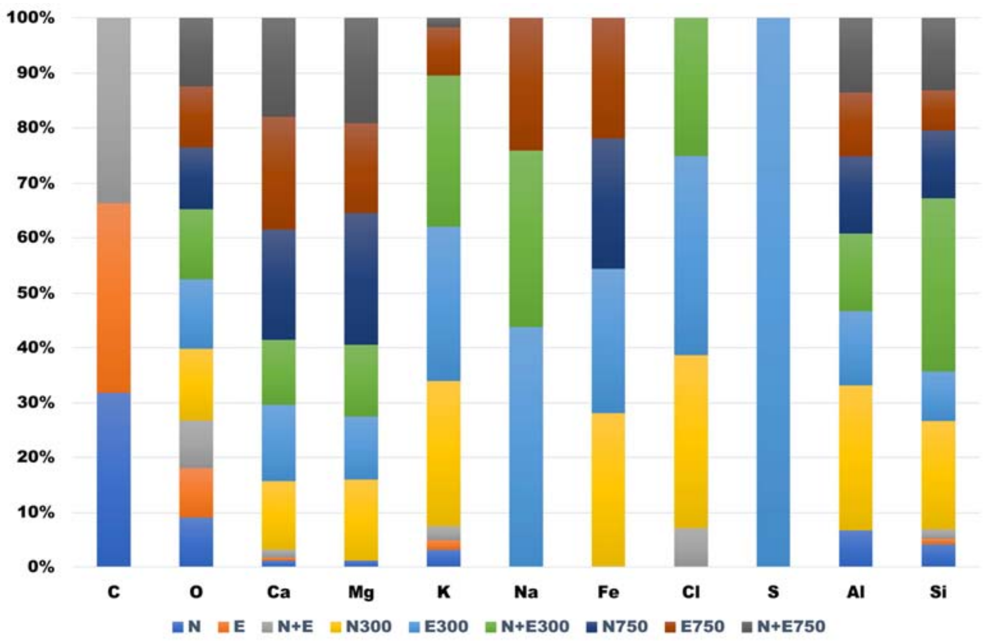

Figure 4. *Pictorial representation of the elemental composition of the various samples with respect to the elements weight percent.*

3.3. XRD Analysis

Figure 5 shows the XRD spectra gives the information of the crystal peaks of the crystalline CaO, $CaCO_3$, MgO, Carbon and presence of various other residue substances from the various samples. The fragments of $CaCO_3$ near 30° are oriented preferentially along the surface because of the perfect cleavage of calcite. [9] The figure shows XRD pattern of the starting materials and those had good agreement with the standards for $MgCO_3$ (JCPDS data file 01-071-1534) [10], $CaCO_3$ (JCPDS data file 00-005-0586) [11], talc (JCPDS data file 00-002-0066) [12] and SiO_2 (JCPDS data file 00-046-1045) compiled by the Joint Committee on Powder Diffraction and Standards (JCPDS). The characteristic peaks for the heated samples correspond to MgO (JCPDS data file 01-071-1176), CaO (JCPDS data file 00-017-0912), and residues of SiO_2 (JCPDS data file 00-046-1045). These resultant powders are the nanocomposites with the inclusion of Ca, Si, C, Mg and O. One such nanocomposites corresponds to enstatite, $MgSiO_3$ (JCPDS data file 00-022-0714) [9] and the other one is to diopside, $CaMgSi_2O_6$ (XRD JCPDS data file 01-072-1497) [9],[13]. The residues are Fe, Cl, Al, Na, S and K as inferred from the EDAX spectra.

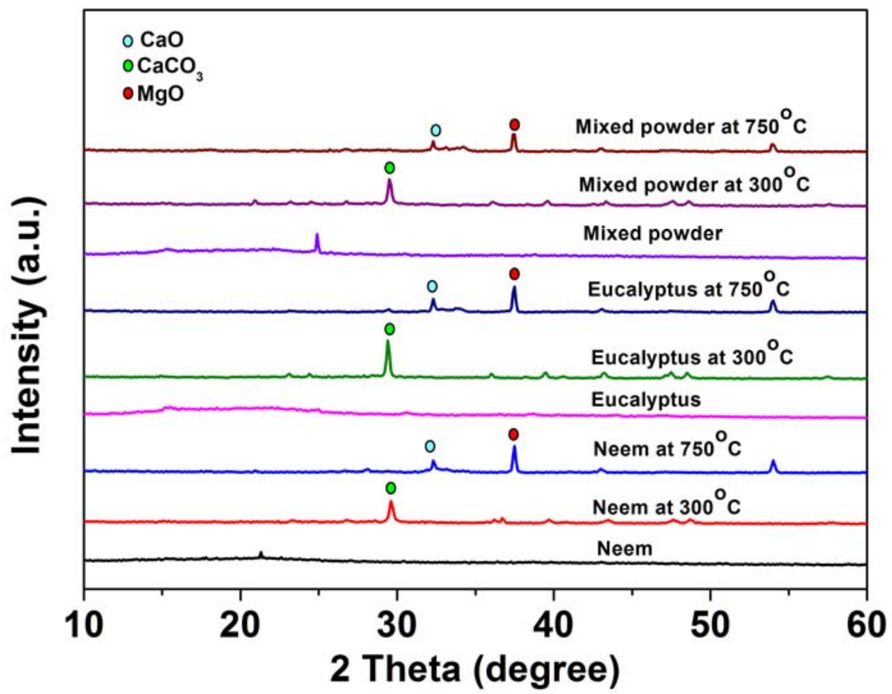

Figure 5. *XRD spectra of the various samples.*

4. Conclusion

This study is the first report of successfully synthesized the pure activated Carbon nanoparticles and its derivates of nanocomposites which includes carbon-based nanocomposites such as calcium carbonate ($CaCO_3$), magnesium carbonate ($MgCO_3$) and non-carbon-based nanocomposites such as enstatite ($MgSiO_3$) and diopside ($CaMgSi_2O_6$) from the defunct Neem and Eucalyptus leaves. The microporous nature of the neem, eucalyptus and mixture of both leaves are seen through the SEM morphology analysis. The XRD gives the detailed description of the crystallinity nature of the prepared powder and confirms the presence of the various nanocomposites. The study using EDAX carried out to study the various elements present in the prepared powder and presence of nanocomposites with the activated carbon. As a future scope, the waste neem and eucalyptus leaves from Karunya Institute of Technology and Sciences on the foothills of Western Ghats Hills may be effectively used for the supercapacitors as activated carbon in medical device applications.

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Declaration

The authors declare no conflict of interest.

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