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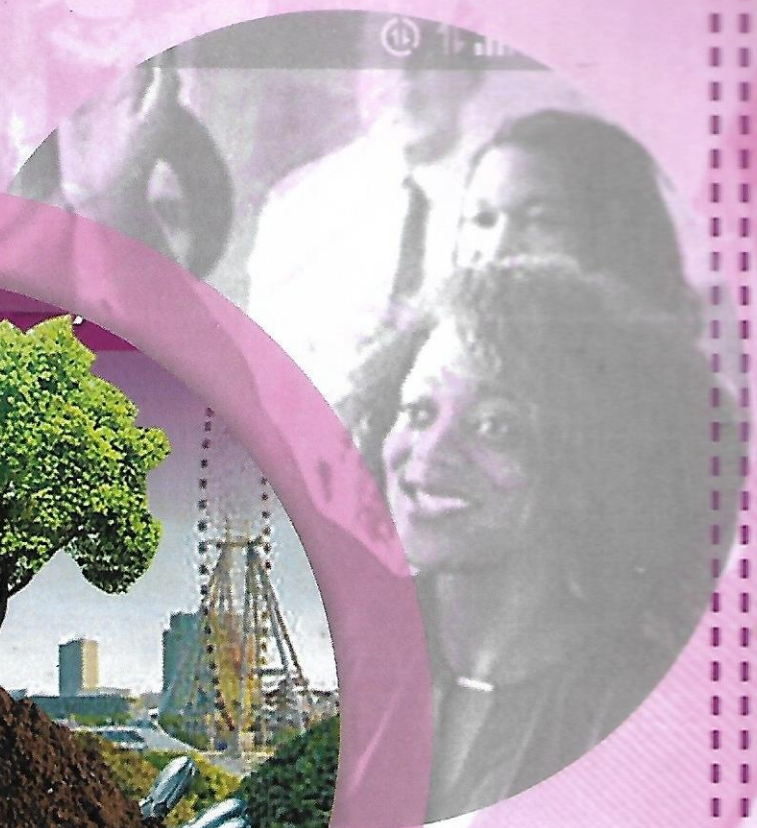

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Development of Nickel-Ferrite – Activated Carbon ($NiFe_2O_4$ - AC) Adsorbent Via Polyethylene Glycol (PEG) Assisted Sol-Gel Combustion Method

Bitrus James Davou, Moses Aderemi Olutoye, Manasseh Auta
Chemical Engineering Department, Federal University of Technology, PMB 65 Minna Niger State, Nigeria

Email: jamesbitrus007@gmail.com, 08029856929

ABSTRACT
The aim of this work is to synthesize and characterize Nickel ferrite – activated carbon ($NiFe_2O_4$ – AC) adsorbent via polyethylene glycol (PEG) assisted sol-gel auto-combustion method using nitrate precursors and activated carbon from corn cobs and calcination procedure in nitrogen atmosphere. The physicochemical properties of nickel ferrite-activated carbon were studied by X-ray diffraction, (XRD), scanning electron microscopy (SEM), and Brunauer-Emmet-Teller (BET) analysis. The diffraction peaks for carbon from XRD analysis were 26.66° and 43,42 ° and that of nickel ferrite were 35.80 °, 43.42 °, 57.58 ° and 62.99 °. The average crystallite size D_c = 35.23 nm. The surface area, pore volume and pore size for activated carbon and nickel ferrite – activated carbon were determined to be 980.4 m^2g, 0.20 cc/g, 2.132 nm and 2657.0 m^2/g, 0.2641 cc/g, 2.128 nm respectively. These results proved that nickel ferrite-activated carbon adsorbent with good adsorption properties was successfully synthesized.

Keywords: *Adsorption, activation, nano-adsorbent, nickel ferrite, polyethylene glycol* .

1 INTRODUCTION

In recent years, nano-particle materials have been investigated for their potential use as adsorbent. The smaller size of the nanoadsorbent increases the surface area (Gubin et al., 2005) which boosts the chemical activity and adsorption capacity of nanoparticles for the adsorption of metals on their surface (Gubin et al., 2005; Kalfa et al., 2009). Nano-adsorbents possess two main properties: innate surface and external functionalization (Muzammil et al., 2016). Size, surface chemistry, agglomeration state, shape and fractal dimension, chemical composition, crystal structure and solubility are some of the factors controlling nanoadsorbents properties (Muzammil et all, 2016). Nano-particle are prominent as compared to other substances such as normal scale alumina and titanium dioxide as a result of their fine grain size and chemical activity (Kalfa et al., 2009; Zhang et al., 2008). The frequently used nanoparticles for the adsorption of heavy metals are activated carbon and carbon nanotubes, manganese oxide, grapheme, zinc oxide, magnesium oxide, titanium oxide and ferric oxide (Gupta el al., 2015). Spinel nickel ferrite is one of the most versatile important magnetic materials owing to its high chemical and electrochemical stability, high saturation magnetization, catalytic behavior, low conductivity and high permeability that makes nickel ferrite an, effective, efficient and capable adsorbent material (Goldman A., 2006; Gunjakar et., 2008) and is considered good adsorbent for heavy metal removal because of its high surface-to-volume ratio, tunable size and shape and tunable magnetic property (Kang et al., 2015). Activated carbon, in many instances, the most used not only because of its high adsorptive capacity (Kadirvelu et al., 2003; Masrh and Rodriguez-Reinono, 2006; Sircar et al., 1996) and its intrinsic physicochemical properties such as porosity, specific surface area and the nature of surface, but also because of the abundance of raw material from which it can be prepared.

Composition and microstructure are two factors that influence the properties of synthesized materials, and are sensitive to the preparation methodology used (Jiang and Yang, 2007). It is known that the crystal size is related to the relative interdependence between the nucleation and growth steps, which can be strongly influenced by the solution chemistry and precipitation conditions (Cedeno-Mattei and Perales-Perez, 2009). Various methods such as co-precipitation method (Sivakumar et al, 2011; Sagadevan et al., 2018), wet chemical co-precipitation technique (Patange et al., 2010; Sivagurunathan and Gibin, 2016: Tahir et al., 2017), gel-assistant hydrothermal route (Chena et al., 2010,), hydrothermal method (H. Li et al., 2010), thermal treatment method (Mahmoud and Abdul, 2011), sol-gel auto-combustion method (Rezlescu et al., 2006: Shobana and Sankar, 2009; Petrisor et al., 2015), citric acid combustion method (J. Zhu et al., 2006), organic gel-thermal decomposition method (Guo et al., 2010)), microwave synthesis (Bensebaa et al, 2004: Berthelin et al., 2008; Fu & Lin, 2009) and micro-emulsion (Liu et al., 2000; Hirai et al., 1999) has been used in the synthesis of nickel ferrite.

This present paper is focused on synthesizing and characterizing nickel ferrite-activated carbon ($NiFe_2O_4$ – AC) adsorbent via polyethylene glycol (PEG) assisted sol-gel combustion method.

2 METHODOLOGY

2.1 PRODUCTION OF ACTIVATED CARBON

The activated carbon was produced through a two-step chemical activation process. Corn cobs were local sourced, washed and dried under sun light for a week. Sufficient quantity was then sized reduced and pre-carbonized at 400°C for one hour in inert atmosphere after which the pre-carbonized sample was then impregnated with potassium hydroxide (KOH) at 1:3 precursors to KOH for 2 hours. The mixture was dried and then thermally treated at 710°C for 30 minutes. The sample obtained was allowed to cool and was washed and filtered several times with distilled water until a neutral solution is obtained. The sample obtained was then dried at 110°C for 8 hours, after which the sample was grind into powder. During the activation process, the following reactions take place (Lillo-Rodenas et al., 2003; Joseph et al., 2006).

$C + 2KOH \quad 2K + H_2 + CO_2$ (1)

$C + 2KOH \quad 2K + H_2O + CO$ (2)

$CO_2 + 2KOH \quad K_2CO_3 + H_2O$ (3)

2.2 SYNTHESIS OF NICKEL FERRITE-ACTIVATED CARBON

The Nickel ferrite-activated carbon was synthesized by polyethylene glycol (PEG) assisted sol-gel combustion method. 0.17mol $Fe(NO_3)_3.9H_2O$ and 0.085mol $Ni(NO_3)_2.6H_2O$ (98.99% pure JHD) were dissolved in minimum amount of polyethylene glycol at room temperature. The stoichiometric ratio of iron nitrate to nickel nitrate was 2:1. 5g of activated carbon was added into the mixture and the sol was heated to 100°C and stirred vigorously to form a wet gel. The gel was dried at 150°C for 5 hours. The aim of the PEG addition is to act as an anti-foaming agent, reducing agent and prevent an increase in size of the nanoparticles. The nitrates ion acts as an oxidant and provides an in situ oxidizing environment for the decomposition of the organic component. The product obtained was then calcined at 500°C for 1 hour.

3 RESULTS AND DISCUSSION

The XRD pattern of the $NiFe_2O_4$-AC nanoparticles was recorded using powder X-ray diffractometer with a diffraction angle between 10° and 75°. Figure 1 shows the XRD pattern of the as-prepared $NiFe_2O_4$-AC nanoparticles. The pattern shows distinct peaks indicating the formation of single face cubic spinal $NiFe_2O_4$ crystal structures at 2 theta angle at 35.80°, 43.42°, 57.58° and 62.99°. The peaks at diffraction angle 26.66° and 43.42° indicates the presence of crystallite carbon and amorphous carbon respectively. The average crystallite size of the sample was calculated using Scherrer's equation (Sivakumar et al., 2011),

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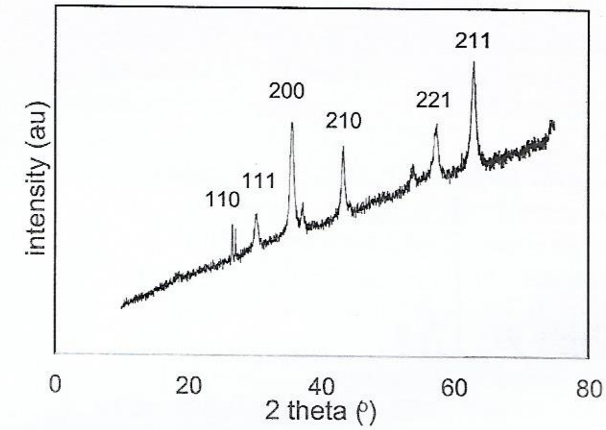

Figure 1: XRD pattern for synthesized nickel ferrite-activated carbon

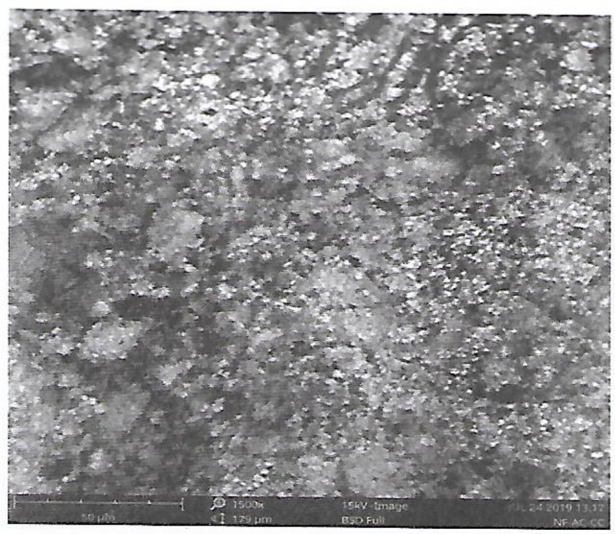

Figure 2: SEM images of NIF_e2O_4-AC nanoparticles

where λ is the X-ray wavelength (CuKa radiation) and equals to 0.154nm, θ is the Bragg diffraction angle, and β is the full width at half maximum (FWHM) of the XRD peak appearing at the diffraction angle θ. The average crystalline size was calculated from the X-ray line broadening using Scherrer's equation and it was found to be 33.73nm.

The morphology and size distribution of the $NiFe_2O_4$-AC nanoparticles were determined using SEM. Typical SEM images of $NiFe_2O_4$-AC synthesized particles are shown in figure 3. SEM micrograph depicts that the samples contain micrometrical aggregation of tiny particles. The existence of the high dense agglomeration indicates that pore free crystallites are present on the surface (Sagadevan, Chowdhury, Rafique, & Brunswick, 2018). The SEM images show the agglomerated form of $NiFe_2O_4$-AC nanoparticles. As the nanoparticles possess high surface energies, they tend to agglomerate and grow into larger assemblies.

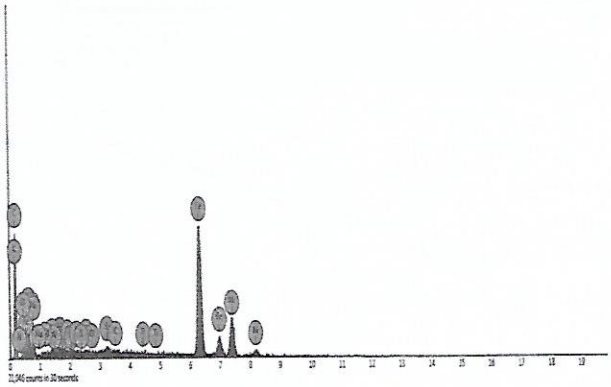

Figure 3: EDX spectra of $NiFe_2O_4$-AC

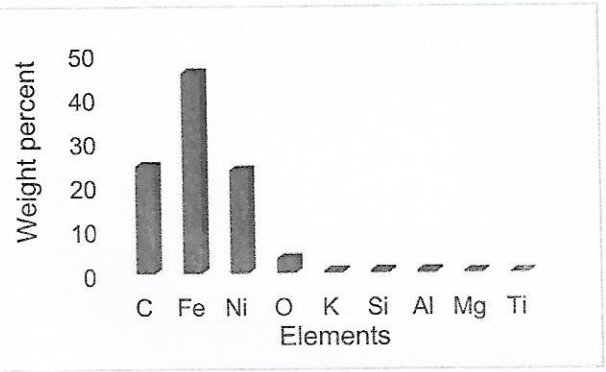

Figure 1: Percentage weight concentration of the various elements present in the sample

Figure 3 shows the EDX spectra for the synthesized NiFe2O4-AC and Figure 4 shows the weight concentration of the elements presents in the synthesized sample. From figure 4, it can be seen that the sample obtained shows the expected stoichiometry of Fe and Ni (i.e. 2:1).

The multipoint BET results is shown in table 1. The surface area for the activated carbon synthesized from corn cob is comparable to reported results by Diya'uddeen et al., 2008; Song et al., 2013).

TABLE 1: BET RESULTS

Sample	Surface area (m^2/g)	Pore volume(cc/g)	Pore size (nm)
Activated carbon	980.4	0.20	2.132
$NiFe_2O_4$-AC	2657.0	0.264	2.128

4 CONCLUSION

Nickel ferrite-activated carbon adsorbent were prepared by PEG assisted sol-gel combustion method and calcination in inert atmosphere. The physicochemical characterization of the so synthesized samples by XRD analysis proves the existence of crystallite nickel ferrite phase and carbon. The agglomerated form of the nanoparticles and stoichiometry was also confirmed and made clear by the SEM analysis and EDX spectra. The BET results strongly suggest the large surface area and pore volume indicates that $NiFe_2O_4$-AC can be used as catalyst and as adsorbent respectively. The average crystallite size of $NiFe_2O_4$-AC nanoparticles was found to be 35.23nm. The PEG plays an important role in controlling the particle size in the synthesis process.

ACKNOWLEDGEMENTS

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