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IN-SITU PRODUCTION OF ZNO SUPPORTED ON ACTIVATED CARBON ADSORBENT FROM ORANGE PEEL VIA PYROLYSIS PROCESS

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ABSTRACT

The present study focuses on the in-situ production of ZnO supported on activated carbon by simple pyrolysis process. The produced adsorbents were characterized via X-Ray Diffraction (XRD), Scanning electron microscope (SEM) and Fourier Transform Infrared (FTIR). The scanning electron microscope depicts the formation of irregular shaped particles of the produced adsorbent while that of ZnO supported on activated carbon produced a pristine particles. The XRD spectral of the activated carbon and the ZnO activated carbon depict the formation of C in the form of amorphous material. The orange peel used for the production of the activated carbon indicate its suitable as a precursor for activated carbon production. The surface properties including crystallinity shows its possible application as adsorbent for the purification of wastewater.

Keywords: *Production, Supported, Activated, Adsorbent, Orange.*

INTRODUCTION

Since the industrial revolution, rapid developments in industrialization, population expansion, and urbanization have largely contributed to the severe air, water and soil pollution ((Kim and Aga, 2007). A high number of pollutants discharged from industrial processes and households annually have caused significant effects on the eco-environment and human life. A number of physical, chemical and biological technologies have been developed to control the pollution successfully (Gadd, 2009). Elements, such as Cr, Cd, Hg, As and Pb are the most common toxic heavy metals in water system.

The improvement of water quality has provided incentives to develop new technologies and/or increase the performance of existing technologies. Various methods exist for the removal of metal ions from aqueous solutions, but adsorption is the most versatile and widely used process because of its extremely high removal efficiency and economic consideration. Additionally, adsorption does not result in any secondary pollution by producing harmful substances during the process. These make it a better option than the common methods and create a research interest in developing low-cost adsorbents. Several inorganic and organic adsorbents have been proposed for use in adsorption methods, including zeolites, biosorbents, and activated carbon

There is also a large amount of interest in the use of nanoparticles for the removal of heavy metals (Afkhami *et al*., 2010) due to their numerous special properties, such as high surface area and adsorption capacity, and simple production (Afkhami *et al*., 2010). Water pollution is of great concern since water is the inevitable requisite for the survival of all living organisms. Water pollution has become a continuous increasing problem on the earth which is danger for living things. Among the various water pollutants, heavy metals require special attention because of their toxic effect on humans and the environment. Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metal ions do not degrade into harmless end products1. The increased use of heavy metals by industries resulted in an increase amount of metallic substances in natural source water (Tangjuank *et al*., 2009).

The sources of metal ion pollutants are wastewaters of mining industries, paints and pigment industries, fertilizer industries, metal plating industries, batteries, pharmaceuticals and tannery industries (Jayaram *et al.,* 2009). A large number of elements fall into this category, but lead, copper and cadmium are those of relevance in the environmental context. Removal of these heavy metals from industrial wastewater has become important because its toxicity in human leads to disruption of the biosynthesis hemoglobin, rise in blood pressure, kidney damage, miscarriages and abortions, brain damage and learning disabilities in children (Iram *et al.,* 2010).

Commonly used methods for the removal of heavy metals from aqueous solutions are chemical precipitation, co-precipitation, adsorption, flocculation, reverse osmosis, ion exchange, electro deposition and filtration. Most of these methods have several disadvantages such as chemical requirements, time consuming procedure, production of large amount of sludge, low efficiency and less cost effective. However, adsorption method is considered to be more efficient, cost effective and free from sludge formation. Fenton with nano inorganic fillers is another advanced process for the mineralization of various pollutants. Nanomaterials have a wide range of applications, as in the technological and environmental challenges in the areas of solar energy conversion, catalysis, medicine and water treatments. The possibility of preparing Fenton's reagent in the form of nanoparticles has opened a new and exciting research field, with revolutionary applications not only in the electronic/modern technology but also in the field of environmental remediation. So, scientists are eager to resolve these issues to make the environment clean.

Nanostructured ZnO materials have received broad attention due to their distinguished performance in electronics, optics and photonics. From the 1960s, synthesis of ZnO thin films has been an active field because of their applications as sensors, transducers and catalysts. In the last few decades, especially since the nanotechnology initiative led by the US, study of one dimensional (1D) materials has become a leading edge in nanoscience and nanotechnology. With reduction in size, novel electrical, mechanical, chemical and optical properties are introduced, which are largely believed to be the result of surface and quantum confinement effects (Biswa, 1998). Zinc oxide is an inorganic compound usually appears as a white powder, and slightly soluble in water. The powder is widely used as an additive in numerous materials and products including plastics, ceramics, glass, cement, rubber (e.g. car tires), lubricants, paints, ointments, adhesives, sealants, pigments, foods (source of Zn nutrient), batteries, ferrites, fire retardants, etc. ZnO is present in the Earth crust as a mineral zincates; however, most ZnO used commercially are produced synthetically. In materials science, ZnO is often called an II-VI semiconductor because zinc and oxygen belong to the 2nd and 6th groups of the periodic table, respectively. This semiconductor has several favorable properties like good transparency, high electron mobility, wide band gap, strong room temperature luminescence. The application of ZnO/activated carbon adsorbent for the removal of Cadmium and lead from pharmacutical wastewater is not popular in literature.

In this present research, ZnO/activated carbon was developed via in-situ-wet impregnation and characterized for surface area, surface morphology/elemental analysis, crystallinity and functional groups via BET, SEM/EDX, XRD and FTIR, respectively.

Materials, Methods and Procedures

Materials

The chemical used in this study are of analytical grade with percentage purity of about 99.99 %. The chemicals include zinc chloride, ethanol and distilled water. The Orange peel sample was sourced from Bosso market, Minna, Niger State, Nigeria from the local orange sellers.

Preparation of activated carbon

Prior to activated carbon production, orange peels was collected, dirt were removed then soaked in hot water for 30 minutes to remove any accompanied dissolved impurities. This procedure will be repeated three times, thereafter; it will be washed with distilled water at ordinary temperature and dried at 110 ^{o}C in air oven. The dried samples will be milled with the aid of a pestle and mortal then sieved using 500 μm sieve size. The sized sample will be heated in horizontal CVD equipment at a temperature of 400 ^{o}C to form an activated carbon with reduced accompanied ash formation. The equipment will be allowed to cool and the sample will be removed, washed and dried at 110 ^{o}C in air oven for 5 h. The resulting sample will be kept for further use and analysis.

Production of ZnO supported on activated carbon

In-situ synthesis method was adopted for the synthesis of the ZnO supported activated carbon. 2.5 g of zinc chloride salt was weighed and dissolved in 10 mL of methanol solution. The resulting solution was impregnated in the produced activated carbon. The resulting mixture was left to age overnight, and then calcined at a temperature of 400 ^{o}C for a period of 1 h in nitrogen environment. The obtained adsorbent; ZnO supported on activated carbon was then characterized to determine the surface morphology, functional group and crystallinity using SEM, FTIR and XRD respectively.

Results and Discussion

The properties of the developed materials (activated carbon and the ZnO supported activated carbon) were characterized to determine the surface properties which give an insight to the adsorptive characteristics of the adsorbent. Figure 1 shows the scanning electron microscope of the developed activated carbon and the ZnO supported on activated carbon.

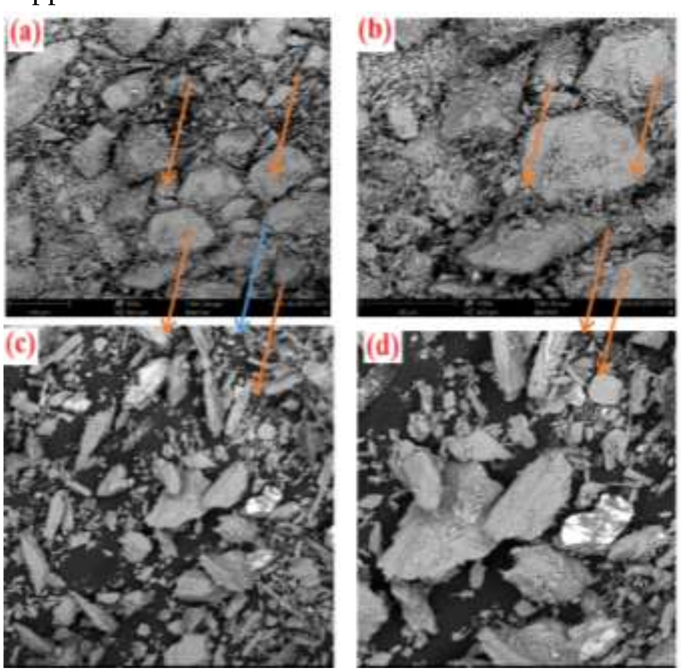

Figure 1: SEM micrograph of the produced (a-b) activated carbon (c-d) ZnO supported on activated carbon adsorbent

From Figure 1 a-b, the surface morphology of the activated carbon depicts the formation of an irregular shaped material with a little formation of cloud-like materials on the surface of the adsorbent. This could be as a result of oxidation process taking place on the surfaces of the adsorbent during the calcination process. The formations of cleared and pristine nature of particles were formed when ZnO was incorporated into the bulk of the adsorbent as depicted in Figure 1 (c-d). The introduction of ZnO; a more crystalline material improved the morphology of the adsorbent.

The surface functional group present on the surfaces of the activated carbon and the ZnO supported activated carbon were also determined and the result is as shown in Figure 2.

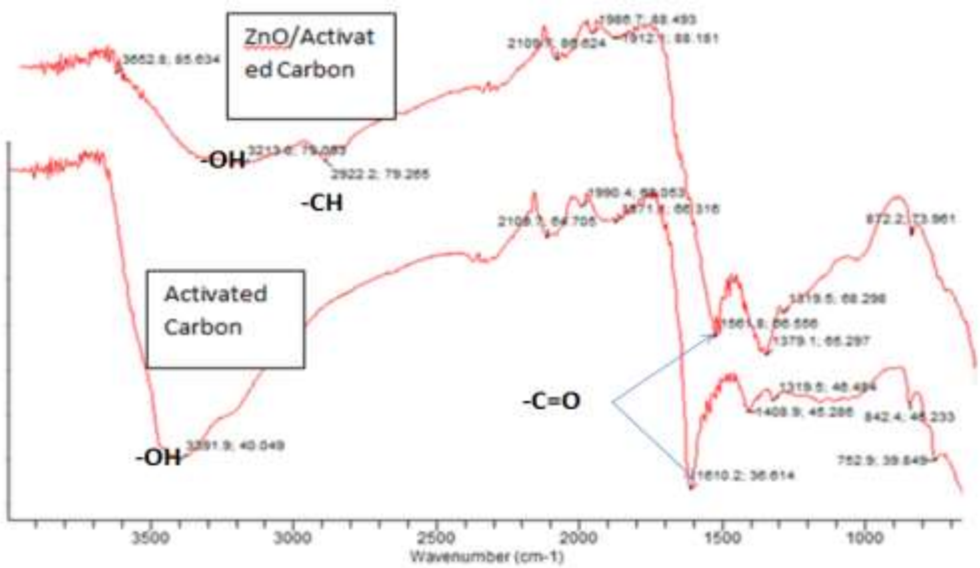

Figure 2: FTIR spectral of the developed activated carbon and ZnO supported on activated carbon adsorbent

Table 1 shows the peaks assignment present on the surfaces of the adsorbents materials. This shows the various absorption bounds present on the produced adsorbents.

Table 1: FTIR peaks assignment of the developed activated and ZnO supported on activated carbon adsorbents

Peaks	Assignment
3391.9 cm^{-1}	-OH
2922.2 cm^{-1}	`-CH Stretching
1610.5 cm^{-1}	-C=O
872.3 cm^{-1}	-CH out of plane

The presence of –OH bound is generally observed in both the adsorbents. The formation of –OH bound is linked to the presence of moisture in the materials while the peak at 2922.2 cm^{-1} indicates -CH stretching of aliphatic hydrocarbon presence in the developed ZnO supported activated carbon. The absence of this peak on the surface of the pure activated carbon could be as a result of the formation of amorphous

materials which covers the surfaces of the activated carbon after the calcination process. The presence of carboxyl group is highly observable and noticeable at the absorption band of 1610.5 cm^{-1} throughout the adsorbent. According to Yalcin *et al.* (2012), peak 872.3 cm^{-1} indicates –CH groups which are out of plane.
The crystallinity of the produced activated carbon and ZnO supported activated carbon adsorbents were determined via XRD technique and the results are depicted in Figure 3.

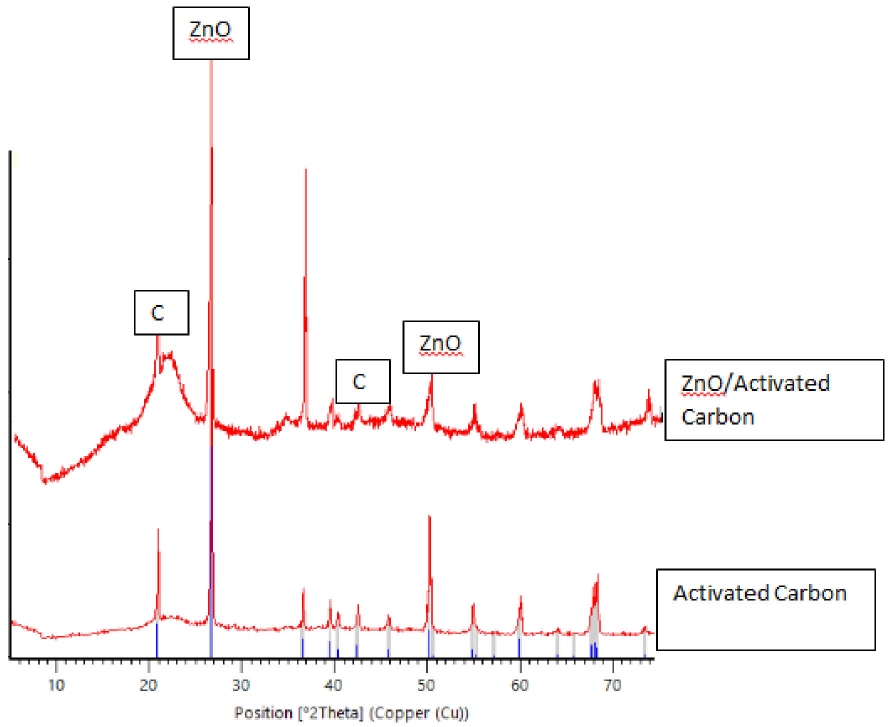

Figure 3: XRD spectra of the developed activated carbon and ZnO supported on activated carbon
The crystallinity of the developed material showed in Figure 3 shows the presence of several peaks at varied diffraction angles. The presence of carbon is observed at two different diffraction angles; 24.1^{o} and 43.2^{o}. This is a characteristics peaks of carbon material. The peak formation of the carbon materials is found to be wide, the wideness indicate the amorphous nature of the particles produced. The peaks at 28.54 and 50.32 ^{o} are attributed to the presence of ZnO on the surfaces of the adsorbent.

Conclusion

From the various analysis conducted, the produced activated carbon produced via pyrolysis process resulted to the production of high grade activated carbon. The SEM of the ZnO supported activated carbon adsorbent revealed formation of pristine particle sizes with reduced amorphous materials. The presence of C and ZnO incorporated materials are observed via the use of XRD technique.

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