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Spectral Analysis of Manganese Oxide Nano-Particles and Magnetic Sand Composites Using Scanning Electron Microscopy

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***Abstract* - The synthesis of manganese oxide and magnetic sand nanocomposites was reported in this study. The MnO_2 nanoparticles was synthesized using biological approach in which aqueous Cassia tora leaf extract was used to bioreduced potassium permanganate ($KMnO_4$) to manganese oxide and characterized using Fourier Transformation Infrared (FTIR) spectroscopy, UV-visible spectrophotometer, XRD and SEM. The UV-visible spectrophotometer gave absorption at 290 nm, while the crystalline size was obtained from XRD technique. Physical method was adopted for magnetic sand synthesis using vibrating mill. This was achieved by Agate Moter before sieving through 63 microns' sieve. The formed magnetic sand was characterized using FTIR, XRD, X-ray fluorescent (XRF). The result of the XRD shows that the crystalline size of the particles. The XRF shows that the sand contains high percentage of SiO_2 about 74.38%. The result shows that the nanocomposite can be used for the degradation of methyl orange and can be reused for a series of cycles without a significant decrease in the degradation ability.**

Keywords: Synthesis, Characterization, Nanoparticles, Photocatalysis, Degradation.

I. INTRODUCTION

Nanotechnology makes it possible to manipulate matter at the molecular level (well below 100 nanometers), providing valuable information for synthesizing new materials with special properties and high reproducibility. increase. In this sense, a significant part of the scientific community is now focused on highly exciting and relevant research areas such as the synthesis of new nanostructured materials that can absorb the sun's photon energy and convert it chemically or electrically [1]. To access new varieties of materials functional with extraordinary properties and application in the basic scientific and technological community, the nano structured materials have been widely explored [2]. In most areas of physics, a chemistry and material science, metal oxides plays a very vital role [3]. Metal oxides are the result of the tendency of metals to coordinate so that the oxide ions form a coordination sphere around the metal ions to form a closed structure. Metal oxides are of great chemical interest because they are very sensitive to changes in composition and structure in many of its physical, magnetic, optical and chemical properties. A better understanding of the chemical composition of the crystal can be found by additional investigation of these relationships. Metal oxides attract the attention of scientists primarily because of their ease of formation and versatility. [3].

Photo-catalytic activity of many metal oxide nanoparticles such as Gd-doped $BiFeO_3$ have been evaluated and proven as efficient photo-catalysts for photo-catalytic degradation of methylene blue, methyl orange, organic pollutant, inhibiting bacteria growth, decomposition of NO_2 and photo-catalytic water splitting, owing to their ability to absorb ultraviolet radiation, due to their sturdy chemical stability, reduced cost, non toxicity and a relatively higher photo-catalytic activity [3].

However, the amount of ultraviolet light they absorb is very small, around 1% to 5%, and it is believed that this can be improved by using a different nanomaterial or by mixing two or more nanomaterials. In the past decade, the study of semiconductor nanoparticles has increased due to their potential applications in photosynthesis, gas sensors, solar cells, UV light emitters, electronic and optical devices, fuel cells, and smart materials [3]. In particular, much attention is paid to its photosynthetic properties in purifying the environment and degrading toxic and organic compounds. The main requirement to improve the photocatalytic activity is to increase the surface area and improve the crystallinity. These requirements are found in materials with a crystalline nanostructure. Several methods have been used to prepare metal oxide nanoparticles, including hydrothermal, sol-gel, chemical vapor deposition (CVD), and hydrolysis. To obtain efficient photocatalytic activity, the nanomaterials must be crystalline; H. must grow at warm temperatures or at very low rates [4].

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The pollution of water and the environment at large by organic synthetic none degradable/decomposable materials such as phenol, methyl orange, rhodamine B has been of great concern causing cancer, skin, respiratory and digestive system diseases [5]. Phenols particularly when ingested affect central nervous system, cause loss of consciousness and collapse in both human and animals. The presences of these substances affect aquatic life since they make water to have a characteristic taste and colour [6]. Therefore, there is need to device a means of degrading or decomposing these substances into none harmful substances by the used of photo-catalyst which were safe for the environment. This research work thus aims at synthesize, characterize and determine photo-catalytic activity of MnO_2/Al_2O_3/Magnetic sand nanocomposite.

II. MATERIALS AND METHODS

Hereby analytical grades of materials only were used. FT-IR Spectrophotometer (Perkin Elmer) in the range of 4000-400cm-1, UV-visible spectrophotometer (Jenway 6405) in the range of 200-600 nm, XRD (Empyrean panalytical model), Scanning electron microscope (SEM, JEM 2100, JEOL, Japan).

2.1 Preparation of Cassia Toraleaf extract

Cassia Tora leaves were collected within the University and was identified in the Department of Botany, Adamawa state University. The aqueous extract used for this experiment was prepared by continuously stirring 10 g of dried leaf pollen with 100 ml of boiled and cooled distilled water on a magnetic stirrer. The dried leaf powder suspensions were soaked in water for 3 hours and treated with Whatman no. 1. The instantaneous filtering method [7] was used.

2.2 Synthesis of MnO_2 Nanoparticles

About 0.2 M aqueous solution of potassium per manganate ($KMnO_4$) was prepared and use for the synthesis of manganese nanoparticles 5 ml of Cassia toraleaves extract was added into 50 ml of aqueous solution taken together with of 0.2 M potassium per manganate ($KMnO_4$) for reduction into MnO2 and kept at room temperature for 30 min [8].

III. RESULTS AND DISCUSSIONS

3.1 UV-Visible spectroscope of MnO_2 nanoparticle

Nanoparticles generally have absorption properties in the UV-visible range and the absorption intensity of nanoparticles generally increases with increasing nanoparticle concentration [9]. In this study, the synthesized MnO_2 nanoparticles showed a characteristic absorption peak at 290 nm as shown in Fig. 1. Which is similar to the one reported by [10].

The characterization of MnO_2 nanoparticle using UV-Visible spectroscope gave a sharp absorption peak at 285 nm.

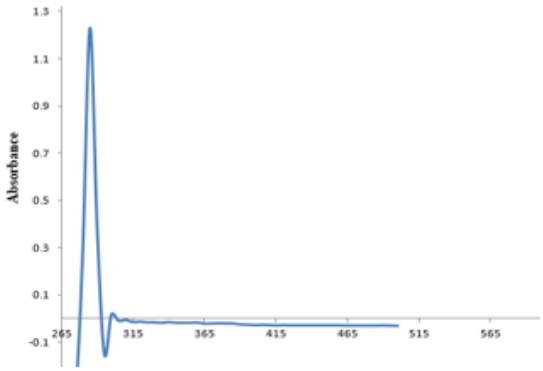

Figure 1: UV-Visible of MnO_2 nanoparticle

3.2 FTIR analysis of MnO_2, Magnetic Sand Composite and Cassia tora leaves

This result is in agreement with that reported by [12]. In magnetic sand composite, the stretching vibration spotted around 774.92 cm^{-1} helps to tell about different forms of silica[13]. Hence, the band at 774.92 cm-1 shows that the silica is in the form of alpha quartz. A sharp band is observed at 727.25 cm -1. A possible explanation for this observation is that the sand in the sample may be contaminated with another mineral, albite, which adsorbs very close to: 727.25 cm-1. Albite has the chemical formula NaAlSi3O8. Geochemical analysis of other study samples reveals traces of Na, Si and Al. This effect lends credence to the interpretation of the band observed at 727.25 cm -1. A sharp peak is observed at 694.74 cm-1. This absorption can be taken to represent the symmetric Si-O resonance curvature [14]. They are band-like at 694 cm-1, 693 cm-1, and 695 cm-1 [14]. Also, the presence or absence of an absorption band at 694.74 cm-1 helps determine whether the observed liquid is crystalline or amorphous. Amorphous silica does not absorb at this frequency, while crystalline silica does [14]. So we conclude that the grains in the sand are better in the crystalline form. The observation of a slight shift in the position of the characteristic peak at 694.74 cm-1 may be due to associated inorganic or crystalline defects. An example of such a binder mineral is the case of kaolin. Without this interference, we may have observed absorption at 694.74 cm^{-1}. The band at 3749.71 cm-1 can be taken to represent the expansion of the crystallized OH hydroxyl. The theoretical crystalline hydroxyl OH of kaolin is 3645 cm -1 [14]. The absorption band at 1002.93 cm-1 can also be attributed to the so-called resonance in kaolin (the main component of kaolin is

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kaolin). 1456.94 cm-1 can be taken as the harmonic band 727.25 cm-1, which represents the absorption band of the suspected albite mineral [14]. Also, FT-IR of Cassia dura leaves showed 7 peaks corresponding to OH; C-H bond stretching, carboxylic acid OH, alkanes C-H stretching, C-O stretching of starch, starch C=O fold, aliphatic amine C-N stretching.

Table 1: FT-IR data of MnO_2

Frequency cm-1	Functional groups
3323.98	O-H
1614.44	C=O
1359.89	C-H
707.45	Mn-O

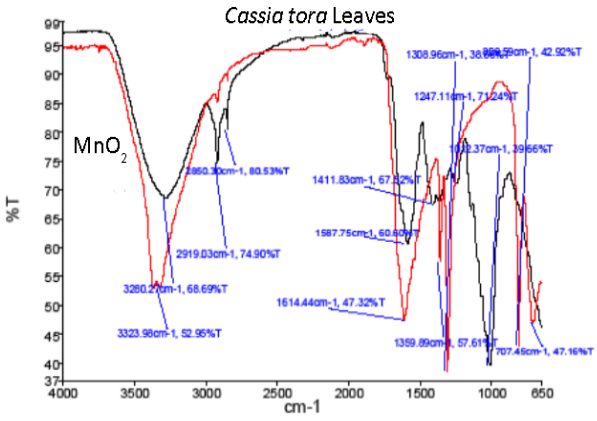

Figure 2: FTIR spectrum of cassia tora leaves and MnO_2

Table 2: FT-IR of Magnetic Sand

Frequency cm-1	Functional groups
3749.71	O-H
1699.83	C-H
1542.17	C=O
1002.93	C-O

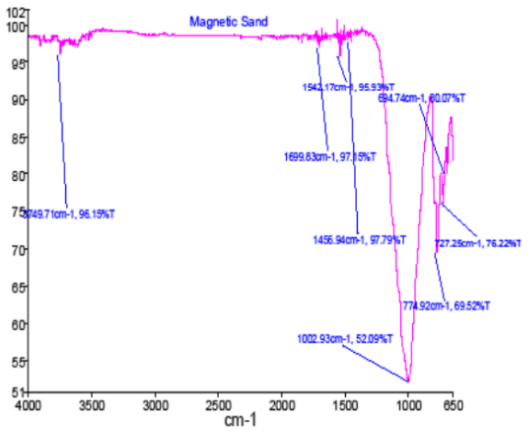

Figure 3: FT-IR spectrum of magnetic sand

3.3 XRD analysis of $MnO_2$and magnetic sand

The result of the XRD analysis of MnO_2 as presented in Table 4.3 shows the crystalline plane at various degree (211,101, 222, 400, 210, 134, 220 and 002) which are well index to pure tetragonal structure according to JCPDS 10799 card. As shown in XRD component such full width high maximum (FWHM), the wavelength (d) in angstrom, and the 2 theta. These values were used to calculate the crystalline size and it can be seen from Figure 4 that the wavelength decreases as angle 2 theta increases.

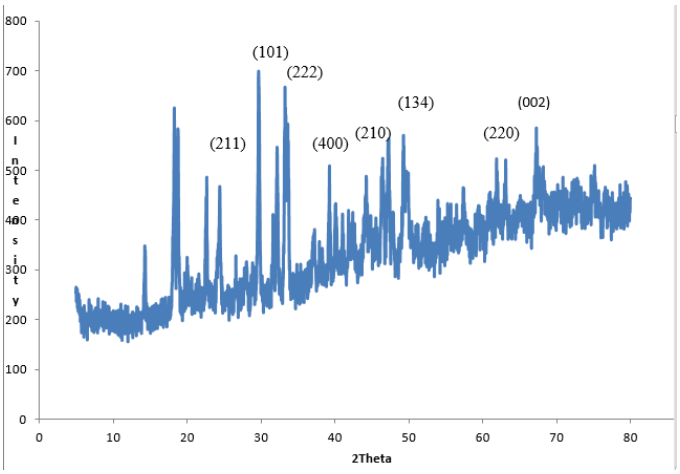

Figure 4: XRD spectrum of MnO_2

Figure 5 presented the XRD of the magnetic sand with full width maximum high (FWHM), wavelength (d) in angstrom and 2 theta that is angle of diffraction. These values were used in computing the size of the magnetic sand. Just like others the angle 2 theta increases with decreasing wavelength.

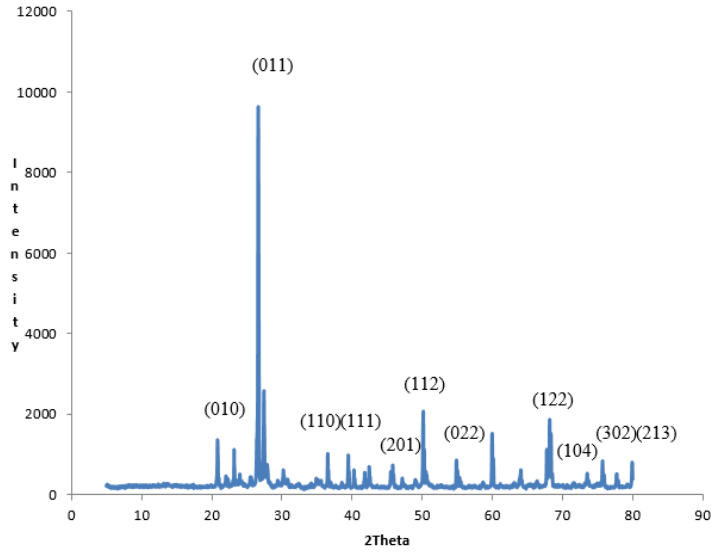

Figure 5: XRD spectrum of magnetic sand

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3.5 The SEM of Manganese Oxide Nanoparticles

The scanning electron microscopic (SEM) analysis of the MnO_2 solution control bioreduction from $KMnO_4$ solution were early distinguishable from the lumps owing to their size differences. This was evident from the SEM image in Figs. 6 to show the accumulation of manganese oxide nanoparticles that occur during the synthesis process.

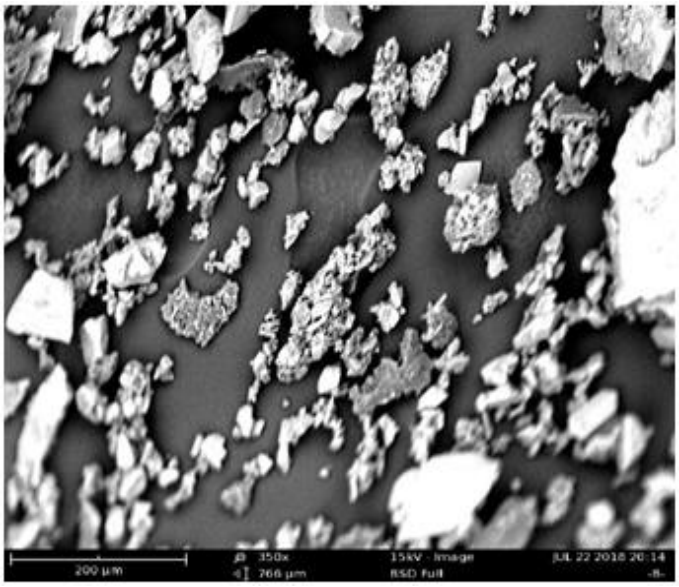

Figure 6: Scanning Electron Microscopy (SEM) of MnO_2

It can be seen that the formed MnNPs are moderately dispersed and little aggregated. SEM images of these compounds clearly showed that most of the particles have a polymorphic material morphology. The SEM picture was very similar overall.

IV. CONCLUSION

The biosynthesis of Manganese oxide nanoparticles were done using aqueous extract of Cassia dura leaves as a biological diluting agent for the reduction of potassium permanganate to manganese oxide, Vis-UV Vis spectrophotometer, FT-IR, SEM, XRD. The results show that the average size of agglomerated manganese oxide nanoparticles is 73 nm. The magnetic sand particles were characterized by UV-Visible, FT-IR, XRF and XRD spectrophotometers. The results show that the sand particle has about 70.16% SiO_2. Lastly, the photo catalytic ability of the nanocomposite was tested using methyl orange as a model and it was found that the nanocomposite was reversible as photocatalyst. This shows that the photo degradation of methyl orange follow a first order kinetic.

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