Research Article

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Synthesis and Characterization of Heterogeneous Catalysts from Magnetic Sand and Kaolin

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ABSTRACT

In this study, magnetic sand and kaolin obtained from the Nigerian states of Adamawa and Bauchi, respectively, were impregnated and studied for the catalytic property of the hybrid material. The SEM micrograph showed pore structures consistent with catalyst materials. X-ray fluorescent data showed the presence of various dopant-like impurities in the sample which act to substitute some parts of essential atoms in the spinel structure, however, not forming another individual phase. X-ray diffraction analysis confirmed crystalline phases of the hybrid material being kaolinite ($Al_2Si_2O_5(OH)_4$), muscovite ($K_4Al_{12}Si_{12}O_{40}$) and quartz, Si_3O_6 minerals with an average crystallite size of 74.60 nm.

1. Introduction

Catalysis is the key to chemical transformations. Most industrial syntheses and nearly all biological reactions require catalysts. Furthermore, catalysis is the most important technology in environmental protection, i. e., the prevention of emissions [1]. A suitable catalyst can enhance the rate of a thermodynamically feasible reaction but cannot change the position of the thermodynamic equilibrium [2].

Heterogeneous catalysis involves systems in which catalyst and reactants form separate physical phases [2]. Heterogeneous catalysts exhibit many advantages, such as environmentally friendly, easy separation, and simple post treatments [3].

Heterogeneous catalysis was first observed by Berzelius and Mitscherlich, who determined that reactions could be fastened by solids. In 1895, the definition of catalysis as the acceleration of chemical reactions by the presence of foreign substances, which are not consumed was proposed by Ostwald. Progress in catalysis is connected with Paul Sabatier who was awarded the 1912 Nobel Prize for his work on the hydrogenation of ethylene and carbon monoxide over Nickel and Cobalt catalysts. No less than 15 Nobel prizes have been awarded for studies on catalysis and many chemists around the world are repeatedly advancing the catalysts they have, and are motivated to discover new ones[10].

Kaolin clay is a cheap and versatile raw material which can be found in numerous geographical locations and has been used successfully in the synthesis of mesoporous aluminosilicates [4] and a host of microporous zeolite frameworks [5-9].

2. Results and Discussion

2.1. Scanning Electron Microscopy

The morphological characteristics of the solid particles as observed by Scanning Electron Microscopy (SEM) are depicted in Figure 1. It can be seen that the material has a layered structure and a two-dimension porous structure with small basal spacing. The layers were clearly kept apart, giving rise to large pore structures as shown in themicograph.

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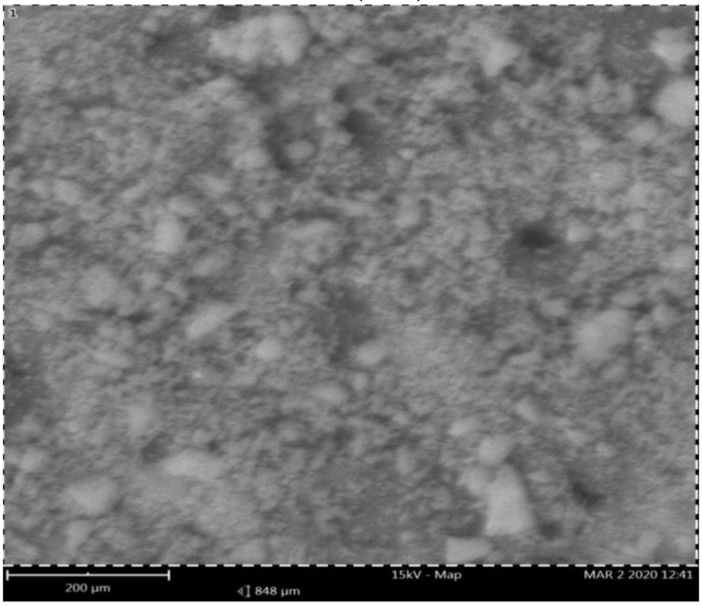

Figure 1: SEM Micrograph of Magnetic Sand/Kaolin

2.2. X-ray Fluorescence

Results of XRF analysis of the Magnetic sand/Kaolin materials as shown in Table 1 made it evident that Si is the major chemical constituent with 23.721% followed closely by alumina with 19.138%, Fe with 7.2852% and Mg with 1.97%. Other elements were found to make up 1.00% by composition of the material, most of which constitute impurities. Of important note is Lead (Pb) and Thorium (Th) with their presence in minute quantity. However, the high level of Si, Al, Fe and Mg may be due to the presence of kaolin and sand. The presence of these impurities would positively alter the catalytic behavior of the material by acting as dopants substituting some part of the Fe and other essential atoms in the structure without forming any other individual phase.

Table 1: XRF Result of Chemical Composition of Magnetic Sand/Kaolin Catalyst

S/No	Elements	Concenteration (%)
1	Fe	7.2852
2	Ni	0.00230
3	Cu	0.00016
4	Zn	0.00814
5	Ga	0.00456
6	Ce	0.0210
7	W	0.0036
8	Ta	0.0256
9	Mg	1.97
10	Al	19.138
11	Si	23.721
12	P	0.0698
13	S	0.0535
14	Cl	0.118
15	K	0.2645
16	Ca	0.1105
17	Ti	0.7878
18	V	0.01405
19	Cr	0.00337
20	Mn	0.01094
21	Ba	-0.0070
22	As	0.00342
23	Br	0.000098
24	Rb	0.00100

25	Sr	0.0561
26	Y	0.001889
27	Zr	0.1063
28	Nb	0.001446
29	Sn	0.030
30	Pb	0.00041
31	Th	0.00030

2.3 X-ray Diffraction

XRD diffractogram of pure Magnetic sand/kaolin showed major mineral constituents in the materials were kaolinite minerals ($Al_2Si_2O_5(OH)_4$) with the presence of Muscovite ($K_4Al_{12}Si_{12}O_{40}$) and quartz, Si_3O_6 (Figure 1). The XRD patterns at 2θ = 5º - 75º reveals the nature of Magnetic sand + Kaolin which exhibits well resolved and intense narrow peaks appeared at 27º, followed by 25º, 12.5º while less intense peaks were found at 9º, 20º, 20.5º, 21º, 21.5º, 45º, 31º, 35º, 36º, 36.6º, 37.8º, 38.5º, 39.6º, 40.3º, 41.2º, 45.5º, 45.8º, 48.1º, 49.6º, 50.1º, 51.1º, 55º, 55.5º, 56.9º, 60º, 61.3º, 64.2º, 65.3º, 68º, 68.5º, 68.7º, 70.4º, 72.5º and 73.5º. Kaolinite is the major part of the mineral. X-ray diffraction exposed the characteristics of the materials more clearly due to the different arrangements by the minerals. The presence of Kaolinite may be derived from Kaolin source. By investigating the most intense peak, the crystallite size was calculated from its line broadening and the average crystallite size was gotten to be 74.60 nm.

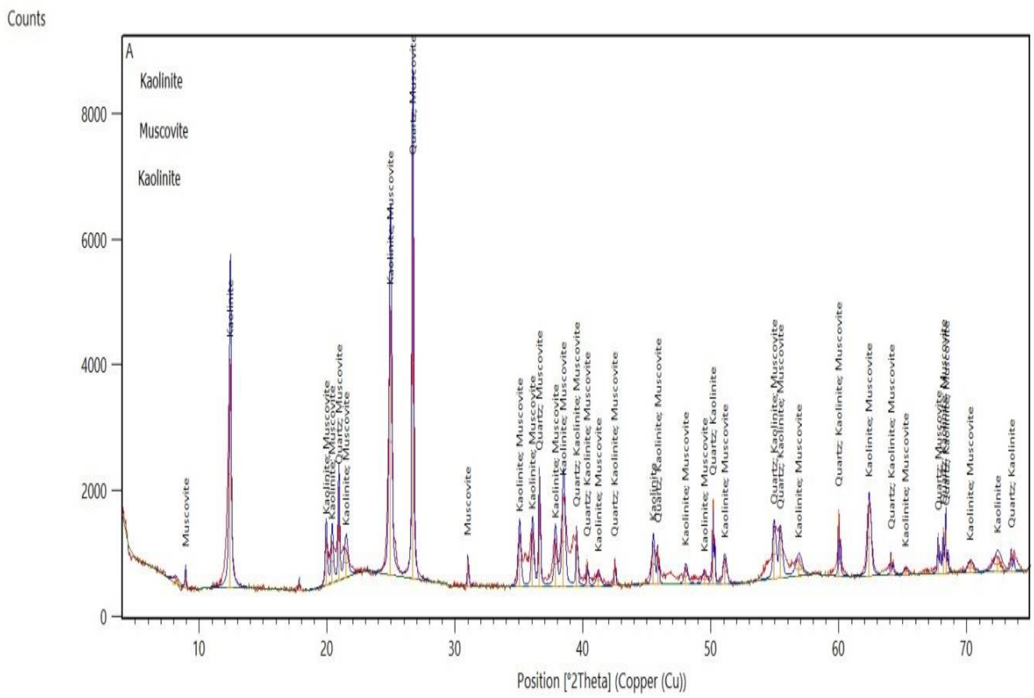

Figure 2. XRD spectrum of Magnetic sand/Kaolin

3. Experimental

3.1. General

Magnetic sand used for this work was obtained directly from its deposit in Bazza Mountain, Michika LGA of Adamawa State, Nigeria with the aid of a magnet bar and packed into containers. 500g of Kaolin was collected from Alkeleri LGA of Bauchi State, Nigeria.
Co-impregnation of the magnetic sand and kaolin active components was done in a single step.

3.2. Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM), serial number MVE01570775 and model number 800-07334, manufactured by PRO: X: Phenonm World, was used to examine the surface morphology of the catalyst.

3.3. X-ray Fluorescence

X-ray florescence was deployed to ascertain the chemical composition of the catalyst materials. XRF was carried out on a standard method EDXRF analyzer using monochromatic radiation with K-Alpha1 wavelength 1.540598 Å by a linear analysis technique.

The catalyst samples were prepared for analysis by crushing using a Tema vibrating mill before sieving with a 63 microns sieve to give a uniform particle size. 10 g of the powdered samples were dried in an oven at 110°C for 24 hours before major elemental analysis in the computerized XRF and the condition for trace elemental analysis set to give the result in elemental form.

3.4. X-ray diffractometer (XRD)

X-ray diffractometer (XRD) was employed in phase identification of the catalyst, using an EMPYREAN diffractometer system. Finely ground materials were homogenized, average bulk composition determined and 1 g of the powdered sample was compressed in the sample holder before being placed in the XRD cabinet. The data was collected from 4° to 75° 2θ at tube current of 40mA and 45VA tension. Fixed Divergent Slit size of 1° was used and the goinometer radius was 240 mm.

4. Conclusion

In this research, Magnetic sand has been impregnated with Kaolin by a simple, cheap and innovative method, and studied for the catalyst properties of the resultant hybrid material. This catalyst is being developed for application in the production of biodiesel by transesterification of seed oils. The material characterization, done by SEM, XRF and XRD analysis indicated that the material possesses a large, layered pore structure with small basal spacing characteristic of heterogeneous catalysts. In addition, the presence of a number of dopant-like impurities was confirmed by analysis. The presence of these constituents in the material essentially makes a case for the catalyst potential of the material because they act to substitute some parts of essential atoms in the spinel structure, without forming their own individual phase. The results of this study are in tandem with earlier investigations done by many researchers and with international standards. From these findings and the comparison with previous works done by earlier researchers such as Tukur *et al*. [11] who studied the potential of magnetic sand for the transesterification of Luffa seed oil, it can be deduced that the hybrid material of magnetic sand and kaolin will make a very useful application as a heterogeneous catalyst for several chemical processes such as the production of biodiesel by transesterification of various seed oils.

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