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ANALYSIS OF THE PHYSICAL, MECHANICAL, AND THERMAL PROPERTIES OF A CLAY-SUPPORTED-METAL OXIDE NANOCOMPOSITE

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Abstract
Nanotechnology encompasses the development of material structures at very small scales to achieve specific properties, through which they can strengthen the effectiveness of materials, while, being lightweight, more durable, reactive, and entangled. It involves manipulation of matter at the atomic scale. This study develops and analyzes the physical, mechanical and thermal properties of a clay-supported-metal oxide nanocomposite. The metal oxides used were CuO and ZnO metal oxides with bentonite as the supported clay. CuO and ZnO nanoparticles were synthesized by green synthesis using *bitter* leaf extract as the capping and reducing agent. The bentonite clay was purified, functionalized, and calcined. The prepared materials were mixed in certain proportions and molded to obtain a bentonite/CuO/ZnO Nanocomposite. The bulk density of the composite was determined using an experimental method, followed by mechanical tests such as impact tests using the Izod method, tensile strength, percent elongation at break, tensile strength at break, and yield strength on the composite using the ASTM E384 standard method. The bentonite/CuO/ZnO/ nanocomposite was also characterized using SEM/EDS, and XRD. SEM/ EDS showed that the bentonite/CuO/ZnO hybrid system obtained has a plate structure and was characterized by a large grain size distribution.

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1. Introduction

Nanotechnology has great importance in improving products, treating diseases, and serving humanity in all aspects of life. In addition, it provides great hope for future scientific revolutions in physics, chemistry, biology, material engineering, and others. (Duncan, 2011). Nanocomposite materials have advanced properties that are useful for different applications. These nanocomposites exhibit enhanced thermoelectric properties, making them attractive for applications in thermal energy storage. Composite materials are prepared from a combination of two or more different materials with distinct chemical or physical characteristics. The result of the addition of nanoparticles is a drastic improvement in properties that can include mechanical strength, toughness, and eletrical or thermal conductivity. Nanomaterials can be nanoscale in one, two, or three dimensions (Kargozar and Mozafari, 2018). Metal oxide-supported clays and clay minerals, including pillared clay, are composite materials used in applications such as catalysis and adsorption, as well as for many other functional purposes. Clays are silica alumina minerals with layered structures composed of aluminium silicates that are formed by tetrahedral and octahedral sheets, where the layers possess net negative charges and contain cations, such as Na^+, K^+, Ca^+, etc., which occupy the interlamellar space. The amenability of clays for modification lies in the exchangeable properties of the cations; as such, they can easily be replaced by other cations or other molecules. Many advantages are designed and obtained by improving the physicochemical properties of these materials from those of their raw forms for applications such as high surface area, chemical stability, and enhancement of specific characteristics related to homogeneous distributions (Qiu et al, 2020; Mody et al, 2012). The modification of a metal oxide photocatalyst into a heterostructure and composite is an important strategy for developing photocatalytic activity (Khlebtsov and Dukman, 201I). In recent times, nanoclay has initiated more concentration because of its distinctive physicochemical properties and characteristics. Clay-based nanocomposite can overcome the restrictions of individual clay and other constituents such as low selectivity, pH dependency, small specific surface area, and low water wet ability. Clay-based nanocomposite can be used as a cost-effective and novel adsorbent for the adsorption of organic contaminants from water and wastewater. Nanoclay materials are the best minerals for the synthesis of novel materials and afford a cost-effective sustainable solution to various industries for health and environmental safety. Based on their developed routines, clay/polymer nanocomposite exhibited the capacity to remove various contaminants such as metal ions, microorganisms, phenols, pesticides, pigments, and dyes from water and wastewater (Khan and Saeed, 2019). Compared with conventional composites, polymer nanocomposites exhibit remarkable enhancement in their properties when a very small amount of fillers is added to them. Nanoclay hybrid composites have a wide range of consumer applications in food packaging and in biomedical applications (He et al, 2019; Rovera et al, 2020; Mageswari, 2016). Nanoclays are nanoparticles of layered mineral silicates, which present anisotropic plate-like structures approximately 1 nm thick and 100 nm in diameter. The main types of clay nanoparticles incorporated into hydrogel nanocomposites include montmorillonite (MMT), bentonite, and other silicate clays. Nanoclays can general be found in abundance in nature. Nanoclays are phyllosilicate minerals with a platelet-like shape, flaky soft structure, low specific gravity, and high speed ratio. Some physical, chemical, and mechanical properties are greatly enhanced when clay components are included in a polymer matrix (Martinez et al,2016; Andrieviski, 2014; Wu et al). Today's major nanocomposite food packaging is made from nanomaterials such as silver nanoparticles, zinc oxide and clay. Nanocomposites have been growing at a rapid rate because of their large number of applications. In the next 10 years, worldwide production will exceed 600,000 tons in different areas of engineering applications (Melaine et al, 2017). Nanocomposites have also attracted the field of automotive and industrial applications by enhancing their mechanical properties. They can be used or applied in various engineering applications. Various extensive research activities are being undertaken by researchers in the field of nanotechnology to examine the functions and applications of nanomaterials. Diallo et al, (2016) studied the physical and enhanced photocatalytic properties of green synthesized SnO_2 nanoparticles via asphalathus linearis. Shah et al, (2015) also examined the properties of green synthesis of metallic nanoparticles via biological entities. Sun et al, (2017) reviewed the applications of nanoparticles in enhanced oil recovery. He et al, (2019) also comprehensively reviewed the current applications of nanotechnology in food and agriculture. Rovera et al, (2020) studied the effect of nanoinspired oxygen barrier coatings for food packaging applications. Afroza et, (2019) analyzed the physical, mechanical, thermal, and morphological properties of date palm mat (DPM) and palmyra palm fruit (PPF) fiber-reinforced high-density polyethylene hybrid composites. This study explored the mechanical, physical, and thermal properties of composite materials obtained from clay-supported metal oxide nanoparticle in more detail.

2.0 MATERIALS AND METHOD

2.1 EXPERIMENTAL PROCEDURE

A green synthesis approach was used to synthesize the CuO and ZnO nanoparticles because of its ecofriendly, cheap and energy saving properties. Bentonite, was purified, functionalized and calcined. The materials were then mixed in certain proportions to form a bentonite/CuO/ZnO nanocomposite. The following reagents were used: Copper sulfate pentahydrate ($CuSO_4$. $5H_20$), Zinc Nitrate, Sodium Hydroxide (NaoH), Bitter leaf extract (*vernonia amygdalina*), and distilled water.

Purification and Functionalization of Bentonite Clay

Bentonite was ground into powder. To purify it, some grams were dissolved in 1L of deionized water, and stirred for 6 h continually. The sediment was discarded, and then centrifuged at 5000 rev/min for 30 min. The bentonite obtained with 1L of 1M NaOH solution was dissolved for 12 h to exchange the interlayer cations in the bentonite. It was centrifuged, rinsed with deionized water, and dried at 600^0C. The dried bentonite was calcined at 200^0C for 1h.

Bitter leaf (*Vernonia amygdalina*) collection, extraction and preparation

Bitter leaves (*Vernonia amygdalina)* were obtained from the rural area of Bayelsa, Nigeria. The leaves were washed twice with distilled water and dried under room temperature in a dust-free environment 2 weeks. The dried leaves were pounded into small pieces using a mortar and pestle and stored in a dry container. Bitter leaf was chosen because of its abundance and easy availability and it serves as a reducing and capping agent. The leaves (10g) were then weighed using an electric weighing balance into a beaker. 500ml of water was measured and added. The mixture was stirred in a water bath kept at 60^0C for duration of 2 h. The mixture was removed and allowed to cool, after which it was filtered using whatmann filter paper and then stored in the refrigerator at 4^0c for further use. it serves as a reducing and capping agent.

Synthesis of Copper oxide nanoparticles (CuONPs)

A measured quantity (1.498g) of Copper sulfate pentahydrate ($CuSO_4$. $5H_2O$) was dissolved in 20ml of distilled water in a beaker. The solution was kept in a magnetic stirrer set at 30^0C for duration of 3hours after which a blue coloration was observed. 50ml of the bitter leaf extracts was added to the copper solution that was already on the stirrer. A color change was observed. After stirring for duration of 30 min-1hour, a dust-like particle was observed under the beaker, indicating the formation of copper oxide nanoparticles. Figure 1 depicts the synthesized copper oxide nanoparticles.

Figure 1: Synthesized Copper oxide nanoparticles

Synthesis of Zinc oxide nanoparticles (ZnONPs)

A measured quantity (7.4372g) of zinc nitrate was dissolved in 250ml of distilled water in a beaker. The solution was kept in a magnetic stirrer set at 60^0C and stirred for 30 min. 75ml of the extract was added in dropwise manner to the zinc nitrate solution that was on the magnetic stirrer. A colour change was observed. The colour changed from oil/gold to golden brown. While the solution was stirring, a few drops of 1M NaoH solution were added, after which a whitish cloudy appearance that shows the formation of zinc oxide nanoparticles was formed. The stirring was prolonged for duration of 1h.

Figure 2: Synthesized Zinc oxide nanoparticles

Composites Preparation

Five samples of the Composite were prepared by compression molding. The mixture was weighed at certain ratios, as shown in Table 1, and thoroughly mixed in a blender. The mixture was poured into the mold (15 x 15 cm^2) and finally hot pressed at 150^{o}C for 5 min between two steel plates under a pressure of 100 KN. The composite was cooled for five min at room temperature and 100 KN pressure. The finished composite was then released from the mold.

Table 1: Percentage Composition of the Samples

Sample	CUONPs % composition	ZnONPs % composition	Bentonite % composition	Total % composition
A	47.5	47.5	5	100
B	45.0	45.0	10	100
C	42.5	42.5	15	100
D	40.0	40.0	20	100
E	37.5	37.5	25	100
F	35.0	35.0	30	100

Determination of Bulk Density of Composites

The bulk density of the composites was determined with the aid of Eq. (1) and in line with the method used by (Afroza et al, 2019).

$$Z = \frac{W_c}{L \times B \times H} \quad (1)$$

Where Z ($\frac{g}{cm^3}$), W (g), L (cm), B (cm), and H (cm) are the bulk density, weight, length, breadth and height of the composite, respectively.

Mechanical Tests

(i)Tensile Strength Test

A tensile strength test was carried out to determine the tensile strength, percent elongation at break, tensile strength at break, and yield strength of each sample of the composite.

(ii) Hardness Test

The ASTM E384 standard was used for hardness test, and specimens are grinded and polished mechanically. The grinding is made by emery paper (320, 500, 1000 μm) in size, while the polishing is made by using wool cloth and alumina (1μm) in size. Microscope equipment allows for precise indentation placement. In this test, the hardness of the specimens was determined by measuring accurately with the aid of an MMT-X7A Microhardness tester. Small indentations were produced by the application of 200 N for 20 s. To obtain reliable statistical data, analysis points were spaced to eliminate the effect of near indentations. Three indented samples were evaluated, and their averages were calculated. The average values for the five samples were reported.

(iii) Impact test on the composite

Impact tests were also carried out on the composite using an Izod 9000 series impact test machine. This machine is designed to perform Izod impact tests on various samples, from bars/dumbbells to pipes, in accordance with specific standards. To perform this test, the sample is placed into a holding fixture with the geometry and orientation determined by the type of test. An object with a known weight and height is released to impact a sudden force on the specimen. This sudden impact helps to determine the actual behavior (toughness or brittleness) of the specimen. Tougher materials have higher impact strengths whereas, brittle materials have low impact strengths.

Determination of the thermal conductivity of the composite

Relative method of longitudinal heat flux is used to determine the thermal insulation conductivity of the composite, which involves comparing the heat flux through a sample placed between a heater and a standard in a stationary thermal mode. The instrument consists of a homogenous carrier, a temperature sensor to measure differences in the surface, and several temperature sensors to determine the surface temperature. The thermal flux (ϕ) and the temperature difference between the surfaces of the test plates (ΔT) were measured to determine the thermal insulation conductivity (γ) of the composite using Eq. (1), as used by Schmidt et al, (2023):

$$\gamma = \frac{p_{sd}}{A\{T_1 - T_2\}} \quad (1)$$

Where γ ($\frac{W}{m\,K}$), p_s (watt), T_1(K) , T_2(K), and A (m^2) are the thermal conductivity, power supplied as heat flux to the heating plate, average temperature on the warm side of the specimen, and average temperature on the cold side of the specimen, respectively.

SEM/EDS analysis

The nanocomposite was characterized using SEM/EDS and XRD. SEM/EDS is a combined technique that uses a scanning electron microscope and energy-dispersive X-ray spectroscopy to analyze materials. SEM provides the imaging component, whereas EDS is used for detection. While traditional microscopy uses light to create an optical signal, scanning electron microscopy uses electrons. EDS is then used for detection and analysis. The surface morphology of the composites was examined using a field emission scanning electron microscope. SEM analysis was performed on the fracture surfaces of the tensile specimens.

3.0 RESULT AND DISCUSSION

Mechanical Properties

The composite possesses better mechanical properties due to the presence of clay as compared to Copper-Zinc alloy. The maximum tensile strength was 14.25 MPa because of better fiber-matrix interfacial adhesion. However, the tensile strength gradually decreased for the composite after . This decreased trend in the tensile strength of the composites may be attributed to low fiber-matrix adhesion between fiber and HDPE with increasing fiber content in the composite. The elongation at break of the hybrid composites for various wt% of fiber content is shown in Figure 5. 100% HDPE presents the highest value of elongation at break. The elongation at break of the composites gradually reduced with the increase of fiber content from the composites.

Thermal Property

The result obtained showed that the presence of clay in the composite further reduced the mobility of the electrons, resulting in lower conductivity of the composite as compared to Copper-Zinc alloy.

Physical Property

The effect of clay content on the bulk density of the composite is very high. It increased the bulk density of the composite.

SEM/EDS

SEM micrographs showed that bentonite is a raw material with a layered structure and has a large grain size distribution. The bentonite/ZnO/CuO hybrid system obtained has a plate structure and was characterized by a large grain size distribution, as depicted in Figure 1.

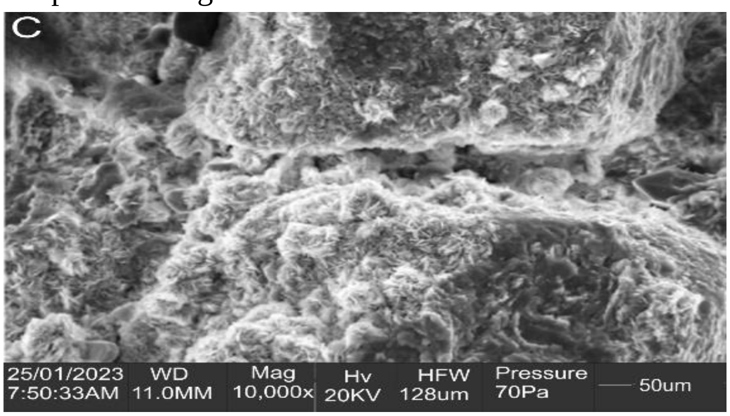

Figure 3: SEM results for bentonite/cuo/zno nanocomposites

XRD

The XRD diffratogram of zinc oxide nanoparticles, copper oxide nanoparticles and bentonite /CuO/ZnO composite are as shown in figures 4, 5, and 6, respectively. Figure 7 also depicts the EDS analysis of the composite.

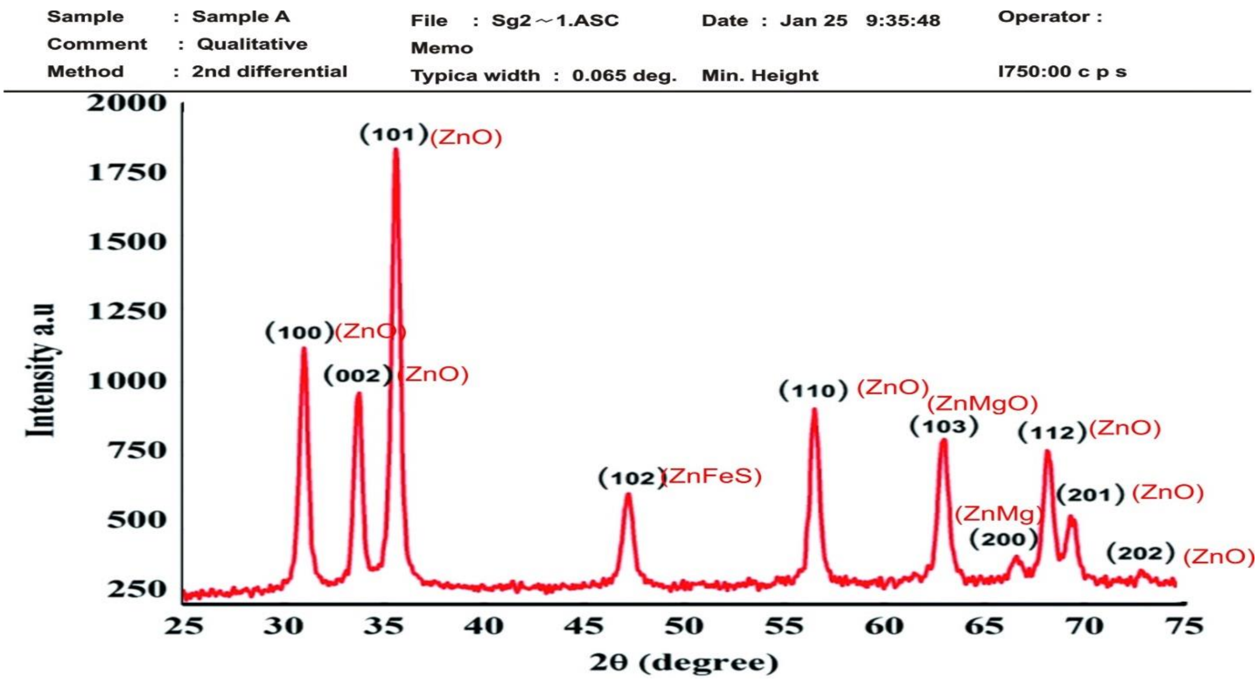

Figure 4: XRD diffratogram of zinc oxide nanoparticles

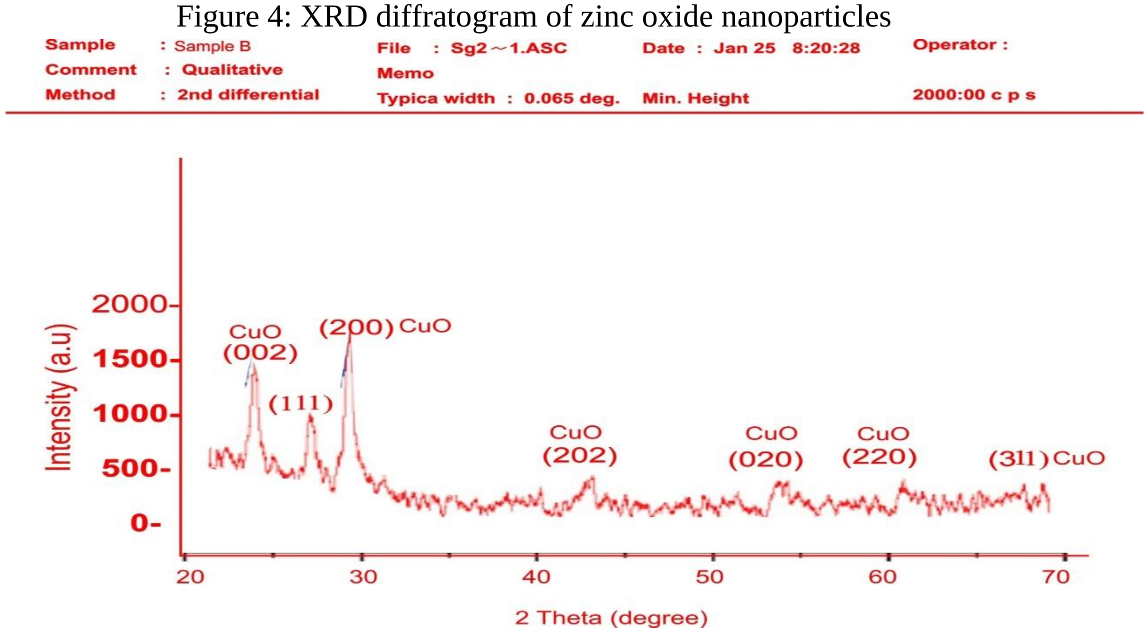

Figure 5: XRD diffractogram of copper oxide nanoparticles

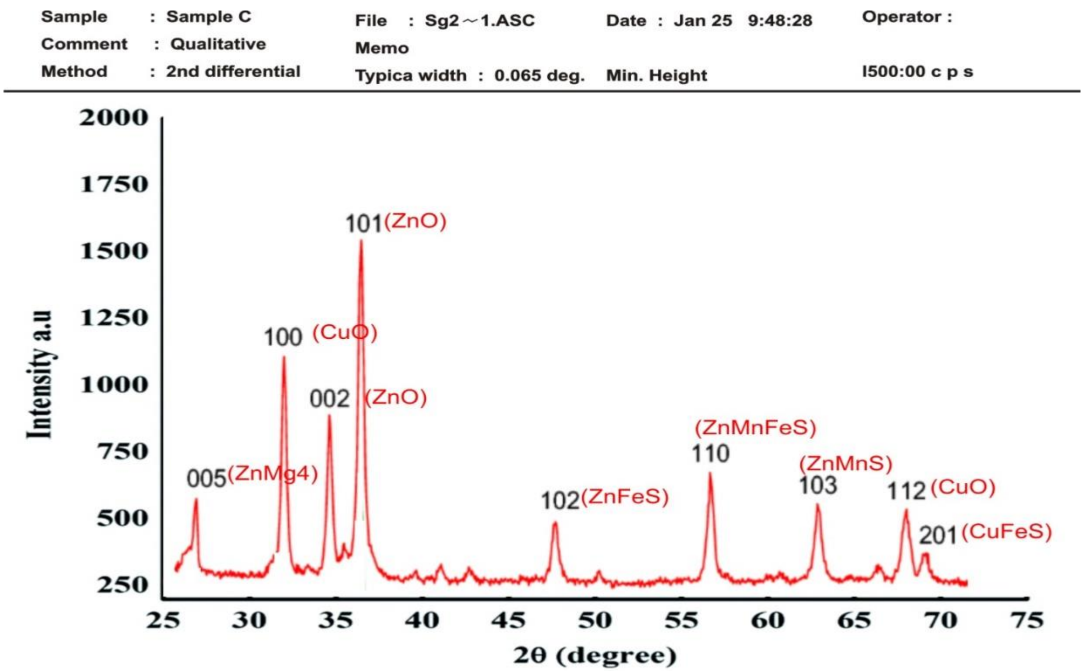

Figure 6: XRD diffractogram of the bentonite/cuo/zno nanocomposite

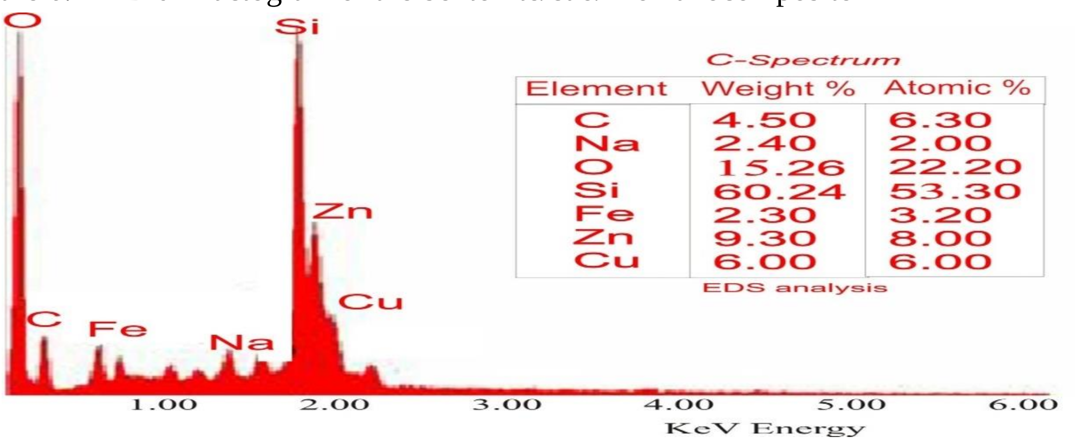

Figure 7: EDS analysis of the bentonite/CuO/ZnO nanocomposite

4.0 CONCLUSSION

From the SEM/EDS and XRD results, the performance characteristics of the bentonite clay- supported metal oxide nanocomposite were analyzed and evaluated. The performance characteristics evaluated for the specimens considered are; plate structure, large grain size distribution, crystalline structure etc. These performance characteristics are attributed to the unique strength, wear resistance, hardness, semiconducting, photo catalytic property, etc. of the bentonite/CuO/ZnO nanocomposites. The bentonite/CuO/ZnO can serve as an additive or reinforcement to a matrix to increase the strength, hardness, wear resistance, photocatalytic activity, semi conductivity, etc. of the matrix.

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