

# Instrumentation Techniques for Nanocomposite Characterization

Mashal Alenazi

University of Bridgeport, Biomedical Engineering, USA

**Abstract:** Improved citrate auto combustion preparation technique is used to synthesis ferromagnetic and ferroelectric phases of the investigated nanocomposite. Atomic force microscope (AFM) is one of the important tools for imaging, measuring, and manipulating matter at the nanoscale. The characterization was performed by elemental analysis, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), physisorption of N<sub>2</sub>, scanning electron microscopy (SEM) and Z potential measurements. The characterization and adsorption experiments showed that the extent of pesticide adsorption was markedly subjected to the structure and features of the surface of each organo-clay and also to the nature of the considered pesticide.

**Keywords:** Nanocomposite, Characterization, Quantities

## I. INTRODUCTION

A sum property of a composite is the weighted sum of the contributions from the constituent phases that is proportional to the volume or weight fractions of these phases. Physical quantities such as density and resistivity are examples of sum properties. For product properties, consider a composite material with two component phases.

The first phase has a property  $A \rightarrow B$  with a proportionality tensor  $dB/dA = X$ ; and the second phase has a property  $B \rightarrow C$  with a proportionality tensor  $dC/dB = Y$ . Then the composite will have the property  $A \rightarrow C$  with a proportionality tensor  $dC/dA = Z$ , where  $Z = Y \cdot X$ ; hence, the name "product property". The product property is achieved in a composite but not seen in the individual phases. The importance of these materials lies on their peculiar properties such as small particle size, swelling capacity, anisotropic shape, reactive surfaces, and high cation exchange capacity (CEC) [1] and [2]. As a consequence, a vast number of applications have been projected for clay minerals; among others, their use as adsorbents of pollutants in order to reduce their environmental impact [3]. The interest in clay minerals has grown as the development of nanoscience and nanotechnology has progressed, due to their nano-sized layers and interlayer space [4], [5] and [6].

Expandable clay minerals (i.e., smectites) have a marked hydrophilic character caused by the strong hydration of the inorganic counter ions present in the interlayer space. The mechanical properties and the fracture mode are dependent on the strength of grain boundary. In the present work, the designed microstructure for ASs nanocomposite is shown in Fig. 1(a); nano-scale SiC particles are located both within the alumina matrix grains and at the Al<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> grain boundary. Owing to the large applicability of Boron in increasing thermal stability, the effect of Boron incorporation on novel Zr–W–N developed by our group has been studied.

The developed Zr–W–N coatings showed remarkable combination of high hardness and wear resistance but exhibited low thermal stability. The effect of Boron incorporation on thermal stability and mechanical properties of Zr–W–N has been studied. Nanocomposite Zr–W–B–N thin films deposited on Si (100) substrate have been investigated in detail as a function of varying deposition parameters to substantiate the influence of microstructures on its mechanical properties. In order to ensure compatibility and stability of the sputter deposited Zr–W–B–N thin films in tribological applications, the present work has been focused to investigate the DC/RF magnetron sputtered nanocomposite Zr–W–B–N thin films with varying B content and hence varying microstructure. The films were characterized by XRD, FE-SEM, AFM, Raman and TEM to reveal the influence of processing parameters on microstructural characteristics and the atomic percentage of B in the Zr–W–B–N thin films were measured using Wavelength Dispersive Spectroscopy (WDS) attached with EPMA.

The hydroxyl-ending alkyl group thus introduced onto the surface of ZnS NCs enhances their dispersity in solvent, allowing the particle size of NCs to be controlled. We have found that the particle size of the ZnS NC characterized by TEM is about 2.6 nm, in agreement with the calculated data from UV-vis absorption spectra according to Brus's model and Debye–Scherrer formula. The surface morphology of the TiAlN/MoN multilayer coatings with different bilayer thickness is shown in Fig. 4a–c, a major concern for TiAlN/MoN multilayer coatings deposited by arc ion plating is the macro-particle (MP) formation, and this phenomenon is due to the evaporation of the metal macro-particles. In plasma environment there are three metal ions Al<sup>+</sup>, Ti<sup>+</sup> and Mo<sup>+</sup> that react with nitrogen. It is believed that very fast evaporation during the cathodic arc process produces excess atoms that are not completely ionized before they arrive at the substrate surface. These excess neutral atoms may combine to form

macro particles during the flight and other reason may be during arc evaporation process if cathode spot stays at an evaporative point for too long it can eject a large amount of macro-particles or droplets. These polymers show the advantages of controlled drug delivery and thermal therapy. There are number of reports for synthesis of magnetic microcapsules in which magnetic nanoparticles often aggregate. Previously we have reported a number of systems as candidates for drug delivery platforms. Recently, drug delivery have attracted a lot of attentions for their advantages of dosage and poisoning. Polymeric carbohydrates are the most promising materials to fabricate nanostructure compounds for drug delivery for their excellent qualities. Carboxymethyl cellulose and other cellulose-based hydrogels are used as a protein carrier. Carboxymethyl celluloses are very important derivatives of cellulose with both carboxylate and hydroxyl groups is considered as green, natural, inexpensive polysugar, stable, non-toxic, biodegradable and biocompatible.

Carboxymethyl cellulose is applied for drug delivery systems for controlled drug release. Magnetic nanomaterials have been applied for magnetic guidance of particle systems in drug delivery. Koneracka et al] reported the magnetic properties of encapsulated magnetite in PLGA (poly D,L/lactide-co-glycolide acid) nanospheres. Butun et al. reported one-step fabrication of biocompatible carboxymethyl cellulose polymeric particles for drug delivery systems. The aim of this study is the synthesis and characterization of a biocompatible magnetite-carboxymethyl cellulose nanocomposite for potential application in drug delivery. We have used a modified co-precipitation method for magnetite casting into a carboxymethyl cellulose (CMC) matrix. In this study, the magnetite-carboxymethyl cellulose nanocomposites were synthesized by a modified co-precipitation polymerization for potential application in drug delivery. The structural characteristics of the particles were examined by Fourier transformed infrared (FTIR), X-ray powder diffraction (XRD), transmission electron microscopy (TEM) and field emission scanning electron microscopy (FESEM) analysis.

## II. CHEMICALS AND SYNTHESIS

The mixture was heated under nitrogen atmospheres at 55 C after degassing using six alternating cycles of evacuation and pressurization with high purity nitrogen (starting with evacuation). Polymerization carried out after the injection of a CoBF/MAA mixture (7.5 mg CoBF dissolved in 74.0 g MAA). The reaction was allowed to proceed for 2 h with continuous stirring under nitrogen atmosphere and then quenched with an ice-water bath. The as-prepared PMAA was precipitated in diethyl ether, and then redissolved in DI water and reprecipitated in diethyl ether for several times. UV-vis absorption spectra were taken with a Perkin-Elmer Lambda 900 UV-vis spectrometer with the scan rang from 260 to 450 nm using DMF as solvent. The powder X-ray diffraction (XRD)

patterns were conducted on a Bruker-AXS D8 ADVANCE X-ray Diffractometer at a scanning rate of 6°/min in 2 $\theta$  ranging from 10° to 80° with CuK $\alpha$  radiation ( $\lambda=0.1542$  nm). Fourier transform infrared (FT-IR) spectra were recorded on a NICOLET-NEXUS 670 spectrometer. The samples were grounded with KBr crystal and the mixture of them was pressed into a flake for IR measurement.

Ferrous chloride tetrahydrate, ferric chloride hexahydrate (Merck) was used as precursors. An aqueous mixture of ferric and ferrous solution were dropped into and ammonium hydroxide solution (1.5 mol/L) at room temperature under mechanical stirring in a three necked flask. Dried nitrogen gas was inflated during synthesis in a closed system to prevent the oxidization of ferrous ions. The color of the solution changed from light brown to black, indicating the formation of magnetite nanoparticle, which was permitted to crystallize. The precipitate magnetite nanoparticle was washed by repeated cycles of centrifugation and re-dispersion in distilled water. The precipitate magnetite nanoparticles were dried in a vacuum oven at room temperature.

The reference Ca-rich Arizona montmorillonite (SAz-1) from the Source Clays Repository of the Clay Minerals Society (Purdue University, West Lafayette, IN), with CEC equal to 1200 mmol/kg, was used for the preparation of the organo-clays. The properties of SAz-1 montmorillonite have been reported elsewhere.

Hexadimethrine (HEXAD) bromide (purity > 95%) and hexadecyltrimethylammonium (HDTMA) chloride (purity  $\geq$  98%) (Fig. S1) were purchased from Sigma - Aldrich. Hexadimethrine is an agglutinant blood cell polycation employed as heparin-neutralizing in pharmaceutical industry. Hexadecyltrimethylammonium is a cationic surfactant which has been widely used, among other applications, for the preparation of organo-clays [3].

The notion that the era of nanomedicines started in the previous century is demonstrated when looking at the development of nanocrystal technology, discussed by Eugene Cooper. Rather simple wet milling techniques led to a decrease of the crystal size down to the hundred nanometer range. These dispersions are stabilized with surface active polymers. The oral bioavailability of nanocrystal based formulations of poorly water-soluble drugs has been clearly enhanced for some drugs. Poorly water-soluble nanocrystals were also used for parenteral injection of imaging material for CT scans. The 'nanocrystal story' clearly shows that rather simple techniques can improve the performance of poorly water-soluble drugs dramatically, although the number of such drugs that are marketed is limited at this point. Recently, much attention has been focused on nanoemulsions and self-emulsified drug delivery systems (SEDDS) to improve the oral bioavailability of poorly water-soluble drugs. Nanoemulsions are non-equilibrium, heterogeneous systems consisting of two immiscible liquids in which one liquid is dispersed as droplets within the other liquid [58]. Self-nanoemulsified drug delivery systems are isotropic mixtures of oil, surfactant, co-surfactant and drug that form fine oil-in-water (o/w) nanoemulsions when

introduced into aqueous phases under gentle agitation. They are stabilized by an interfacial film of surfactant molecules with a droplet size typically less than 100 nm, which guarantees efficient absorption of oil droplets. These systems improve the oral bioavailability of poorly-water soluble drugs by different mechanisms including improved drug solubilisation and protection against physicochemical and enzymatic degradation. The skin is an organ where different nanotechnological approaches are being tested, which GregorCevc reviewed. Microneedles with different shapes and sizes, hollow or solid, biodegradable or non-biodegradable attract a lot of attention. In the case of ballistic injection systems, nanometer sized hard or soft (droplets) material is shot into the skin for intradermal delivery.

### III. CONCLUSION

The molecular basis of the GI drug barrier has been found mainly due to the overexpression of the multidrug efflux pump proteins, P-glycoproteins (P-gp) in the epithelial cell membrane. Additionally, carriers in the particulate form should be able to diffuse further into the mucus layer enabling them to reach the cells of the epithelial layer. However, due to the lack of miscibility between most of the insoluble drugs and commonly used hydrophilic polymers, drug tends to crystallize out of the initially homogeneous drug/polymer solution during storage, especially at high drug loading and/or when exposed to moisture. The morphology of the samples was observed using a low vacuum scanning electron microscope (LV-SEM, JEM-100S) equipped with an energy dispersive X-ray (EDX) detector. Transmission electron microscopy (TEM) was performed using a JEOL4010 electron microscope where the sample ultrasonically dispersed in acetone and then collected on a carbon grid. The atomic force microscope (AFM) was performed as it is a suitable tool for characterizing nanoparticles

### REFERENCES

- [1] Microbial extracellular polymeric substances: characterization, structure and function. Springer Science & Business Media.
- [2] Hajji, P., David, L., Gerard, J. F., Pascault, J. P., & Vigier, G. (1999). Synthesis, structure, and morphology of polymer-silica hybrid nanocomposites based on hydroxyethyl methacrylate. *Journal of Polymer Science Part B: Polymer Physics*, 37(22), 3172-3187.
- [3] Burnside, S. D., & Giannelis, E. P. (1995). Synthesis and properties of new poly (dimethylsiloxane) nanocomposites. *Chemistry of materials*, 7(9), 1597-1600
- [4] F. Bergaya, G. Lagaly General introduction: clays, clay minerals and clay science F. Bergaya, B. Theng, G. Lagaly (Eds.), *Handbook of Clay Science*, Elsevier, Amsterdam (2006), pp. 1-18
- [5] B. Gámiz, R. Celis, M.C. Hermosín, J. Cornejo
- [6] Organoclays as soil amendments to increase the efficacy and reduce the environmental impact of the herbicide fluometuron in agricultural soils *J. Agric. Food Chem.*, 58 (2010), pp. 7893-7901
- [7] D.K. Sattler *Hand Book of Nanophysics: Functional Nanomaterials* CRC Press, Taylor and Francis Group, LLC (2011)