

## Original paper

### Synthesis of Montmorillonite-Copper Oxide Hybrid Nanocomposite

Masoud Fardin<sup>1</sup>, Ali Sadeghian<sup>1</sup>, Narges sadr<sup>1</sup>, Pardis Masaeli<sup>2</sup>, Elahe Aliheydari<sup>3\*</sup>

<sup>1</sup> Department of Biotechnology, Faculty of Basic Sciences, Urmia Branch, Islamic Azad University, Urmia, Iran

<sup>2</sup> Department of Biology, Faculty of Basic Sciences, Isfahan University, Isfahan, Iran

<sup>3</sup> Department of Biology, Faculty of Basic Sciences, Shahr-Rey Branch, Payame Noor University, Tehran, Iran

**Received:** 17 October 2022

**Revised:** 04 March 2023

**Accepted:** 03 March 2023

#### Abstract

**Background and aim:** The synthesis of nanoparticles, especially nanocomposite is of particular importance due to their application in the fields of medicine and industry. Based on this, the present study examines the synthesis of copper oxide hybrid nanocomposite.

**Materials and methods:** During this experimental-laboratory research, the chemical precipitation method of the composite structure was used for the synthesis of copper oxide-clay hybrid nanocomposite and it was confirmed by using FTIR and XRD. In addition, the morphology of nanoparticles was determined by SEM/TEM measurements.

**Results:** The results of the present study show that the processed nanocomposites were pure and had dimensions of 250-200 nm.

**Conclusion:** Overall, the results of this research showed that the nanocomposite of copper oxide and clay prepared in this research has the nature of a desirable nanoparticle and has desirable dimensions. This nanoparticle can be used in medicine and industry.

**Keywords:** *Synthesis, Hybrid nanocomposite, Copper-clay oxide*

---

**\*Corresponding author:** Elahe Aliheydari, Department of Biology, Faculty of Basic Sciences, Shahr-Rey Branch, Payame Noor University, Tehran, Iran

**E-mail address:** [e.aliheydari158@gmail.com](mailto:e.aliheydari158@gmail.com)

## **Introduction**

Composites in which the dispersed phase used is nanoparticles are called nanocomposites, which include polymer-based nanocomposites, ceramic-based nanocomposites, and metal-based nanocomposites [1]. Materials such as clay, metals, etc. are used as reinforcements in nanoparticle nanocomposites. For example, in polymer nanocomposites, small amounts (less than 10% by weight) of nanometer particles are used, which, in addition to increasing the strength of polymers, also reduce their weight. The importance of this type of nanocomposites is due to their lightness [2]. Copper is an important material for biocide applications. Copper ions ( $\text{Cu}^{+2}$ ) alone or in copper complexes have been used for centuries to disinfect liquids, solids and human tissues. Today, copper is used as a water purifier, algicide, fungicide, nematocide, molluscicide, antibacterial and anti-sediment agent [3]. Testing on a set of metal surfaces indicates that surfaces containing copper are the most effective in reducing the viability of bacteria [4].

Several strains of *S. aureus* were killed in less than 90 minutes on pure copper surfaces, while viable organisms of all strains were still detected on stainless steel after 72 hours [5]. Copper levels are used in different stages of food processing as an inhibitor of the growth of two of the most common bacterial pathogens that cause food borne diseases [6]. Also, copper is one of the most toxic metals for heterotrophic bacteria and other microorganisms in water environments, and for this reason, water distribution systems made of copper have a greater potential to suppress the growth and reduce the persistence of some microorganisms in drinking water than distribution systems made of plastic materials. Or have galvanized steel [7], [3]. Polymer clay nanocomposite (PCN) is one of the most important nanomaterials of the current decade with a wide range of applications. Montmorillonite, vermiculite, sepiolite, laponite, bentonite and attapulgite are the main classes of clay that are used as reinforcement in polymer nanocomposites [8]. Clay nanocomposites have special features such as thermal stability, flame retardancy, and anti-corrosion properties, and these features increase the importance of polymer clay nanocomposites in various fields, especially for environmental applications [9]. Nanocomposites based on polymer clay also have the potential of anti-pollution and water systems improvement, so the purification and improvement of polluted soil and air with the help of nanocomposites based on clay is also of interest [10].

Due to the wide spread of bacterial contamination and the infections caused by it and the resistance of bacteria to antibiotics, as well as the extensive clinical, social and economic complications resulting from these contaminations and bacterial resistance [11] and also considering the practical and useful effects of copper oxide-clay nanocomposites [12] and considering that most of the previous studies on antibacterial effects were focused on investigating nanoparticles and limited research has been done in the field of nanocomposites Based on this, the current research investigates the synthesis of copper oxide-clay hybrid nanocomposite and the results of this research have special importance in the field of bacterial infections and can be used in the treatment of antibiotic resistant infections.

## **Material and Methods**

### *Devices*

In order to investigate the vibration of the bonds, the infrared method (FT-IR) was used, which takes place due to the change of the bond length or the bond angle in the molecules, which was carried out with a Bruker Tensor 27 machine made in Germany in the research laboratory of Mohaghegh University of Ardabil [13]. Then, the X-ray diffraction (XRD) pattern of the samples for each sample was first made into a very fine powder and subjected to X-ray beam bombardment

with a wavelength of 0.1-100 angstroms, and the diffraction pattern of each crystal sample with standard diffraction patterns, type The compound was identified and registered in the laboratory of Razi Metallurgy Research Center [14]. Next, in order to investigate the morphology of nanoparticles, FESEM (Field Emission SEM) with MIRA3TESCAN-XMU model was conducted at Razi Metallurgy Research Center [15]. Also, TEM micrographs using Zeiss EM900 transmission electron microscope was prepared and registered at Sharif University of Technology laboratory service center [16]. In this research, copper oxide-clay hybrid nanocomposite was prepared using chemical precipitation method and all experiments were tested and optimized several times in the temperature range of 60-90 degrees and in the range of pH=9-11 and different amounts of raw materials.

#### *Synthesis of Copper Oxide-Resin Hybrid Nanocomposite*

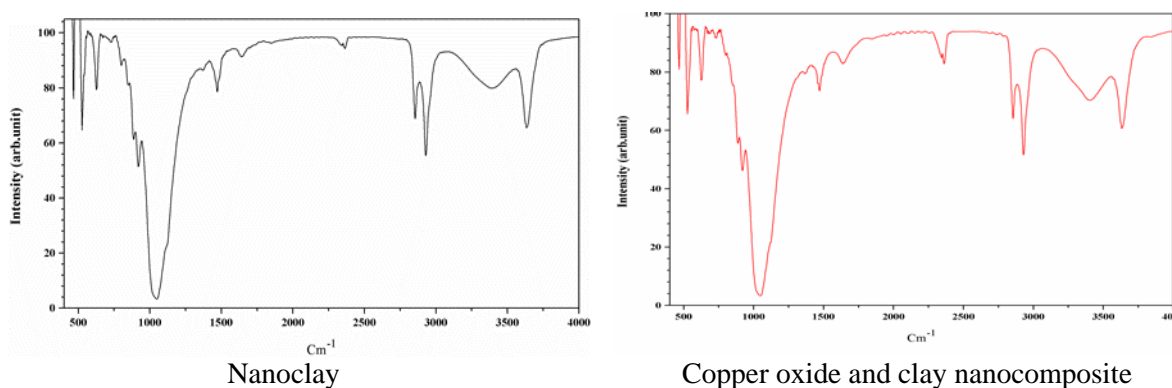
For the synthesis of this nanocomposite, 143.5 cc of double-distilled water is poured into the human body and placed on the Stirrer heater, then 2 cc of 2% glucose solution and 0.5 cc of 1% soda solution, 1 gram of cotton goods and 1 gram of clay are added, and the temperature and pH are controlled. It can be 9 as expected. Then 4 cc of 2% copper sulfate was added. The synthesis time of this nanocomposite is 1 hour, after the synthesis, the deposits were collected and dried in the oven at 90°C for 30 minutes. After the synthesis, the structure, morphology and size of nanoparticles were analyzed and investigated by spectroscopy (FT-IR), X-ray diffraction (XRD) and scanning electron microscope (SEM) [17].

## Results

#### *Interpretation of FTIR Spectra of Silver and Clay Nanocomposite*

The results of the FTIR spectrum recorded for nanoclay showed that the peak observed at 1050 is related to Si-o-Si vibrations, which is actually the main characteristic of nanoclay, Also the peak observed at 465 is related to the bending vibrations of the Si-o-Si group, and the peak located in the 526 region is related to Si-o-Al. The peak appearing in the range of 626 is the vibrations of Mo (oxygen bound to the metal) and the peak located in the area of 726 corresponds to Al-MgOH nanoclay.

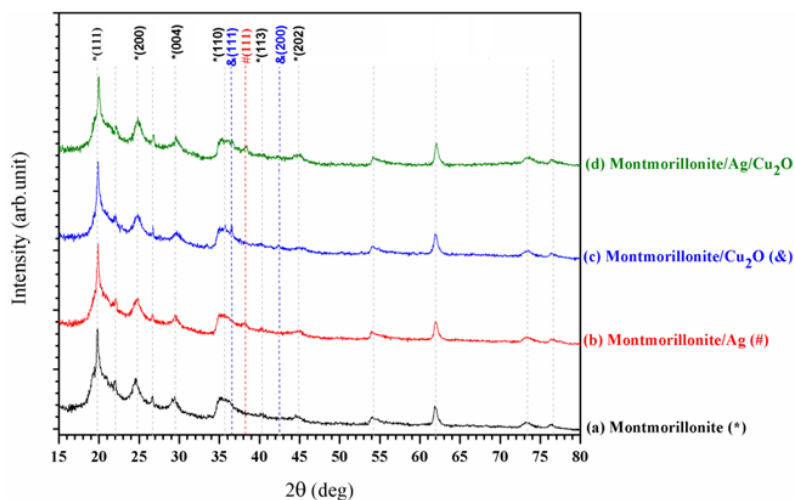
Also, the peak related to region 918 indicates Al-ALOH vibrations, in addition, the peaks observed at 3632-1637-800 are respectively related to the bending, out-of-plane, bending and stretching OH groups of the hydroxyl groups of the nanoclay surface (Figure 1). The presence of copper oxide was fully proven in other analyzes performed in this research such as XRD and FESEM.



**Figure 1.** Spectra of synthetic nanoparticles

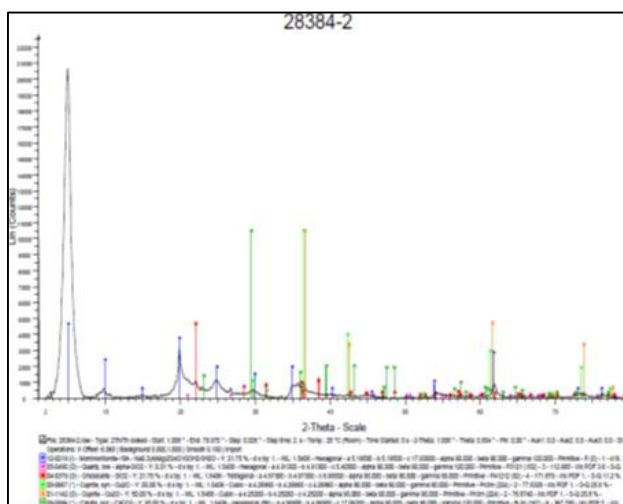
### Interpretation of the XRD Pattern

The results of the X-ray diffraction pattern indicated that the c pattern was the X-ray diffraction pattern related to copper oxide and clay nanocomposite (Figure 2).



**Figure 2.** Spectra obtained from the synthetic nanoparticle. The peaks with the symbol & are related to copper oxide and the three-digit code is written on the corresponding peaks.

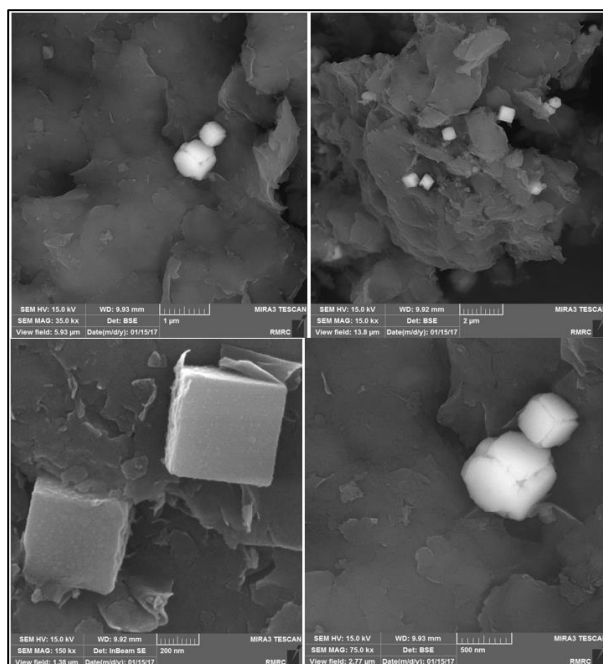
Also, the diffraction peaks of 2 were equal to the angles of 37°, 5° and 42°, respectively, corresponding to the crystal planes of copper oxide (Figure 3) from these crystal planes and diffraction angles, the distance between the crystal planes and the size of the crystals related to this structure can be obtained from Scherer's relation.



**Figure 3.** XRD spectrum of copper oxide and clay nanocomposite

### Investigation of FESEM Images of Copper Oxide and Clay Nanocomposite

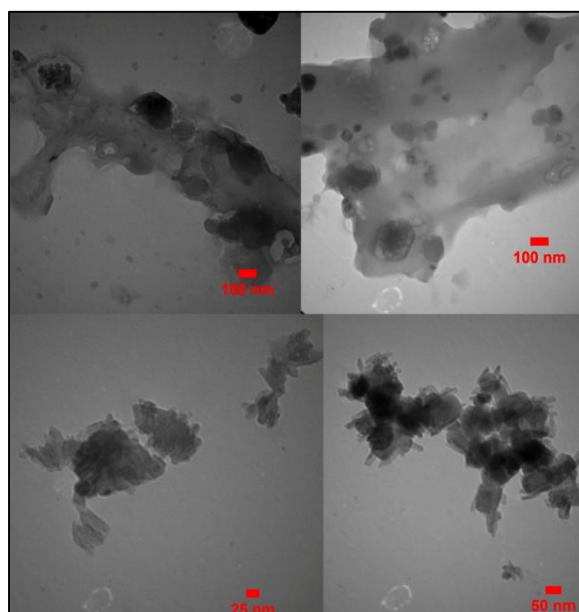
The results of examining the morphology of nanoparticles of field emission electron microscope images of nanocomposite of copper oxide and clay showed the polyhedral shape of copper oxide, which are placed between nanoclay layers, and the size of copper oxide nanoparticles was 200-250 nm (Figure 4).



**Figure 4.** FESEM images of copper oxide and clay nanocomposite at the scale of 2 micron, 1 micron, 500 nm and 200 nm

#### *TEM Electron Microscope Images*

The results of the transmission electron microscope TEM indicate the possibility of studying various things such as the micro structural characteristics of materials, plates and crystal directions, in this research, thin and transparent sections were prepared from the sample, which is clearly evident in the recorded images. Nanoclay layers were seen in all the images and nano copper oxide was stabilized in polyhedral crystal state between the layers (Figure 5).



**Figure 5.** TEM transmission electron microscope images of silver, copper oxide and clay hybrid nanocomposite at the scale of 50 nm and 25 nm

## **Discussion**

Many studies have shown that the synthesis of nanocomposites as well as their antibacterial [3], [5], [6], reinforcing [2], anti-fouling [3] and lightness [2] properties are of significant importance in the field of health [3], [5], [6]. And in this regard, the current research shows that different methods are widely used for the synthesis and preparation of nanocomposites. And they are generally classified into three categories: 1- polymer-based nanocomposites, 2- ceramic-based nanocomposites, and 3- metal-based nanocomposites [18]. Also, the antibacterial properties of nanocomposites, especially copper-clay oxide, have special effects in reducing the survival of surface bacteria and have a high potential for anti-pollution [4], [10]. In another research, the synthesis method of CuO-montmorillonite (CuO-MMT) nanocomposite was described. This nanocomposite was synthesized by a thermal decomposition method and characterized by diffuse reflectance spectroscopy (DRS), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). Also, the resulting particles were considered almost spherical and the size of the particles is in the range of ~3-5 nm. The results of this research show that CuO nanoparticles can exist in the micropores of MMT and the decrease in the size of CuO nanoparticles is also due to their quantum size effect [19]. In another study, for the first time, new nanosorbents (Cr<sub>2</sub>O<sub>3</sub>@PA6, CrO@PA6) and CuO@PA6 as modified polyamide nanocomposites were successfully synthesized and used for the effective removal of U(VI) from aqueous solution [20]. In line with these results, in connection with clay nanoparticles that act as a stabilizing agent, they prevent the accumulation of silver both on the particles and on the surface of thermally condensed coatings [21]. Accordingly, in connection with clay nanoparticles that act as a stabilizing agent, they prevent the accumulation of silver both on the particles and on the surface of the thermally condensed coatings [18]. Also, in another study entitled "Characteristics of Copper Oxide Nanoparticles", it was determined that the potential applications of Copper Oxide Nanoparticles (CuO) were investigated according to its characteristics, and in this study, it was stated that CuO on the nano scale contains traces of pure Cu and Cu<sub>2</sub>O nanoparticles is [22]. Because according to previous research, clay polymers have features such as anti-corrosion and high anti-pollution properties [9], [10]. And also nanocomposites, a unique material with high performance and a combination with special characteristics that are in the fastest demand in the fields of engineering plastics and elastomers and due to their special potential in several fields from packaging to biomedical applications are useful and useful. attention are, According to this research on the synthesis of hybrid nanocomposites and considering the applications of nanocomposites that have new technology and business opportunities for the biotechnology, automotive, aerospace and electronics industries due to their compatibility with the environment [23], And also, according to the general and accepted definition of hybrid nanocomposite, a material created by dispersing inorganic nanoparticles in a macroscopic organic matrix, research is telling that during the last two decades, according to the studies conducted and researches on the mechanical properties of organic-inorganic composites, recent researches have focused on systems such as clay-polymer nanocomposites. Which can be a suitable substitute for traditional fiber reinforced composites, whose applications are far beyond [24]. However, more studies are needed to understand the mechanisms of increasing the basic properties of polymers, creating their nanostructures [24] and revealing the exact mechanism of the effect of hybrid nanocomposites. This study was carried out in the scope of investigating the synthesis of lead oxide-hybrid nanocomposite and it was presented in terms of investigating the application of the nanoparticle processing method. This study has limitations in the medical nanobiotechnology industry and research and the use of antibacterial nanoparticles in the preparation of self-sterilizing surfaces and

its use in health centers and hospitals. The researchers of this project suggest, in the continuation of this research, studies regarding the synthesis of other nanocomposites of single metal oxides and hybrid metal oxides, examining the antibacterial properties of these substances in an animal model and investigating the anticancer and antiviral properties of these substances. Also, the use of these materials in the synthesis of pharmaceuticals and the use of antibacterial nanoparticles should be considered.

## **Conclusion**

Overall, the results of this research showed that the nanocomposite of copper oxide and clay prepared in this research has the nature of a desirable nanoparticle and has dimensions of 200-250 nm. This nanoparticle can be used in medicine, health and industry.

## **Acknowledgment**

We hereby express our appreciation and gratitude to all those who have helped us in the implementation of this research.

## **Conflict of interests**

The authors declare that there are no competing interests.

## **Reference**

- [1]. Ajayan PM, Schadler LS, Braun PV. Nanocomposite science and technology. John Wiley & Sons; 2006.
- [2]. Rajapaksha P, Cheeseman S, Hombach S, Murdoch BJ, Gangadoo S, Blanch EW, Et Al. Antibacterial properties of graphene oxide–copper oxide nanoparticle nanocomposites. *ACS Applied Bio Materials*. 2019;2(12):5687-96.
- [3]. Badrossamay M, Sun G. Enhancing hygiene/antimicrobial properties of polyolefins. In *Polyolefin Fibres 2009* (pp. 262-287). Woodhead Publishing.
- [4]. Wilks SA, Michels H, Keevil CW. The survival of *Escherichia coli* O157 on a range of metal surfaces. *International journal of food microbiology*. 2005;105(3):445-54.
- [5]. Noyce JO, Michels H, Keevil CW. Potential use of copper surfaces to reduce survival of epidemic methicillin-resistant *Staphylococcus aureus* in the healthcare environment. *Journal of Hospital Infection*. 2006;63(3):289-97.
- [6]. Faúndez G, Troncoso M, Navarrete P, Figueroa G. Antimicrobial activity of copper surfaces against suspensions of *Salmonella enterica* and *Campylobacter jejuni*. *BMC microbiology*. 2004;4(1):1-7.
- [7]. Omae I. General aspects of tin-free antifouling paints. *Chemical reviews*. 2003;103(9):3431-48.
- [8]. Ali F, Ullah H, Ali Z, Rahim F, Khan F, Rehman ZU. Polymer-clay nanocomposites, preparations and current applications: a review. *Current Nanomaterials*. 2016;1(2):83-95.
- [9]. Mallakpour S, Rashidimoghadam S. Recent developments in the synthesis of hybrid polymer/clay nanocomposites: properties and applications. In *Hybrid polymer composite materials 2017* (pp. 227-265). Woodhead Publishing.
- [10]. Buruga K, Song H, Shang J, Bolan N, Jagannathan TK, Kim KH. A review on functional polymer-clay based nanocomposite membranes for treatment of water. *Journal of hazardous materials*. 2019;379:120584.
- [11]. Islam MM. Bacterial resistance to antibiotics: access, excess, and awareness in Bangladesh.

Expert Review of Anti-infective Therapy. 2021;19(8):973-81.

[12]. Varaprasad K, López M, Núñez D, Jayaramudu T, Sadiku ER, Karthikeyan C, Oyarzún P. Antibiotic copper oxide-curcumin nanomaterials for antibacterial applications. *Journal of Molecular Liquids*. 2020;300:112353.

[13]. Mehri F, Farbodi M. Preparation of polyaniline – carboxymethyl cellulose – tio<sub>2</sub> hybrid nanocomposite and investigation of its physicochemical and antibacterial properties. *Journal of Advanced Materials and Technologies*. 2016;5(2):31-39.

[14]. Abarashi M, Mesgar Shahroodi S. Effect of silver nanoparticles on the crystallinity of polyethylene. *Advanced Processes in Materials*. 2016;10(2 (37)):105-13.

[15]. Salehi Vaziri H, Shokuhfar A, Seyyed Afghahi SS. Fabrication and investigation of properties of Al/WS<sub>2</sub>-CNT hybrid nanocomposites. *Journal of Science and Technology of Composites*. 2020;7(3):1021-8.

[16]. Mesgari Z, Gharagozlou M, Khosravi A, Gharanjig K. Synthesis and Characterization of Novel Hybrid Nanocomposite Containing Modified Titanium Dioxide Nanoparticles and Tetraisoindole Organic Pigment. *Journal of Color Science and Technology*. 2011;5(3):227-33.

[17]. Ebrahimi ME. Synthesis and manufacturing method of nanostructured materials. 2007.

[18]. Ravichandran K, Praseetha PK, Arun T, Gobalakrishnan S. Synthesis of nanocomposites. In *Synthesis of inorganic nanomaterials 2018* (pp. 141-168). Woodhead Publishing.

[19]. Sohrabnezhad SH, Moghaddam MM, Salavatiyan T. Synthesis and characterization of CuO–montmorillonite nanocomposite by thermal decomposition method and antibacterial activity of nanocomposite. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*. 2014;125:73-8.

[20]. Elzoghby AA, Bakry A, Masoud AM, Mohamed WS, Taha MH, Hassanein TF. Synthesis of polyamide-based nanocomposites using green-synthesized chromium and copper oxides nanoparticles for the sorption of uranium from aqueous solution. *Journal of Environmental Chemical Engineering*. 2021;9(6):106755.

[21]. Mejía HF, Seitz KH, Valdes M, Uheida A, Procaccini RA, Pellice SA. Antibacterial performance of hybrid nanocomposite coatings containing clay and silver nanoparticles. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*. 2021;628:127354.

[22]. Ren G, Hu D, Cheng EW, Vargas-Reus MA, Reip P, Allaker RP. Characterisation of copper oxide nanoparticles for antimicrobial applications. *International journal of antimicrobial agents*. 2009;33(6):587-90.

[23]. Camargo PH, Satyanarayana KG, Wypych F. Nanocomposites: synthesis, structure, properties and new application opportunities. *Materials Research*. 2009;12:1-39.

[24]. Merhari L, editor. *Hybrid nanocomposites for nanotechnology*. New York, NY, USA: Springer US; 2009.