
Facile Methodology of Sol-Gel Synthesis for Metal Oxide Nanostructures

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Abstract

Sol-gel is a low temperature, highly controllable and cost effective method for production of homogeneous, highly stoichiometric and high quality ultrafine nanostructures. Sol-gel route is adoptable way to choose desired shape of the metal oxide (MO) nanostructures such as nanospheres, nanorods, nanoflakes, nanotubes, nanoribbons, nanospheres and nanofibers for shape-dependent applications and comparative accessibility. Biomedical applications involving drug deliveries, mimicking of natural bone and teeth, anti-microbial activities and pharmaceuticals employ sol-gel prepared MO nanostructures because of their low temperature synthesis, homogeneity and purity. Apart from this, sol-gel route is preferred for synthesis of MO-based nanostructures with several ranges of applications such as magnetic applications, energy generation, conversion and storage devices, electronic device applications and sensors and actuators materials. In this chapter, we have discussed about the comprehensive ideas of sol-gel technique to synthesis metal oxide nanostructures.

Keywords: sol-gel, metal oxides, nanostructures

1. Introduction

Sol-gel process have been extensively explored for producing metal oxide nanostructures in the field of engineering and technological applications probably due to the controlled shape and size exhibited by the obtained products. Since the synthesis of silica gel by Ebelman

in 1846, this method has been developed progressively and sol-gel synthesized materials have been implemented in several applications with excellent optical, magnetic, electrical, thermal and mechanical properties [1]. Several forms of materials such as thin films, nanoparticles, glass and ceramics can be achieved using sol-gel method in a cost-effective way [2]. Low temperature chemistry, reproducibility and high surface to volume ratio of obtained products are other features that add merit to this technology [3]. Apart from this, sol-gel process have opened up some new avenues in bioengineering fields including drug delivery, organ implantation, pharmaceuticals and biomaterial synthesis due to the purity and quality of the yields from this process. These advantages have attracted researchers and industrialists to utilize this method widely for past few decades [4]. Metal oxides are class of functional materials with numerous applications and can be synthesized using sol-gel process [5]. Sol-gel synthesis of metal oxide can be done at relatively low temperature compared to the solid-state reactions. In general, sol-gel process involves formation of sol from homogeneously mixed solution, converting them into gel by polycondensation process and finally heat treating the product according to the material required [6]. The formation of crystalline materials such as nanoparticles or thin films and non-crystalline materials like ceramics, xerosol, aerosol and glasses depends upon the final heat treatment steps [7]. This chapter provides the fundamental steps involved in sol-gel process, various controlling parameters, the size- and shape-controlled synthesis of various metal oxides and application of sol-gel synthesized metal oxide nanoparticles in various fields.

2. Feasibility of sol-gel method

Figure 1 shows the schematic diagram of sol-gel method. The experimental set up is very simple. Sol is obtained by either hydrolysis or polymerization reactions by adding suitable reagents in the precursor solution. The sol can be deposited onto preferred substrates as thin films using two techniques, viz. (1) spin coating and (2) dip coating. The gelation process done through condensation of the sol or addition of polymers converts this sol to gel. This gel can be used to form materials of different types such as nanoparticles, xerogel, glass or ceramics depending upon the further processing steps involved. Nanoparticles and xerogels can be obtained by simple evaporation of solvent. The obtained xerogel can be formed as ceramics by heat treatment and glassy nature can be induced by melting techniques. Thus, sol-gel method can be used to obtain different forms of materials, controlled phase and shape and size of the derived materials [6].

The parameters that could be controlled in sol-gel method includes (1) concentration of precursor used, (2) nature of solvent used, (3) pH of the solution, (4) type of additives added and their concentration, (5) pre and post heat treatment of the materials, (6) aging of the solution and (7) nature of polymer used for condensation [8]. The particles formed in gel matrix possess uniform shape and size that enhances the optical, electrical, magnetic and other intrinsic nature of the materials. Low cost, low temperature chemistry, simple experimental set up and highly controllable synthesis are the major advantage of this method over other synthesis procedure. The large surface to volume of sol-gel-derived material makes it suitable

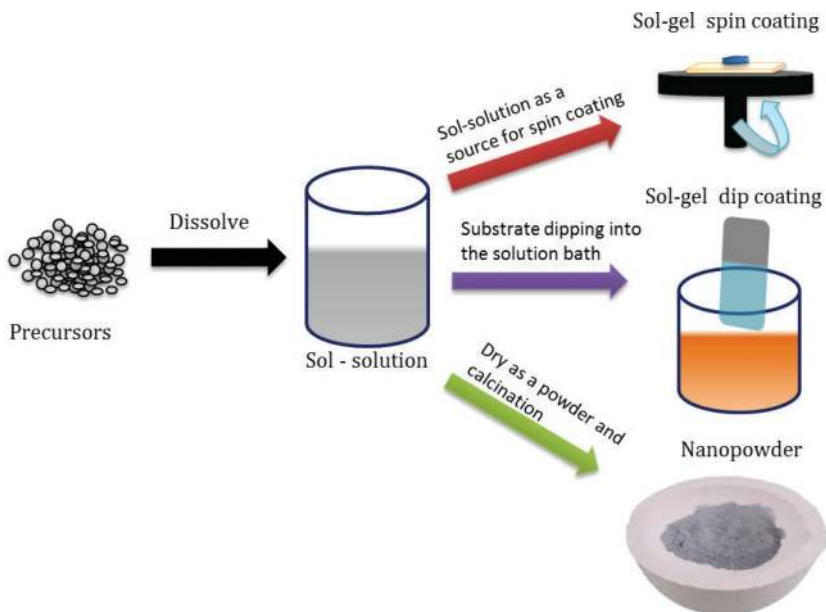


Figure 1. Schematic diagram of sol-gel processing.

for catalytic applications. Apart from this, low temperature chemistry ensures less defects to be induced in the materials formed. The purity and high quality yield with good physical properties makes researchers in biomedical and biotechnical field to adapt this method mostly for biomedical applications [9].

3. MO nanostructures using sol-gel

The synthesis of solid materials often involves wet chemistry reactions and sol-gel chemistry based on the transformation of molecular precursors into an oxide network by hydrolysis and condensation reaction [9–11]. Morphology plays a vital role in improving the properties of any material by enhancing high surface to volume ratio. The control of shape, size and packing structure of the particles in materials plays a crucial role for building next-generation devices and therapeutic materials [1]. Sol-gel method is an excellent tool to deploy a controlled architecture in material chemistry to fabricate metal oxide nanostructures (MONSs). Sol-gel prepared metal oxides have shown to exhibit excellent optical and electrical properties. The review of important MONSs derived from the sol-gel method helps us to understand the factors that could be taken into account for controlling the shape and size of the particles. In general, the solvents, additives, aging time and post heat treatment are few important factors that determine the shape and size of the building blocks of materials being synthesized [6, 12].

ZnO is a multifunctional material, which is widely being explored by many researchers for several years. Sol-gel method is preferably a low-cost simple method for preparation of numerous ZnO nanostructures with implementation in abundant scientific and industrial applications. As an example, XRD pattern of sol-gel dip coating route prepared ZnO thin film is given in **Figure 2**. The polycrystalline nature hexagonal structure of ZnO is exhibited with (002) preferential orientation at $2\theta = 34.48^\circ$. Also, low intensity peaks are exhibited with (100), (101), (102), (110), and (103) lattice orientations as shown in **Figure 2**. Li et al. [13] illustrated that the aging time of sol influenced on the morphology of nanoparticles. They found that the longer time aged sol had better morphology than as synthesized particles. Also, they suggested that the possible reason for this trend may be due to the uneven distribution of colloidal particle size. Previously, the effect of solvent on the growth of ZnO nanorods was explained by Foo et al., where they used ZnO seeds obtained from hydrothermal method as seeds for growth of nanorods [14]. With the advent of technology, flexible electronics is becoming popular in recent past. Low temperature synthesis is mandatory for materials to be coated onto the flexible substrates as the thermal stability of substrate materials has to be taken into account. Sol-gel method opens up a new avenue for low temperature deposition of MONS onto flexible substrates for construction of flexible electronic devices. In this view point, Sun et al. successfully reported uniform sol-gel-derived ZnO thin films at relatively low temperature ($\leq 200^\circ\text{C}$) that can function as efficient transporting layer in inverted solar cells [15]. Kim et al. fabricated sol-gel-derived ZnO on plastic substrates and demonstrated potential for Al/Sol-gel ZnO/Al for low cost flexible memory devices [16]. Xu et al. showed the control of thickness and packing density of the thin films by number of dips and post heat treatment of ZnO sols and their influence on the optical properties of the thin films [17]. ZnO nanowires synthesized by sol-gel route were investigated for photoconductivity studies and origin of the photoresponse was also analyzed by Ahn et al. [18]. Qu et al. demonstrated deposition of ZnO nanorod arrays as an antireflective layer in the polycrystalline Si solar cells [19]. Ghosh et al. adapted a cooling for the sol-gel deposited ZnO films at different cooling rates and uniform nanospheres were attained as a result. They showed that the films exhibited tuning of the optical emission and ultraviolet photosensing properties [20]. Moreover, metal doping is possible by sol-gel dip coating technique [21, 22]. As an example, Chandramohan et al. [22] have prepared Al-doped ZnO thin films and plausibly explained its variation of optical properties by increase of doping composition (**Table 1**). They demonstrated that estimation of various optical constants like band gap (E_g), refractive indices (n, k), dielectric constant (ϵ), optical conductivity (σ), average excitation energy (E_0), oscillator strength (E_d), effective mass (m^*), plasma frequency (x_p), static dielectric constant (ϵ_0) and carrier concentration (N). **Figure 3** represented that the SEM micrographs and EDX patterns of undoped and Al-doped ZnO thin films. The highly transparent thin films showed nanowires protruding from stacked nanorods on SEM inspection that signifies the suitability of these thin films for gas sensors.

Iron oxide is a versatile material, which can be used in many applications such as magnetic storage, drug delivery, sensor and electrochromic applications. Sol-gel process ascertains iron oxide with improved physical and chemical properties due to uniform particle size distribution, controlled phase synthesis and morphology. Dhanasekaran et al. have shown the influence of precursor concentration on the morphology of highly dispersed, single phase Fe_2O_3

nanostructure formed using sol-gel method. The nucleation and growth kinetics affected by the shape and size of the material was elaborated. **Figure 4** shows SEM micrographs of the Fe_2O_3 nanostructures at different $\text{FeCl}_3:\text{FeCl}_2$ concentrations ratio. They have obtained saturation magnetization (M_s) and coercivity values (H_c) at 35 T and 58 A/m, respectively, for Fe_2O_3 nanostructures prepared at $\text{FeCl}_3:\text{FeCl}_2$ molar ratio of 1:1.5 [23]. This observation shows that ferrous oxide nanostructures with low coercivity and high saturation magnetization value could be used as core materials in recording media.

Sol-gel-mediated synthesis of Fe_2O_3 nanorods was described by Woo et al. [24]. They showed that the diameter and length of the rods could be controlled by addition of ($\text{H}_2\text{O}/\text{Oleic acid}$) and the phase could be controlled by the temperature, atmosphere and hydrous state of the

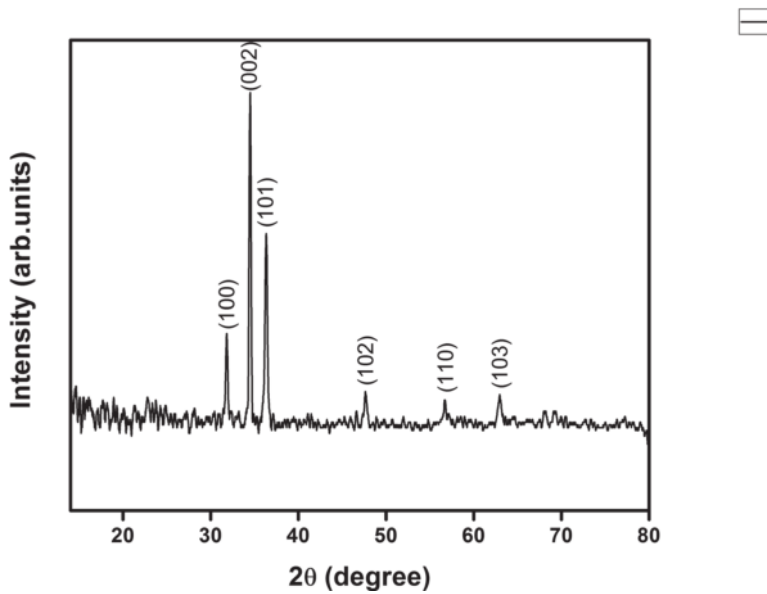


Figure 2. XRD pattern for sol-gel dip coating prepared ZnO thin film.

Sample	E_0	E_d	M_{-1}	M_{-3}	E_g	ϵ_∞	$\omega_p \times 10^{15} \text{ s}^{-1}$	m^*	$N \times 10^{12} \text{ cm}^{-2}$
Pure-ZnO	1.866	4.694	2.515	0.722	3.21	6.97	2.054	0.184	1.594
Al doping—0.01 mM	1.910	3.939	2.062	0.565	3.23	5.8	1.855	0.185	1.635
Al doping—0.03 mM	2.062	3.808	1.846	0.434	3.29	4.91	1.848	0.187	1.137
Al doping—0.05 mM	2.224	3.858	1.732	0.350	3.33	4.598	1.830	0.189	1.055

E_0 , average excitation energy; E_d , oscillator strength; E_g , band gap; ϵ_∞ , static dielectric constant; ω_p , plasma frequency; m^* , effective mass and N , sheet carrier concentration.

Table 1. Calculated optical and dispersion parameters of undoped and Al-doped ZnO films [21].

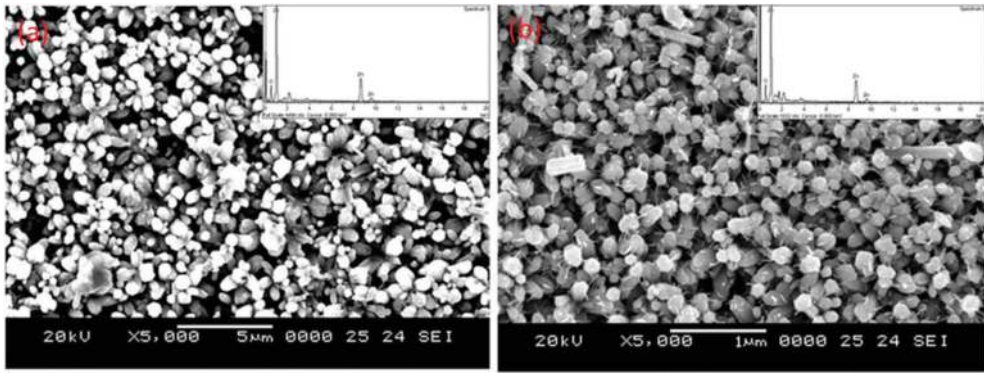


Figure 3. (a) SEM micrograph of ZnO thin film with inset EDX pattern and (b) SEM micrograph of Al-doped ZnO thin film with inset EDX pattern [21].

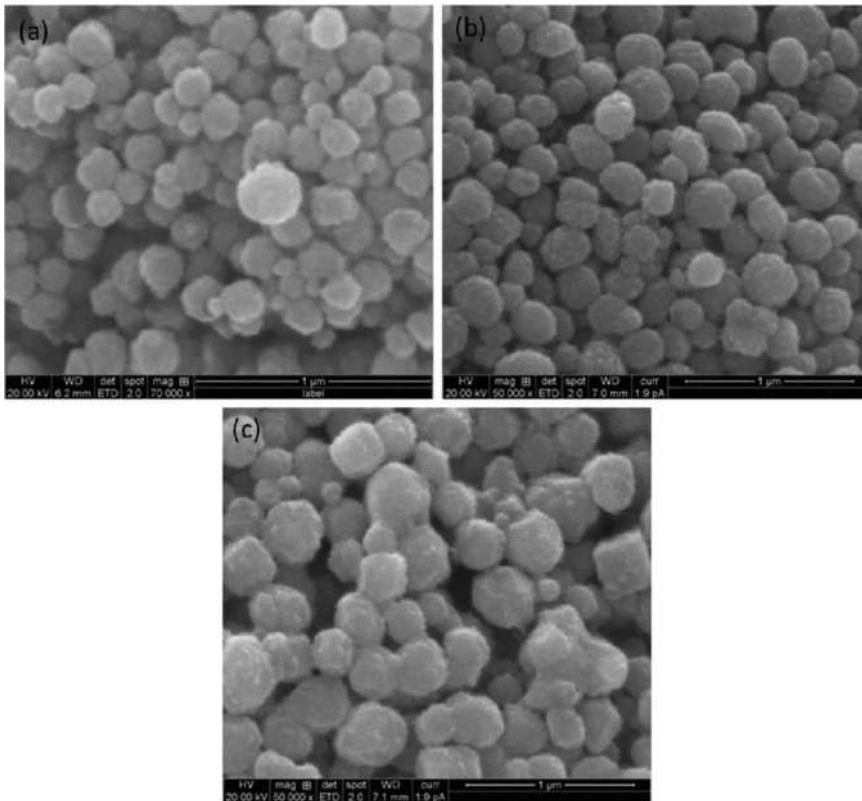


Figure 4. SEM micrographs of sol-gel route synthesized Fe_2O_3 nanostructures using different $\text{FeCl}_3:\text{FeCl}_2$ molar ratios such as (a) 1:1.0, (b) 1:1.5 and (c) 1:2.0.

gel during the crystallization. Liu et al. described α -Fe₂O₃ ultra-fine powder with an average particle size of 6–26 nm preparation by a sol-gel process. The temperature dependence of the electric conductance of the elements made of nanocrystalline α -Fe₂O₃ shows that the gas-sensing properties are strongly related to its surface. The elements exhibited good sensitivity and selectivity to ethyl alcohol, indicating that it is a promising alcohol-sensing material [25]. Leonardi et al. prepared iron oxide nanoparticles prepared by electrospinning and sol-gel method for ethanol sensing properties. Highly uniform shaped spherical rods were obtained using sol-gel method. They concluded that both the methods exhibited a good sensing response. This report also attributes the advantage of low temperature simple sol-gel method over other expensive techniques [26]. Sanchez et al. reported simple one pot sol-gel synthesis of epsilon polymorph of iron oxide stabilized in a silica gel. These reports provide an insight that sol-gel not only controls the shape and size of the material synthesis but also provides an excellent phase control and formation of different polymorphs in an orderly trend [27].

Tin oxide is an important transition metal oxide widely being studied for electronic and sensor devices. The additives also influence the morphology of materials in sol-gel process. The SnO₂ nanostructure was grown on Al₂O₃ substrates by a sol-gel spin coating method by Khadhim et al. A novel H₂ gas sensor based on a SnO₂ nanostructure was operated at room temperature (25°C). They observed that the addition of glycerine as additive to the sol solution increased the porosity of the SnO₂ nanostructure surface, which increased the adsorption/desorption of gas molecules leading to the high sensitivity of the sensor [28]. Seval et al. fabricated homogeneous nanostructured SnO₂ films using sol-gel spin coating method from tin (II) acetate solutions. A p-n heterojunction diode was proposed by depositing SnO₂ film formed at 600°C on p-Si substrates by them. They determined that the interface states played an important role in the conduction mechanism of the diode [29]. The role of polymerization agent was described by Bagherian et al., in their report where SnO₂ nanosheets were synthesized through sol-gel method using gelatin as the natural polymerization agent. The calcination temperature variables such as 650, 700 and 750°C were found to influence the resultant optical and structural properties. Morphological studies manifested that wide area SnO₂ nanosheets were obtained by using gelatin at the chosen calcination temperatures. Being a conductive semiconductor, this unique structure will have prospective applications as a conductive transparent substrate [30].

CuO is one of the most broadly studied metal oxides in semiconductor processing-based applications. Dhanasekaran et al. have demonstrated the influence of bath temperature on dip coating of CuO sol onto glass substrates on the structural and morphological properties of the thin films obtained. Homogeneous distributions of ellipsoidal granular-shaped particles were observed on the surface of the films. These films were exploited to have single phase structure and have excellent optical and magnetic properties. Shrividhya et al. [31] have demonstrated the effect of precursor concentration on the morphological properties of CuO thin films using sol-gel dip method (**Figure 5**).

The copper oxide nanostructures were synthesized using 0.2 and 0.6 M copper nitrate concentrations by a sol-gel method [32]. The X-ray diffraction pattern revealed that the prepared structures are polycrystalline in nature. All the reflections in these patterns could be indexed to standard diffraction patterns of copper oxide (JCPDS card no. # 89-5899). The typical X-ray

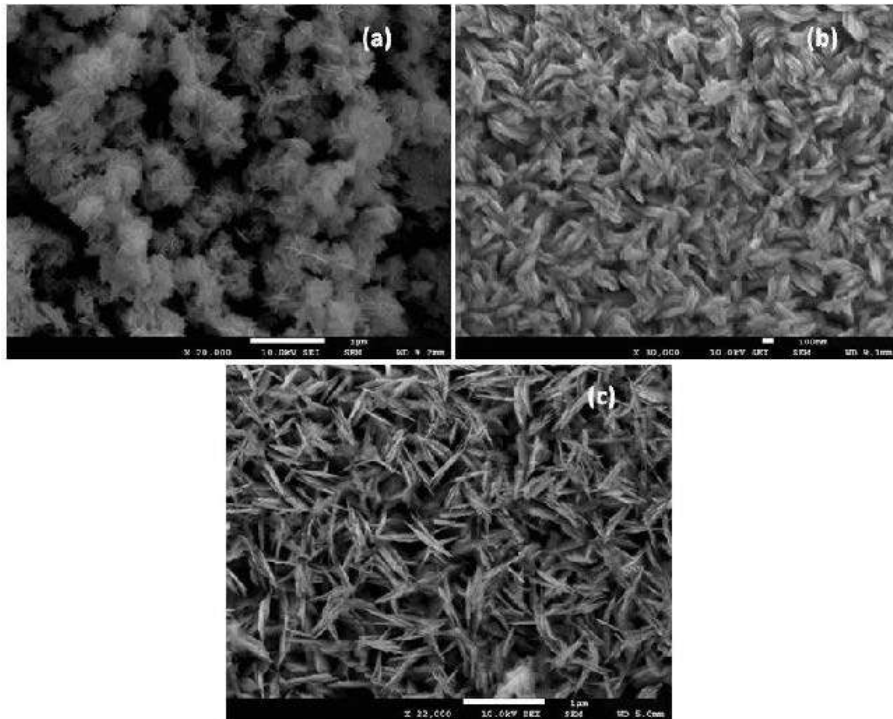


Figure 5. CuO nanostructures prepared by sol-gel dip coating method at (a) 5 mM, (b) 7 mM and (c) 9 mM CuSO₄ salt concentrations [31].

diffraction pattern of CuO nanostructures prepared at 0.2 M copper nitrate concentration is shown in **Figure 6(a)**. The refined unit cell parameters ($a = 4.650 \text{ \AA}$, $b = 3.518 \text{ \AA}$ and $c = 5.225 \text{ \AA}$) conformed well to the reported values of $a = 4.689 \text{ \AA}$, $b = 3.420 \text{ \AA}$ and $c = 5.130 \text{ \AA}$. The highly diffracted peak is observed at an angle $2\theta = 35.511$ corresponding to the (-111) lattice orientation. The various diffraction peaks such as (-111) , (111) , (-202) , (112) , (-113) and (-311) are observed for copper oxide nanostructures prepared at 0.2 M copper nitrate concentration. The full width at half maximum (FWHM) value of a predominant orientation peak is 0.43° . Also the novel reflection plane (110) of the Cu₂O phase emerged on the CuO nanostructures prepared at 0.6 M copper nitrate concentration (**Figure 6(b)**). This may be due to the increase of copper content in the nanostructures. The prepared thin films exhibited good optical properties [32]. Bibi et al. explained the varying annealing temperature on the CuO nanostructures. The morphological changes in turn greatly altered the optical, dielectric and electrical properties [33]. Moreover, NiO thin films prepared by sol-gel technique using different concentrations of nickel sulfate (NiSO₄) and their XRD patterns are presented in **Figure 7(a)**. XRD patterns revealed face-centered cubic polycrystalline nature of NiO thin films oriented along (200) lattice preferential orientations (JCPDS – 89-7130). Moreover, (111) and (220) lattice-oriented XRD reflections are exhibited with low intensity for NiO. The higher concentration of 0.40 M NiSO₄ using prepared NiO optical transmission and absorption spectrum provided in **Figure 7(b,c)** [34].

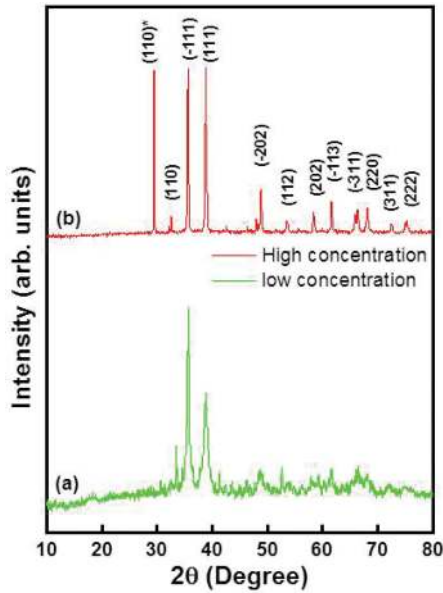


Figure 6. XRD patterns of sol-gel route prepared CuO nanoparticles using (a) low (0.2 M) and (b) high (0.6 M) copper nitrate concentrations [32].

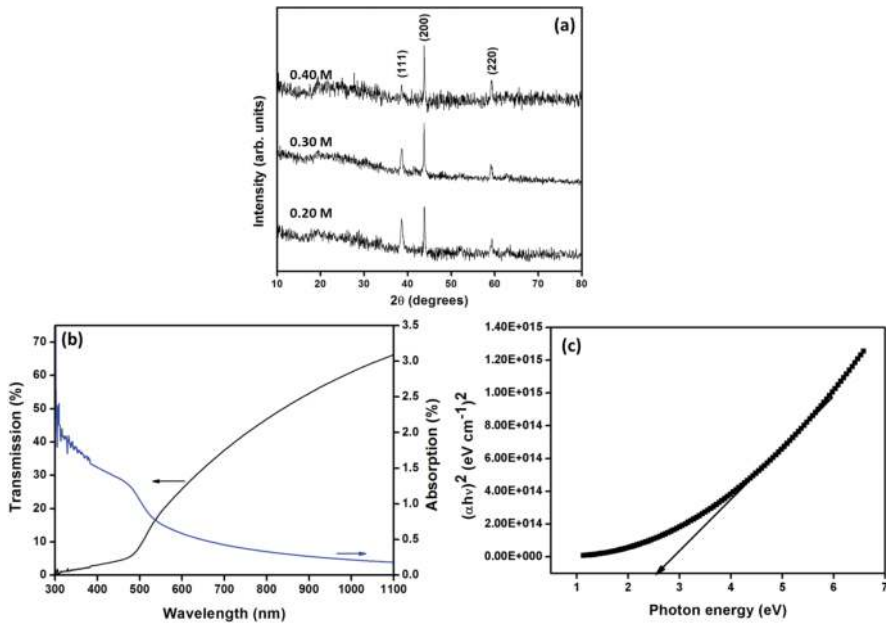


Figure 7. (a) X-ray diffraction patterns of different NiSO_4 concentrations using sol-gel deposited NiO thin films such as 0.20, 0.30 and 0.40 M. (b) Optical transmission and (c) optical absorption spectrum of sol-gel-coated NiO thin film prepared at 0.40 M NiSO_4 .

4. Size-controlled synthesis for various applications

Nanomaterials exhibit a vast shape and size-dependent properties and have found applications in diverse fields, including optics, electronics, mechanics, drug delivery and catalysis. Nanostructures as building block for bottom approaches to material integration encourage functional architecture. Such materials receive considerable interest due to their interesting application-specific properties. Researchers have given extensive reports on such size-controlled applications. As we have already discussed in detail, sol-gel method provides a fine control over the size and shape of the MONS especially for the applications using surface interface chemistry like catalytic activity, optics and electronic devices. SEM images of dip-coated ZnO (ZnSO₄ as a source) thin films which assembled by nanoflower- and nanoplate-like grains are shown in **Figure 8**.

NiO is another functional metal oxides mainly explored for energy-related applications. Dhanasekaran et al. [34] have demonstrated that shape selective synthesis of NiO nanostructures for hydrazine oxidation as amperometric sensor. They synthesized NiO NPs, having pellet, rod, dot and cuboid shapes, using a variety of reducing agents via a simple and low-cost sol-gel approach (**Figure 9**). Also, NiO-silica core-shell (NiO@SiO₂) NPs were prepared using tetraethyl orthosilicate (TEOS) as a source of the porous silica. They concluded nanopellet shape NiO exhibited the better electrocatalytic performance of 953 mA g⁻¹ for hydrazine oxidation. NiO and nickel manganite composite nanostructures have been synthesized by sol-gel route based on sorbitol as a chelating agent. Magnetic properties of the synthesized nanoparticles were analyzed [35]. Mutkule et al. deposited NiO nanostructures by sol-gel synthesis and their ability to sense LPG gas was analyzed by them [36].

Recently, Dhanasekaran et al. [37] demonstrated magnetic properties induced due to the size-controlled preparation in CuO nanoleaf structures (**Figure 10**). The reported saturation magnetization and coercivity values were at 0.24 T and 355.5 Oe, respectively, for nanoleaf structure of CuO (bath temperature at 95°C). Kato et al. illustrated the dependency of physical and chemical properties on size and shape of metal oxides [38]. Verduraz

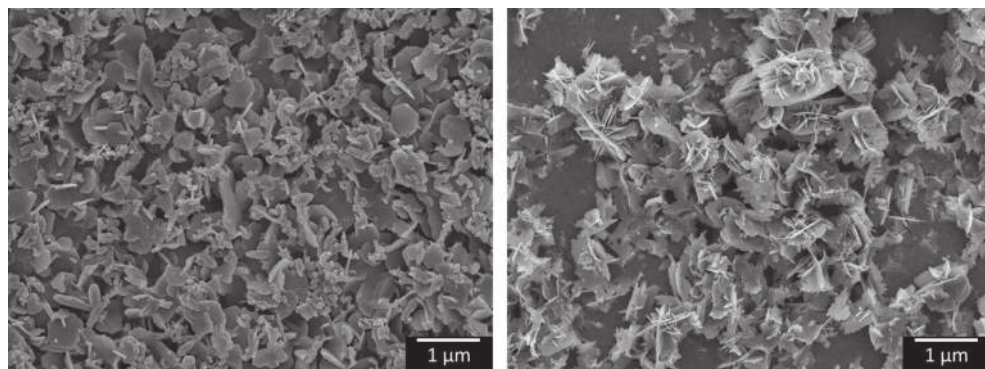


Figure 8. Nanoflower- and nanoplate-like morphological properties of ZnO nanostructures prepared by sol-gel dip coating.

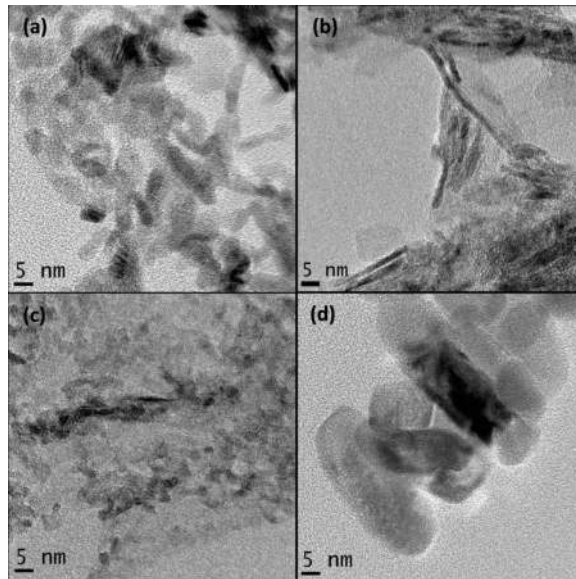


Figure 9. TEM images of NiO nanostructures prepared using different reducing agents (a) NaOH with PEG, (b) NaOH without PEG, (c) NH_3 and (d) Na_2CO_3 [34].

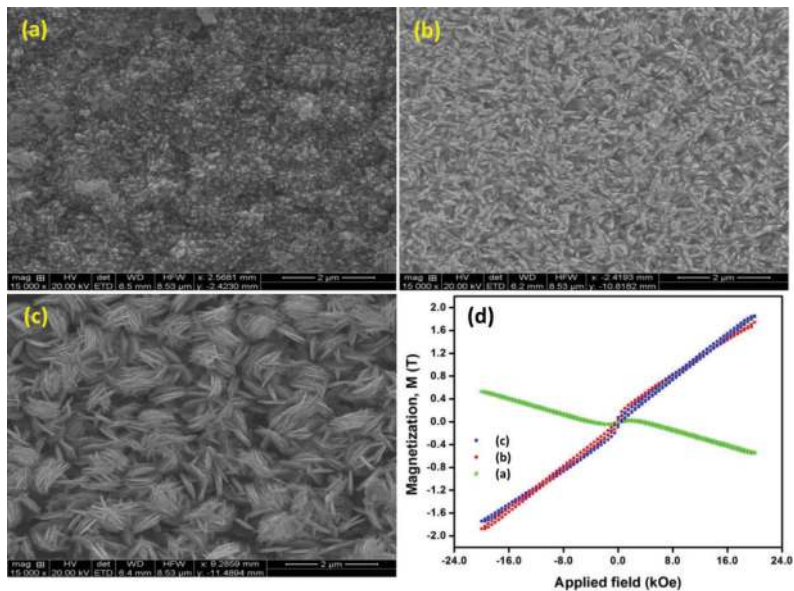


Figure 10. Morphological variations by adjusting bath temperatures for dip-coated CuO nanostructures (a) uneven and rough surface properties at 75°C, (b) elongated ellipsoidal-like grains at 85°C and (c) nanosheet-like morphology with regularly arranged surface at 95°C and (d) M-H hysteresis spectra for different bath temperatures (75, 85 and 95°C) using prepared CuO nanostructures [37].

et al. have reviewed the effect of shape and size control of MONS for catalytic applications [39]. Anandan et al. have presented a report on size and shape effect of NiO nanoparticles on their optical properties [40]. These fundamental understanding of size and shape effect on the properties of materials and knowledge about sol-gel processing would provide researcher a deep insight toward improving the existing properties of materials in a cost-effective and proficient way.

5. Conclusion

In summary, sol-gel method can be used for synthesise of MONSs in a bottom-up approach with perfect control over the shape and size of the derived products. The various steps involved in sol-gel material processing are elaborated. Also, a good level of understanding on the mechanism of shape and size control due to the controlling parameters like aging period, precursor concentration, solution pH, bath temperature and post heat treatment is achieved from the existing literature and also from our work. Uncomplicated and powerful ideas can be executed through simple cost-effective sol-gel processing to advance the technology to the next level.

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References

- [1] Pierre AC. Introduction to Sol-Gel Processing. New York: Springer; 2002. DOI: 10.1007/978-1-4615-5659-6
- [2] Klein LC. Sol-Gel Technology for Thin Films, Fibers, Preforms, Electronics and Specialty Shapes. New Jersey: Noyes Publications; 1989. DOI: 10.1002/adma.19890010816
- [3] Klein LC. Sol-Gel Optics Processing and Applications. New York: Springer; 1994. DOI: 10.1007/978-1-4615-2750-3
- [4] Attia Y. Sol-Gel Processing and Applications. New York: Springer US; 1994. DOI: 10.1007/978-1-4615-2570-7

- [5] Reisfeld R, Jorgensen CK. *Chemistry, Spectroscopy and Applications of Sol-Gel Glasses*. Berlin: Springer-Verlag; 1992. DOI: 10.1007/BFb0036963
- [6] Levy D, Zayat M. *The Sol-Gel Handbook: Synthesis, Characterization and Applications*. Germany: Wiley-VCH Verlag GmbH & Co; 2015. DOI: 10.1002/9783527670819
- [7] Lopez TM, Avnir D, Aegerter MA. *Emerging Fields in Sol-Gel Science and Technology*. New York: Springer US; 2003. DOI: 10.1007/978-1-4615-0449-8
- [8] Livage J, Ganguli D. Sol-gel electrochromic coatings and devices: A review. *Solar Energy Materials and Solar Cells*. 2001;**68**:365–381. DOI: 10.1016/S0927-0248(00)00369-X
- [9] Aegerter M, Mennig M. *Sol-Gel Technologies for Glass Producers and Users*. New York: Springer Science; 2004. DOI: 10.1007/978-0-387-88953-5
- [10] Livage J. Sol-gel processes. *Current Opinion in Solid State and Materials Science*. 1997;**2**:132–138. DOI: 10.1016/S1359-0286(97)80057-5
- [11] Guglielmi M, Carturan G. Precursors for sol-gel preparations. *Journal of Non-Crystalline Solids*. 1988;**100**:16–30. DOI: 10.1016/0022-3093(88)90004-X
- [12] Guglielmi M, Kickelbick G, Martucci A. *Sol-Gel Nanocomposites: Advances in Sol-Gel Derived Materials and Technologies*. New York: Springer-Verlag; 2014. DOI: 10.1007/978-1-4939-1209-4
- [13] Li Y, Xu L, Li X, Shen X, Wang A. Effect of aging time of ZnO sol on the structural and optical properties of ZnO thin films prepared by sol-gel method. *Applied Surface Science*. 2010;**256**:4543–4547. DOI: 10.1016/j.apsusc.2010.02.044
- [14] Foo KL, Hashim U, Muhammad K, Voon CH. Sol-gel synthesized zinc oxide nanorods and their structural and optical investigation for optoelectronic application. *Nanoscale Research Letters*. 2014;**9**:429. DOI: 10.1186/1556-276X-9-429
- [15] Sun Y, Seo JH, Takacs CJ, Seifert J, Heeger AJ. Inverted polymer solar cells integrated with a low-temperature-annealed sol-gel-derived ZnO film as an electron transport layer. *Advanced Materials*. 2011;**23**:1679–1683. DOI: 10.1002/adma.201004301
- [16] Kim S, Moon H, Gupta D, Yoo S, Choi Y-K. Resistive switching characteristics of sol-gel zinc oxide films for flexible memory applications. *IEEE Transactions on Electron Devices*. 2009;**56**:696–699. DOI: 10.1109/TED.2009.2012522
- [17] Xu L, Li X, Chen Y, Xu F. Structural and optical properties of ZnO thin films prepared by sol-gel method with different thickness. *Applied Surface Science*. 2011;**257**:4031–4037. DOI: 10.1016/j.apsusc.2010.11.170
- [18] Ahn SE, Ji HJ, Kim K, Kim GT, Bae CH, Park SM, Kim YK, Ha JS. Origin of the slow photoresponse in an individual sol-gel synthesized ZnO nanowire. *Applied Physics Letters*. 2007;**90**:153106. DOI: 10.1063/1.2721289.
- [19] Qu Y, Huang X, Li Y, Lin G, Guo B, Song D, Cheng Q. Chemical bath deposition produced ZnO nanorod arrays as an antireflective layer in the polycrystalline Si solar cells. *Journal of Alloys and Compounds*. 2017;**698**:719–724. DOI: 10.1016/j.jallcom.2016.12.265

- [20] Ghosh S, Basak D. A simple process step for tuning the optical emission and ultraviolet photosensing properties of sol-gel ZnO film. *RSC Advances*. 2017;**7**:694–703. DOI: 10.1039/C6RA25921A
- [21] Chandramohan R, Dhanasekaran V, Ezhilvizhian S, Vijayan TA, Thirumalai J, John Peter A, Mahalingam T. Reprinted from *Journal of Materials Science: Materials in Electronics*, Spectral properties of aluminium doped zinc oxide thin films prepared by SILAR method. With permission of Springer. 2012;**23**:390–397. DOI: 10.1007/s10854-011-0439-1
- [22] Chandramohan R, Vijayan TA, Arumugam S, Ramalingam HB, Dhanasekaran V, Sundaram K, Mahalingam T. Effect of heat treatment on microstructural and optical properties of CBD grown Al-doped ZnO thin films. *Materials Science and Engineering: B*. 2011;**176**:152–156. DOI: 10.1016/j.mseb.2010.10.017
- [23] Dhanasekaran V, Anandhavelu S, Polychroniadis EK, Mahalingam T. Microstructural properties evaluation of Fe₂O₃ nanostructures. *Materials Letters*. 2014;**126**:288–290. DOI: 10.1016/j.matlet.2014.04.065
- [24] Woo K, Lee HJ, Ahn JP, Park YS. Sol-gel mediated synthesis of Fe₂O₃ nanorods. *Advanced Materials*. 2003;**15**:1761–1764. DOI: 10.1002/adma.200305561
- [25] Liu XQ, Tao W, Shen YS. Preparation and characterization of nanocrystalline α -Fe₂O₃ by a sol-gel process. *Sensors and Actuators B: Chemical*. 1997;**40**:161–165. DOI: 10.1016/S0925-4005(97)80256-0
- [26] Leonardi SG, Mirzaei A, Bonavita A, Santangelo S, Frontera P, Panto F, Antonucci PL, Neri G. A comparison of the ethanol sensing properties of α -iron oxide nanostructures prepared via the sol-gel and electrospinning techniques. *Nanotechnology*. 2016;**27**:075502. DOI: 10.1088/0957-4484/27/7/075502
- [27] Lopez-Sanchez J, Serrano A, Del Campo A, Abuin M, Rodriguez de la Fuente O, Carmona N. Sol-gel synthesis and micro-Raman characterization of ϵ -Fe₂O₃ micro and nanoparticles. *Chemistry of Materials*. 2016;**28**:511–518. DOI: 10.1021/acs.chemmater.5b03566
- [28] Kadhim IH, Hassan HA. Hydrogen gas sensing based on SnO₂ nanostructure prepared by sol-gel spin coating method. *Journal of Electronic Materials*. 2017;**46**:1419–1426. DOI: 10.1007/s11664-016-5166-1
- [29] Aksoy S, Caglar Y, Caglar M, Ilıcan S. Influence of annealing temperature on the structural and optical characteristics of nanostructure SnO₂ films and their applications in heterojunction diode. *Journal of Nanoelectronics and Optoelectronics*. 2016;**11**:115–121. DOI: 10.1166/jno.2016.1885
- [30] Bagherian S, Zak AK. X-ray peak broadening and optical properties analysis of SnO₂ nanosheets prepared by sol-gel method. *Materials Science in Semiconductor Processing*. 2016;**56**:52–58. DOI: 10.1016/j.mssp.2016.07.021
- [31] Shrividhya T, Ravi G, Hayakawa Y, Mahalingam T. Determination of structural and optical parameters of CuO thin films prepared by double dip technique. *Journal of Materials Science: Materials in Electronics*. 2014;**25**:3885–3894. DOI: 10.1007/s10854-014-2103-z

- [32] Dhanasekaran V, Soundaram N, Seong-II Kim, Chandramohan R, Srinivas Mantha, Saravanakumar S, Mahalingam T. Optical, electrical and microstructural studies of monoclinic CuO nanostructures synthesized by a sol-gel route. Centre National de la Recherche Scientifique (CNRS) and The Royal Society of Chemistry. 2014;**38**:2327–2333. DOI: 10.1039/c4nj00084f
- [33] Bibi M, Javed QA, Abbas H, Baqi S. Outcome of temperature variation on sol-gel prepared CuO nanostructure properties (optical and dielectric). Materials Chemistry and Physics. 2017;**192**:67–71. DOI: 10.1016/j.matchemphys.2017.01.074
- [34] Dhanasekaran V, Park HJ. Shape-selective synthesis of NiO nanostructures for hydrazine oxidation as a nonenzymatic amperometric sensor. The Royal Society of Chemistry Advances. 2016;**6**:86101–86107. DOI: 10.1039/c6ra12805j
- [35] Freitas Cabral AJ, Remedios CMR, Ospina CA, Carvalho AMG, Morelhao SL. Structure of antiferromagnetic NiO/ferrimagnetic NiMn₂O₄ composite prepared by sorbitol-assisted sol-gel method. Journal of Alloys and Compounds. 2017; **696**:304–309. DOI: 10.1016/j.jallcom.2016.11.272
- [36] Mutkule SU, Navale ST, Jadhav VV, Ambade SB, Naushad M, Sagar AD, Patil VB, Stadler FJ, Mane RS. Solution-processed nickel oxide films and their liquefied petroleum gas sensing activity. Journal of Alloys and Compounds. 2017;**695**:2008–2015. DOI: 10.1016/j.jallcom.2016.11.037
- [37] Dhanasekaran V, Park HJ, Kim SI, Thaiyan M. Magnetic, structural and optical behavior of cupric oxide layers for solar cells. Journal of Alloys and Compounds. Elsevier. 2016;**686**:616–627. DOI: 10.1016/j.jallcom.2016.06.032
- [38] Kato K, Dang F, Mimura KI, Kinemuchi Y, Imai H, Wada S, Osada M, Haneda H, Kuwabara M. Nano-sized cube shaped single crystalline oxides and their potentials; composition, assembly and functions. Advanced Powder Technology. 2014;**25**:1401–1414. DOI: 10.1016/j.apt.2014.02.006
- [39] Bozon Verduraz F, Fievet F, Piquemal JY, Brayner R, El Kabouss K, Soumare Y, Viau G, Shafeev G. Nanoparticles of metal and metal oxides: Some peculiar synthesis methods, size and shape control, application to catalysts preparation. Brazilian Journal of Physics. 2009;**39**:134–140. DOI: 10.1590/S0103-97332009000200002
- [40] Anandan K, Rajendran V. Morphological and size effects of NiO nanoparticles via solvothermal process and their optical properties. Materials Science in Semiconductor Processing. 2011;**14**:43–47. DOI: 10.1016/j.mssp.2011.01.001

