



(REVIEW ARTICLE)



Review article: Sol-gel method, “synthesis and applications”

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World Journal of Advanced Engineering Technology and Sciences, 2023, 08(02), 160–166

Publication history: Received on 28 January 2023; revised on 21 March 2023; accepted on 24 March 2023

Article DOI: <https://doi.org/10.30574/wjaets.2023.8.2.0071>

Abstract

The sol-gel method is one of the techniques discovered in the past, but its use began in the sixties of the last century with the increasing use of this technology because of its features that are not found in traditional methods. This technique is defined as the guided method for the formation of inorganic oxides, with gelatinous structures, which are converted into solid glass (amorphous structures) at low temperatures, and can be defined from a thermodynamic point of view as the formation of a relatively stable solid phase at a given temperature, starting from the liquid phase. Sol-gel process is used to produce ceramic nanoparticles

Keywords: Sol-gel; Synthesis; Acidity; Calcination; Precursor

1. Introduction

The production of different materials required to reach for the ability to large quantities and specific physio-chemical properties which make it typical for many or specific applications. The sol-gel method when define for synthesized nano materials is a bottom-up rearrangement to forming structure with at least one diameter less then 100 nm by set of irreversible reaction [1]. While the sol-gel for synthesized classic or typical materials means forming materials in macro scale by using sources and specific condition. In the two definitions the mechanism include “starting with initial homogeneous particles which represent the sol phase and that will be convert to heavy particles with three-dimensional and that refers to a gel form. Generally, the sol-gel method commonly used as a popular with limiter industrial application as compare with methods such as chemical or physical vapour deposition [2-3]mechanical or collision with high-energy pellets, and electrochemical methods. May be the mains benefit which encourage to make it the most popular method were represent by abilities to produce materials with high quality , homogeneous size and shape [4]. in addition to cheap and simple strategy that could be applied in easy requirements with high purifies. In figure 1 the typical skim for the chemical forming of a colloidal solution which is the sol phase then with times convert to more density phase of a gel with double mixture liquid and solid . The times represent one of the important requirements to forming solid or sedimentation materials . In this review , some of the experimental investigations were summarized the influence of processing factors such pH, calcinations/annealing temperature and capping agents, on morphological and some properties of materials that commonly synthesized by sol-gel method.

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Figure 1 The typical skim for sol-gel process

2. Influence of pH

The effect of acidic or H^+ ions and alkaline or OH^- ions present in the solution which means the influence in sol state and that responsible to forming the primary particles (semi-dissolved) and that effect with the concentration of it [5] when the hydrolysis and condensation reactions occurring in this time. the morphology of synthesized ZnO particles [6] were shown a variation with change the pH of the solution. The reason behind the variance in morphology and size can related to the variation in the number of nuclei and growth ratios.

The pH of a medium significantly also affects on crystal structure and surface morphology of TiO_2 nanostructures [7] when pH between 2.5 and 4.5 was caused produce a rutile crystalline phase, while anatase phase was formed at pH 6 and the value between them was mixture for anatase and rutile.

WO_3 nanoparticles [8] morphology, crystallinity, chemical bonds, and optical properties were significantly affects with change pH . Increase pH of solution from 1 to 2 leads to crystal phase change with raises more ratios for the hexagonal phase percentage, and increases nanoparticles size. The physio-chemical properties also witness a blue shift at the optical absorption with increasing pH value.

Synthesized of VO_3 [9] shown mainly influence on pH for the two reactions, hydrolysis and condensation, after vanadium salt was dissolved in water . the experiments refers to internal proton transfer between V-OH groups which causing decrease the positive charge of vanadium at $pH < 2$. all of this causing the formation of $[VO_2(OH)_2]^+$ which characterized with forming two $V = O$ bonds in cis positions [10].

The physical properties of synthetic silicate gels were shown variance, when various pH values were applied on gel structure and morphology. Experiment Results shown that gels prepared at pH 3 was contained fewer primary particles then 7 and 9 when diameters larger than 7.2 nm and 30 nm respectively.[11]

3. Calcination effect

Thermal treatments after drying [8] the synthesized materials which represent by calcination were shown reduce in energy bandgaps for materials after calcined samples .Synthesized VO_3 from NH_4VO_3 as sources of V with oxalic acid in a DMSO- H_2O as solvent by hydrothermal treatment causing produced flat particles like a rose structure, which stable until 500 °C without destroying the flower morphology as show in figure 2. the flower nanostructures are reported as VO_3 nanostructured which reduced as petaloid clusters or hollow dandelions [12].

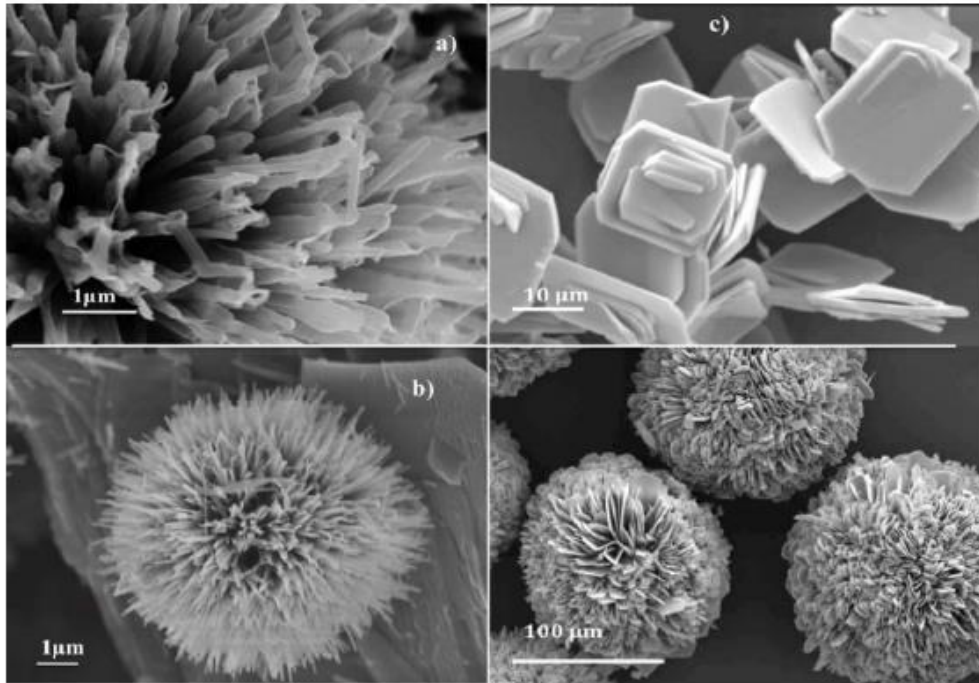


Figure 2 Nanostructured VO₃ obtained by self-assembling of nanoparticles: a) and b) VO_x nanotubes, c) and d) nanospheres from Platelets and flower respectively

The effect of calcination time on synthesis TiO₂ NPs which [13] produced from Aloe vera leaf extract and thermal treatments at 500 °C with different times . The increased in time which spend for calcination caused forming small diameter of TiO₂ with 23 ± 2 nm after 5h of calcination and 83 ± 5 nm after 1 h of calcination in addition to rices crystalline nature, the photocatalytic activity of synthesized TiO₂ was shown removed of azo dye Remazol Red Brilliant for the catalyst after 5h of calcination more then 1h respectively under visible light irradiation.

The effect of calcination temperature [14] was observed on particle size, crystallite size, and phase transition of synthesized TiO₂ nanoparticles, which witnessed particle size, crystalline size, and the crystallization with Increasing calcination temperature. The influence of calcination was appears in the change of phases TiO₂ when shown mixture of anatase and brookite at lower calcination temperature as reported in figure 3 while a three-phase mixture appeared when 500–600 °C was the temperature of calcition, and higher temperature, was responsible two shown the rutile phase at more 800 °C.

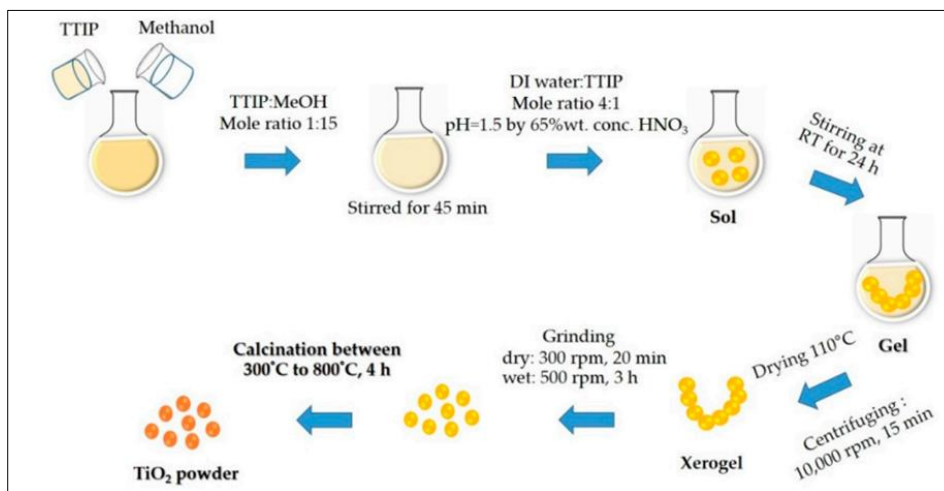


Figure 3 The skim for synthesis TiO₂ by sol-gel and calcination at different times and temperature

4. Effect of precursors

Generally additional cations in a sol, with mechanism of the hydrolysis and condensation reactions mostly requiring organometallic precursors which sequentially added to avoid most probable problems as coprecipitation[15].The sol-gel syntheses some research were reported that Si precursor was added to the reaction mixture before Al and Na precursors [16] when the bioglass, are made with Ca which involves the addition Na precursor to the reaction mixture after addition a Ca precursor [16].The synthesis of viable gels, with multiple additions and different precursor materials may be lead to unstable a consistent method when change the pH condition, which may required initial synthesis conditions, such as mixing times and previous adjusted catalyst addition .

Films of titanium dioxide were deposited on substrate of silica glass with dynamic spin coating and different concentrations of titanium tetra isopropoxide precursor in stable solutions, which characterized to determine the morphology and optical properties of titania surface in thin films. The results reported that lower concentration causing produced uniform, stable film and higher optical properties, while increased concentration, causing degrade adhesion of the film in addition to reduce surface and optical properties [17].many reported litterateurs studied the influence of precursors on the steps of sol-gel method starting from hydrolysis and condensation, with highlight in the crosslinking between alkyl and chloroalkyl series [18]. The results mentions that the inductive and steric effects on the sol-gel process accrued due to hybrid alkyl chain and the chlorine atom in sol and that improve the roles of four-fold rings due to the local periodicity associated which increases with higher percentage of precursor [19]. The nature of physical phase for precursor effect on the sol-gel method [20] when the precursor was not dissolved causing produce MgF_2 as product at the surface of in addition to the commercial alkoxides were hydrolysed with very little vale . reaction with moist air, and hence, HF under these reaction conditions (nonprotic organic solvent) is not able to break off Mg-O-Mg moieties in the resulting $Mg(OR)_x(OH)_y$ phases.sodium tungstate dihydrate [21] was used as a precursor to synthesized (WO_3) NPs by using two methods sol-gel and hydrothermal as shown in figure 4. The works include two parameters in addition to synthesized routs which was calcination/synthesis temperature which responsible to shown specific structural and morphological of the product. The results shown that synthesis method acted as parameter controlling on the morphology to forming the plate or rod of WO_3 which changed the crystalline within increasing hydrothermal temperature. .

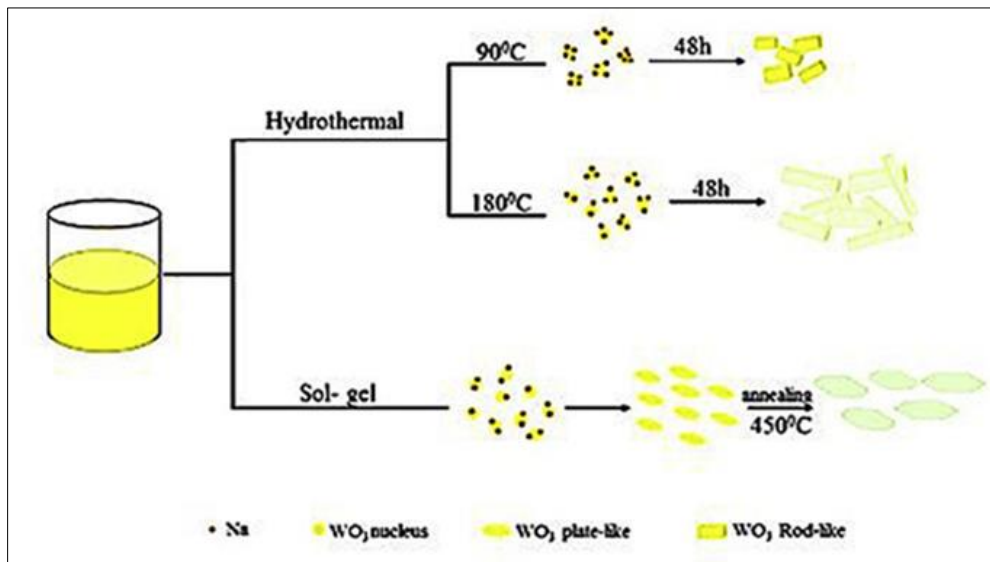


Figure 4 The skim for synthesized WO_3 with two different routs

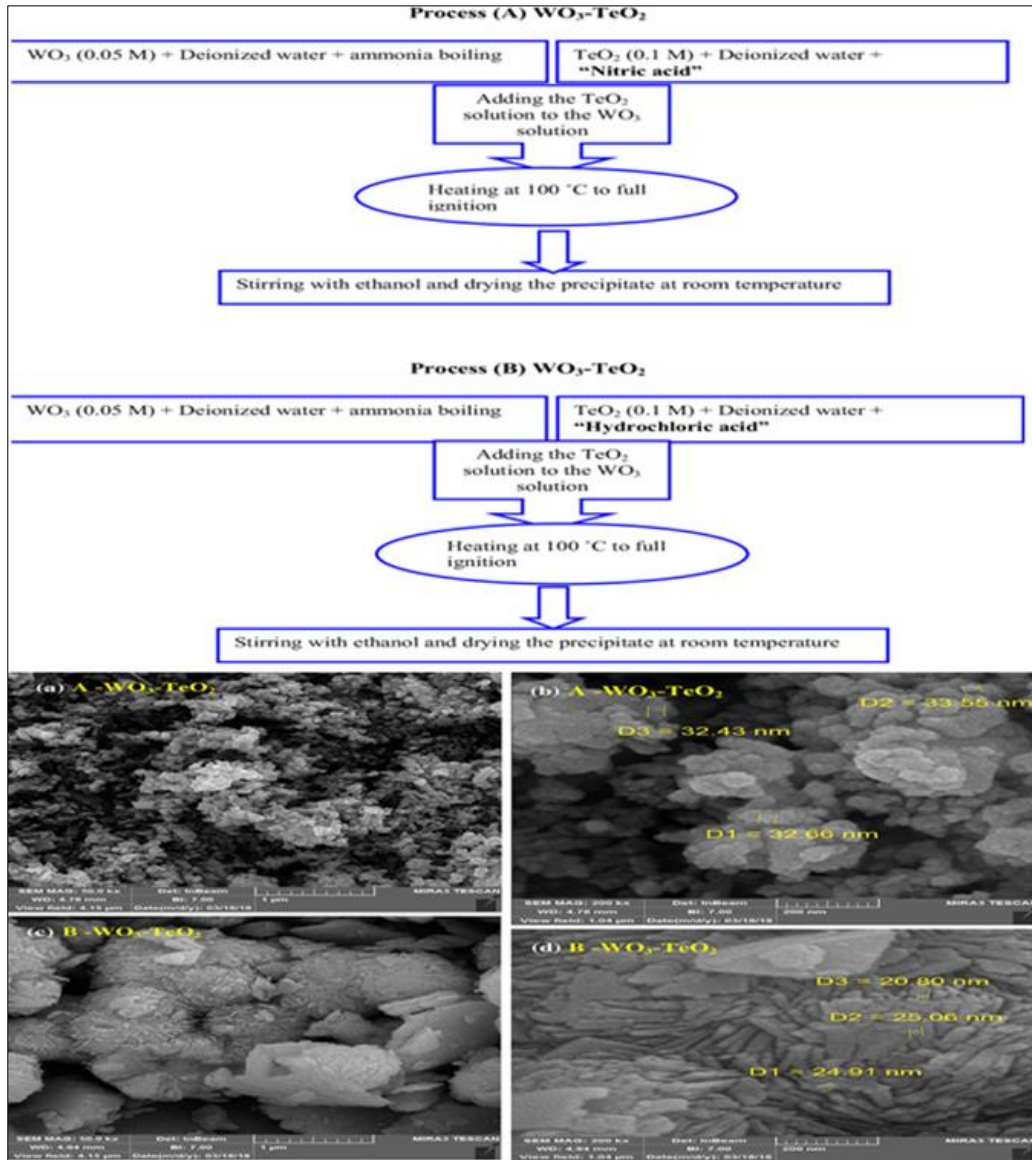


Figure 5:The skime for synthesized binary composites (the upper part) represent the steps of perpetration, while (the lower part) include scanning electron microscopy

Binary nano materials of WO_3 - TeO_2 were prepared from different precursors solutions in acidic media HNO_3 and HCl . The results reported that the WO_3 - TeO_2 nanoparticles size which prepared in HCl solution was smaller than the NPs which produce in HNO_3 solution. The images of FE-SEM for NPs which produced from HNO_3 solution showed hexagonal and tetragonal, while HCl solution formed uniformly polygon monoclinic structure of rod[22]

4.1. Application

Many applications were succeeded when used sol-gel as method to synthesis and fixed different materials in variance surfaces , thus in this part we will highlight in common applications which summarized in figure5. The Thin films are commonly used depend to fixed NPs on specific surface with characterized in nanometers or micrometers in thickness, which suitable as light barriers, storage means, corrosion resistant layers, conduction enhancement. the reflectance and absorption with transmitting are amazing behaviour for synthesis solar cell [23], such as TiO_2 or SiO_2 and ZnO in pristine or modified with many elements such Ag [24]. the medical and environmental filed were used sol-gel as method for synthesis chemical sensing, such silica in pristine or hybridization covalently when bonded with macro and small molecule.

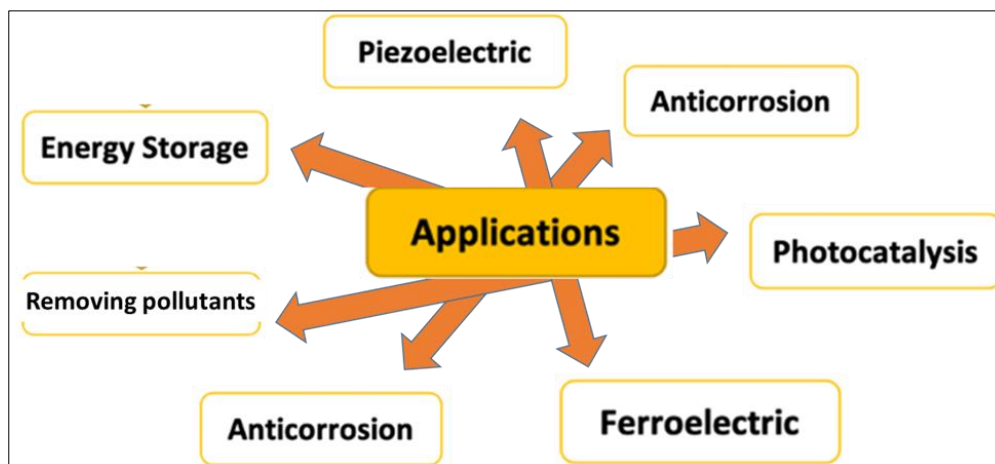


Figure 6 The skime for summary application of sol-gel method

When used sol-gel method, it could be added verity of functional groups that can modifying textile compound which succeeded for antimosquito [25], water repellency [26], ultraviolet (UV) protection, abrasion resistance, tensile and thermal properties. May be China represent the best example for benefit from sol-gel for clean energy sources and energy conversion with new electrochemical devices, such as supercapacitors, and fuel cells. The active topic for energy represent by higher energy density, and safety which could [27] be achieved by depend on sol-gel as method for engender huge compound of Nano materials.

5. Conclusion

The sol-gel method did not represent only ways to synthesized NPs but know it represent the main ways to manufacturing NPs with large quantities and specific qualities. The amazing and external behaviour which make sol-gel the ideal method was the abilities to enhance the selectivity of method by controls the pH , precursors ,and calcination temperature with times of thermal treatments. All of these parameters were succeeded to synthesis specific materials with specific physio-chemical properties for any applications.

Compliance with ethical standards

Acknowledgments

Sincere appreciation and gratitude to Al-Karkh University of Science , College of Science , and College of Energy and the Environment to support us by providing scientific sources.

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