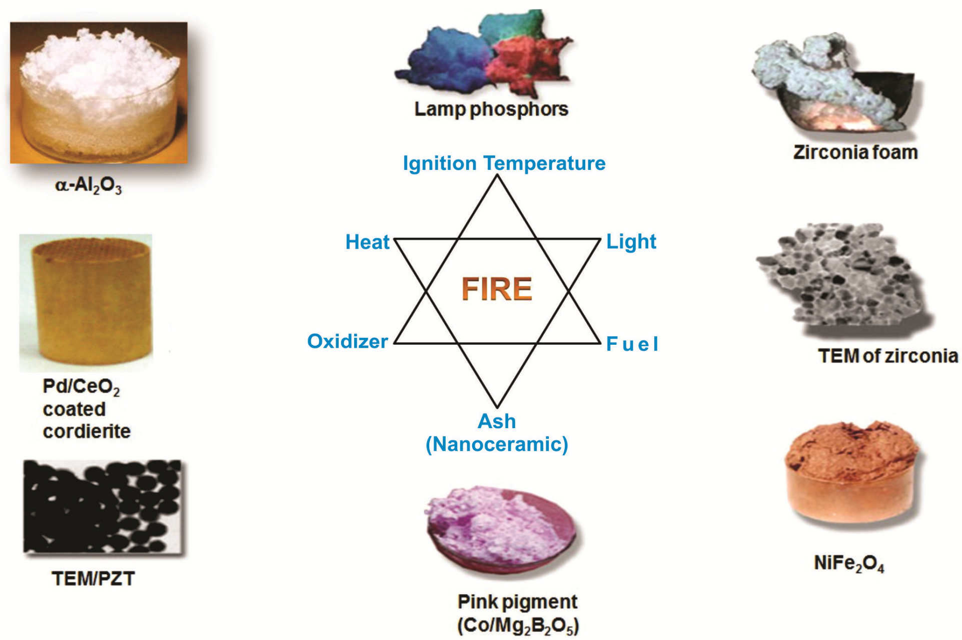
Solution Combustion Synthesis of Nanoceramic Materials : A Bibliography



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PREFACE

Solution combustion synthesis (SCS) was accidentally discovered in 1988 at Indian Institute of Science (IISc), Bengaluru, India. SCS involves an exothermic redox chemical reaction between an oxidizer like metal nitrate and a fuel in an aqueous medium. The aqueous solution containing stoichiometric amounts of redox mixture when rapidly heated boils, foams and catches fire to yield voluminous oxide product within few minutes. Alumina and related materials were the first ceramic powders prepared by corresponding metal nitrates and urea mixtures. SCS has now been standardized and used to prepare a variety of nanoscale (0, 1, 2 and 3 dimensions) oxide materials, metals, alloys, and sulfides with desired composition, structure, morphology and property for any specific application. SCS has several advantages over other known methods in terms of cost, time, equipment etc. Ease of in-situ atomic level doping of desired metal ions in the oxide matrix resulted in the preparation of ceramic pigments, phosphors, semiconductors, and catalysts. Evolution of large volumes of gases during combustion yields high surface area porous materials. The popularity of SCS method in the synthesis and manufacture of nanoceramic powders is due to their applications as abrasives, structural ceramics, pigments, phosphors, semiconductors, superconductors, catalysts, solar energy conversion and storage and environmental (air, water, and soil) pollution control and remediation. Today, a nanoceramic is a billion-dollar industry and growing further. In the last 3 decades since the first publication on SCS in 1988, SCS has become a very popular method and nearly 2500 publications are available on the topic. We have attempted to put them together here and list them under the following headings: Books and book chapters, review articles, Ph.D. theses from IISc., papers, and patents. The publications are listed under various catagories of ceramics: structural ceramics (alumina, chromia, and zirconia and related materials), catalysts, optical ceramics, electroceramics (magnetic, dielectrics, semiconductors), energy materials (fuel cells, batteries, solar cells and supercapacitors); and miscellaneous oxide materials The analysis of the data shows that the largest numbers of papers are on optical ceramics (737) followed by structural ceramics.and others (643), catalysts (615), electroceramics (557), and energy materials (264).

We hope this book will be useful to chemists, physicists, materials scientists, chemical engineers, and ceramic industries.

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Solution combustion synthesis of nanoceramic materials: A bibliography

1. Ceramics

In the history of civilization of Mankind, various periods are named by the materials used. For example, Stone or Ceramic age, Bronze Age, Iron Age, Polymer or Synthetic Materials age etc. Presently, we are in Nano Materials age. Traditional ceramics are naturally occurring materials like stone, sand, clay which were used as building materials to make bricks, tiles, pots, etc heralded the first ceramic age. Advanced ceramics which includes functional and structural ceramics mostly synthetic ceramics like alumina, aluminates, ferrites, chromites, zirconia etc. brought in the second ceramic age. Today we are in the third ceramic age i.e. nanoceramic age. Every new materials age has made our life more comfortable and better and nanoceramics are going to play a vital role in our lives. Nanoceramics are inorganic oxide materials in the size range of 10-100 nm. The advantage of nanoceramics over the advanced ceramics is their unique and superior properties compared to the coarse powders. Today nanoceramic oxide powders are important due to their wide range of applications such as in electronics, energy, biomedical, defence, paints, and coating industry. Nanosize oxides are also used as catalyst and stabilizer for the chemical manufacturing industry and also for environmental remediation. Nanosized oxides will play a significant role in clinical diagnosis and drug delivery systems. They are also going to revolutionize the miniaturization of electronic devices. Nanomaterials are usually added in small quantities to improve the performance of the base material. According to a report by Grand View Research, Inc., the global nano metal oxides market is expected to reach USD 9.48 billion by 2025. The demand for nanosize oxides is very huge as seen from the plot (Fig.1). Among the nanosize oxides, silicon dioxide occupies lion share and is extensively used in the manufacture of glass, coatings, rubber and medical, optical, and electronic devices. Iron oxide is used as a contrast agent in magnetic resonance imaging systems due to its superior magnetic properties. Titanium dioxide and zinc oxide are widely used in cosmetics, paints, and coatings.

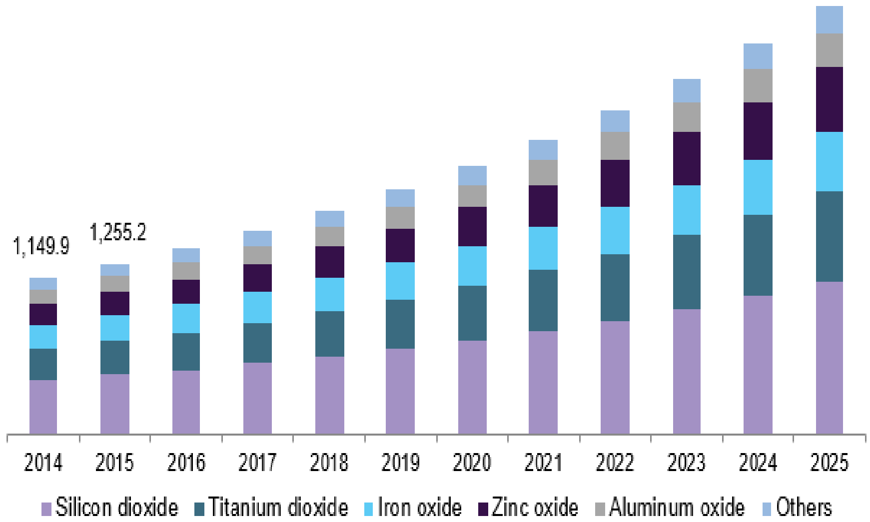

Fig.1. Market survey for different nanosize oxides (Source: Google)

So, in this context, the challenge is the synthesis and manufacture of nanosize oxide powders economically. Among several synthesis methods practiced worldwide, fire or combustion synthesis is very promising and a few salient features of this method are discussed.

2. Fire or Combustion Synthesis of Ceramic Materials

Fire is a source of heat and light. The well-known fire triangle has three components: an oxidizer, a fuel and ignition temperature (Fig.2).

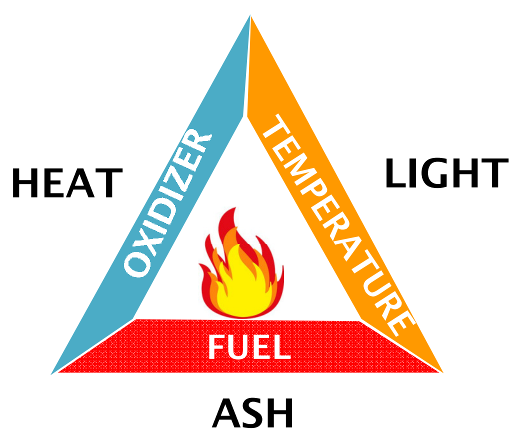

Fig.2. Fire triangle

An oxidizer is usually an electron acceptor like non-metal (oxygen, fluorine) or a compound containing oxidizing groups like nitrate or perchlorate. A fuel is an electron donor such as metal (carbon, hydrogen or hydrocarbon). Combustion is a redox (electron transfer) reaction which is exothermic. It can be smoldering (solid state decomposition or solid-gas reaction) or flaming (gas phase reaction). A redox compound like ammonium dichromate (containing both an oxidizer group and a reducing group) or metal hydrazine complexes which are fuel rich ignite at low temperature (<300 ºC) catch fire and burn autocatalytically in the air with evolution of gases to yield fine particle oxide materials. On the other hand, a redox mixture (metal and non-metal) ignites at high temperature (> 1000 °C) catches fire and burns with a flame (Temp. 3000 ºC) to yield refractory materials. Thus, the reactants could be in solids (SHS), liquid(SCS) or gas phase (Flame synthesis). Recently, Nersisyan et al have discussed various combustion synthesis (CS) methods for the preparation of 0, 1, 2 and 3-dimensional nanostructured materials. It is an excellent and comprehensive review covering all aspects of CS and its products including limitations, advantages and future prospects of each process. In the following section, the two commonly used combustion synthesis methods viz., self propagating high temperature synthesis (SHS) and solution combustion synthesis (SCS) have been briefly discussed.

2.1. Self-propagating High-temperature Synthesis (SHS) of Ceramic Materials

Self-propagating high-temperature synthesis (SHS) was the name first coined by Prof.A.G. Merzhanov, to describe highly exothermic processes involving the combustion of metals and non-metals. SHS, in its original form, pertained to a chemical reaction ‘‘bed’’ where ignition (Temp~ 1500˚C) was first initiated at one end with a heat source (heated wire, electric spark, laser beam, etc.). After ignition, the reaction front moves through the ‘‘bed’’ in a self-propagating mode (hence the process title) leaving behind solid products. The process is highly exothermic (Temp. 3000-4000 °C) yield refractory solid products like borides, carbides, nitrides, silicides etc. The materials synthesized by SHS process include carbides, borides, silicides, etc and are listed in Table-1 along with their applications. SHS is commercially developed in Russia to make ceramic materials. A handbook on SHS Research and Development, published in 1999 gives an account of work carried out around the world.

Table-1. SHS Materials and their Applications

Materials	Chemical Formula	Application
Carbides	TiC, SiC	Abrasives, cutting tools, ceramic reinforcements
Borides	TiB_2, LaB_6	Abrasives, Cutting tools, Cathodes
Silicides	$TiSi_2$, $MoSi_2$	Heating elements, electrical connectors, Schottky barriers for electronics
Aluminides and Titanites	AlNi, TiNi	Aerospace and turbine materials, shape memory alloys
Nitrides	NbN, Si_3N_4	Ceramic engine parts, ball bearings, nuclear safety shields
Hydrides	MgH_2, $ZrNiH_3$	Hydrogen storage and catalytic materials
Oxides	$YBa_2Cu_3O_{7-x}$, $La_{0.8}Cr_{0.2}CrO_3$	High temperature superconductors, gas sensors, fuel cells
Chalcogenides and Phosphides	Sulfur or phosphorus	High temperature lubricants, semiconductors

After ref. A. Verma, Scientific American, Form from fire, Scientific American, 2000, 283, pp.58-61

2.2. Solution Combustion Synthesis (SCS) of Ceramic Materials

In 1988, Patil and Kingsley from the Indian Institute of Science, Bangalore, India accidentally synthesized the high temperature form of alumina by the combustion of a solution containing stoichiometric quantities of aluminium nitrate (oxidizer, 20 g) with urea (fuel, 8 g). The solution containing the above redox mixture when rapidly heated around 500°C boiled, underwent foaming followed by a flame to yield voluminous alumina in few minutes (Fig.3). The exothermicity of the redox reaction of decomposition of nitrate and urea almost reached 1500°C. Combustion of above solution containing trace quantities of cobalt and chromium gave blue and pink colored alumina respectively (Fig.3). The high exothermicity of combustion was also further used to make rare earth ions doped oxide phosphors and noble metal ion substituted ceria and titania catalysts.

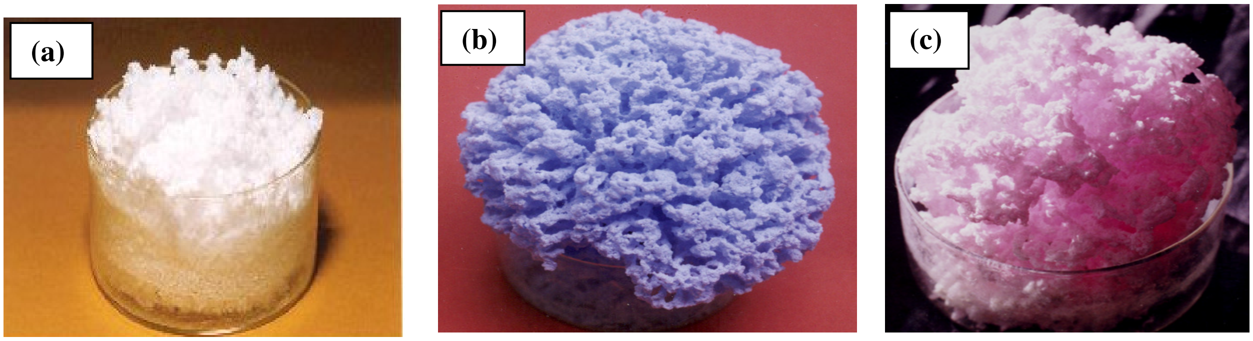

Fig.3. Solution combustion synthesized α-alumina (a) without any dopant (b) with Co dopant and (c) with Cr dopant.

To understand the highly exothermic nature of this reaction, concepts used in propellant chemistry were employed. A solid propellant contains an oxidizer like ammonium perchlorate and a fuel like carboxy terminated polybutadiene together with aluminum powder and some additives. The specific impulse (I_{sp}) of a propellant, which is a measure of energy released during combustion is given by the ratio of thrust produced per pound of the propellant. It is expressed as

$$I_{sp} = k\sqrt{\frac{T_c}{\text{Molecular Wt. of gaseous products}}}$$

The highest heat T_c (chamber temperature in the rocket motor) is produced when the equivalence ratio (ϕ_c = fuel/oxidizer ratio) is unity.

The equivalence ratio (F/O) of fuel and oxidizer mixture is expressed as follows:

$$\phi_c = \frac{\sum(\text{Coefficient of reducing elements in specific formula}) \times (\text{Valency})}{\sum(\text{Coefficient of oxidizing elements in specific formula}) \times (\text{Valency})}$$

A mixture is said to be stoichiometric when $\phi_c = 1$, fuel lean when $\phi_c > 1$, and fuel rich when $\phi_c < 1$. Stoichiometric mixtures produce maximum energy.

The fuel/oxidizer molar ratio (F/O) required for a stoichiometric mixture ($\phi_c = 1$) is determined by summing the total oxidizing and reducing valencies in the fuel and dividing it by the sum of the total oxidizing and reducing valencies in the oxidizer.

2.3. **Modified equivalence ratio**

In solution combustion calculations, the valency of the oxidizing elements was modified and considered as negative, and the reducing elements as positive, similar to the oxidation number concept familiar to chemists. Accordingly, the elemental valency of C, Al, and H is +4, +3, and +1, respectively, and oxidizing valency of oxygen is taken as -2. The valency of nitrogen is considered to be zero. Using the modified concept all types of oxide materials with the desired composition and structure were prepared. This has been illustrated below. In SCS metal nitrates are used as oxidizers and organic compounds like urea, glycine and hydrazides are used as fuels.

Based on the valency concept the oxidizing valencies of mono-, di-, tri-, and tetravalent metal nitrates are -5, -10, -15, and -20, respectively and the fuel, oxalic acid dihydrazide (ODH), $C_2H_6N_4O_2$ will have a reducing valency of +10. Accordingly, for a divalent $M(NO_3)_2$—ODH mixture the equivalence ratio (ϕ_e) is, 10/10 = 1.0 and for a trivalent $M(NO_3)_3$—ODH mixture it is 15/10 = 1.5. The mole ratios of metal nitrates and ODH become 1 : 1 for the preparation of AO type oxides, 1 : 1.5 for the preparation of A_2O_3 type oxides, 1 : 2 : 4 for the preparation of AB_2O_4 type oxides, 2 : 1 : 4 for the preparation of A_2BO_4 type oxides, and 1 : 1 : 3 for the preparation of ABO_3 type oxides. Such mixtures on rapid heating ignite and burn completely without leaving any carbon residue. Assuming complete combustion for some of these oxides, theoretical equations can be written as follows:

AO:

$$A(NO_3)_2(aq) + C_2H_6N_4O_2(aq) \rightarrow AO(s) + 2CO_2\,(g) + 3N_2(g) + 3H_2O(g)$$

where A = Mg, Zn, Ni, Cu, ...

A_2O_3:

$$2A(NO_3)_3(aq) + 3C_2H_6N_4O_2(aq) \rightarrow A_2O_3(s) + 6CO_2(g) + 9N_2(g) + 9H_2O(g)$$

where A = Al, Cr, Fe, ...

AB_2O_4 (Spinel):

$$A(NO_3)_2(aq) + 2B(NO_3)_3(aq) + 4C_2H_6N_4O_2(aq) \rightarrow AB_2O_4(s) + 8CO_2(g) + 12N_2(g) + 12H_2O(g)$$

where A = Mg, Mn, Co, Ni, Cu, Zn, B = Al, Fe, Cr, .

A_2BO_4 (K_2NiF_4 type):

$$2A(NO_3)_3(aq) + B(NO_3)_2(aq) + 4C_2H_6N_4O_2(aq) \rightarrow A_2BO_4(s) + 8CO_2(g) + 12N_2(g) + 12H_2O(g)$$

where A = La, ... B = Mn, Co, Ni, Cu, Sr, ...

ABO_3 (Perovskite):

$$A(NO_3)_3(aq) + B(NO_3)_3(aq) + 3C_2H_6N_4O_2(aq) \rightarrow ABO_3(s) + 6CO_2(g) + 9N_2(g) + 9H_2O(g)$$

where A = La, ... B = Al, Fe, Cr, ...

$A_3B_5O_{12}$ (Garnet):

$$3A(NO_3)_3(aq) + 5B(NO_3)_3(aq) + 12C_2H_6N_4O_2(aq) \rightarrow A_3B_5O_{12}(s) + 24CO_2(g) + 36N_2(g) + 36H_2O(g)$$

where A = Y,B Al, Fe, . . .

$AB_{12}O_{19}$ (Hexaferrites):

$$A(NO_3)_3(aq) + 12B(NO_3)_3(aq) + 19C_2H_6N_4O_2(aq) \rightarrow AB_{12}O_{19}(s) + 38CO_2(g) + 57N_2(g) + 57H_2O(g)$$

where A = Ba, Sr, ... B = Al, Fe, ...

It is interesting to note that the number of moles of ODH required for the combustion synthesis of oxides is equal to the number of oxygen atoms in the product, e.g., 4 mol of ODH for AB_2O_4

and A_2BO_4 type oxides and 3 mol for ABO_3 type oxides. This is true for any fuel with a reducing valency of +10 and metal nitrates as oxidizer. The combustion of metal nitrates-ODH mixtures is nonflaming, with evolution of gases yielding nanosized oxide materials.

The general scheme for the preparation of any oxide material is shown in Fig.4.

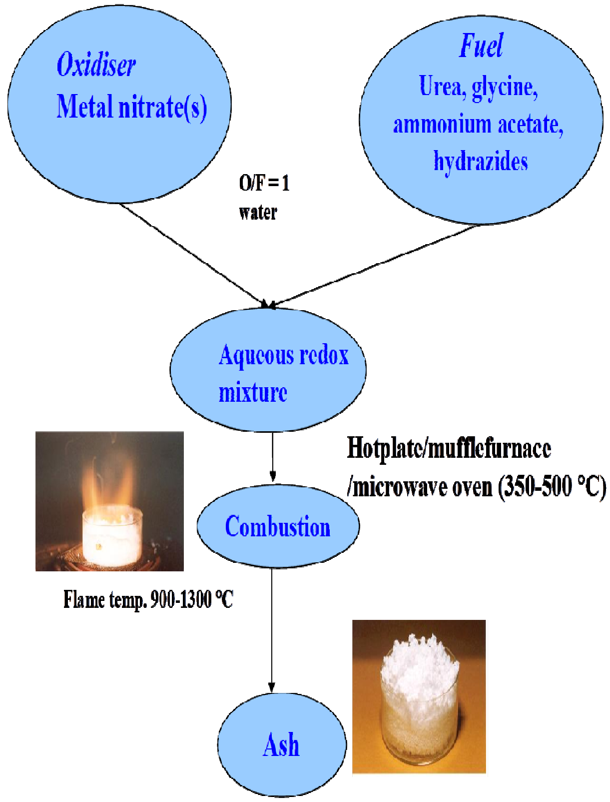

Fig.4. Flow-chart depicting the steps involved in solution combustion synthesis.

During the next decade and a half, the SCS process was standardized with respect to various processing parameters such as stoichiometry, the role of fuel, the source of ignition etc. Several innovations with the choice of fuels such as glycine, hydrazides, starch, sugar and mixed fuel were made. Fuels like carbohydrazide and oxalic acid dihydrazide were found to be suitable for making nanoscale oxide materials as the combustion was non-flaming (smoldering). All types of simple and complex metal oxides were prepared and charcterized. The properties and applications of nanocrystalline oxide materials prepared by SCS have been summarized in our book published in 2008.

2.4. Salient features of SCS

- SCS is a low temperature initiated, gas producing, self-propagating combustion process yielding voluminous, fine particle oxides which are sinteractive.

- It is a simple, fast and economically attractive method to make ceramic oxide materials.

- Being a solution process, it is possible to tailor-make any desired oxide with composition, structure, morphology, and property for a specific application.

- SCS enables to dope or incorporate atomic level metal ions in most of the oxide matrices to obtain phosphors, pigments and catalyst materials.

- It is an integrated approach (both breaking down and building-up) to make nanoscale oxide materials.

- It is a preferred method for making nanoceramic materials economically.

Some technologically important nanoceramic materials prepared by SCS are listed in Table-2.

Table-2. Technologically important nanoceramic materials, their properties and applications

Nanoceramics	Property	Applications
Al_2O_3, CeO_2,	Hardness	Abrasive
TiO_2, CeO_2, Fe_2O_3, M/Al_2O_3, $Ce_{1-x}M_xO_{2-y}$, Pt/TiO_2	Catalysts, photocatalyst	Catalysts, Three-way catalyst for auto exhaust
TiO_2, ZnO	UV-vis sunlight absorbing	Photocatalyst, Sun screen and paint
$BaTiO_3$, ZnO, Al_2O_3, PZT	Dielectric	Sensors, MEMS
γ - Fe_2O_3, $BaFe_{12}O_{19}$, MFe_2O_4	Magnetic	Cancer detection and remediation, Sensors and memory devices
TiO_2, Fe_2O_3, Cr_2O_3, MAl_2O_4, M/Al_2O_3, M/ZrO_2, $RE/ZrSiO_4$ (RE= Rare earth ion, M = transition metal ions)	Colours	Ceramic pigments
Eu^{3+}/Y_2O_3(Red), Eu^{2+}, Tb/Ba-Hexaaluminate	Luminescence	Phosphors CFL, Colour TV picture tube
Al_2O_3, ZrO_2, ZTA, Mullite, Cordierite, Tialite	Refractory	Toughened ceramics
MgO, CaO & ZnO	Adsorbent	Defluoridation & COD from paper mill effluents
YSZ(Y_2O_3-ZrO_2), Ni/YSZ La(Sr)MO_3, M=Mn, Cr $CaTiO_3$, $CaZrTi_2O_7$, $Ba_{1.23}Al_{2.46}Ti_{5.54}O_{16}$, $Ln_2Ti_2O_7$, NASICON ($Na_{1-x}Zr_2P_{3-x}Si_xO_{12}$, sphene ($CaTiSiO_5$) based titano silicate glass ceramic	Electrolyte Anode Cathode/Interconnect Sinteractivity and hydrothermal stability	Solid Oxide Fuel Cell Materials Radioactive waste immobilization materials SYNROC materials

2.5. Present status and future prospects of SCS

Today, SCS method of making ceramic materials has become very popular and practiced around the world as evident from the large number (over 2000 since 1988) of research papers published on this topic (Fig. 5) and their applications in different areas (Fig. 6).

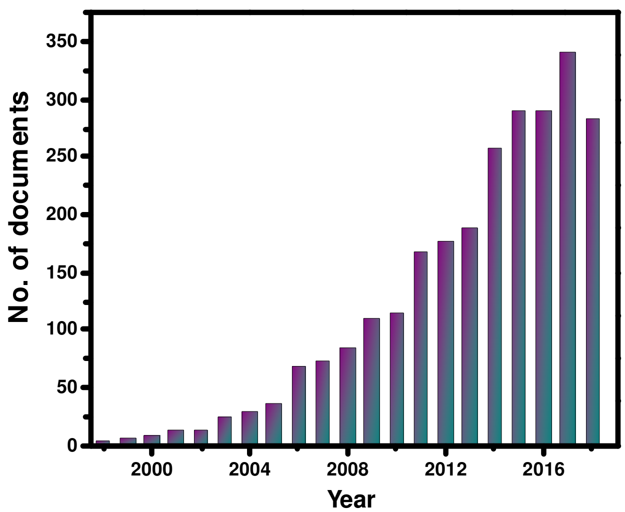

Fig.5. Scopus analysis showing increasing trend in the publication of SCS.

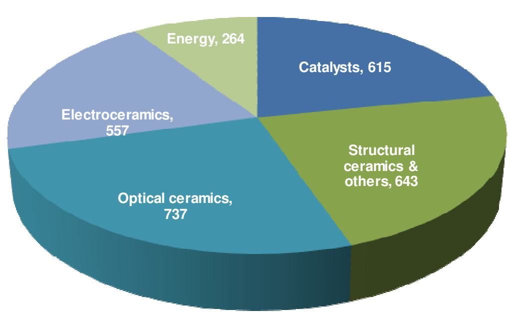

Fig. 6. Scopus analysis depicting the % of publications related to different applications.

In the last three decades, the development and progress made in SCS of ceramic materials have been phenomenal as seen by the number of highly cited publications on SCS (1-10). Innovations in the processing parameters of SCS like the choice of fuels and control of atmosphere has resulted in the formation of non-oxide materials like sulfides, metals, and alloys. Since the publication of our book in 2008, the number of publications and review articles has increased. The review articles (11-15) are devoted to applications of SCS derived nanoceramic materials in the generation of clean energy (fuel cells) and environment protection and remediation (TWC, defluoridation of water and synroc); catalysis (generation of hydrogen from biofuels and H_2-O_2 recombination catalysts) and optical materials.

Future of SCS appears to be bright and promising. Efforts should be made in making nanodevices with multifunctional chips. The large number of patents on SCS indicates the importance of nanoceramics to ceramic industry. The problems of handling nanoceramic powders and environmental pollution due to the emission of greenhouse gases like NOx and carbon dioxide during manufacturing need to be addressed. SCS continues to be a great challenge and provides a golden opportunity to young entrepreneurs.

Some highly cited (>200) publications and recent reviews on SCS (1988-2018)

1.J.J.Kingsley, K.C.Patil, A novel combustion process for the synthesis of fine particle α-alumina and related oxide materials, (1988) *Materials Letters,* 6 (11-12), pp. 427-432. **(No. of citations=538).**

2.Chick, L.A., Pederson, L.R., Maupin, G.D., Bates, J.L., Thomas, L.E., Exarhos, G.J., Glycine-nitrate combustion synthesis of oxide ceramic powders (1990) *Materials Letters*, 10 (1-2), pp. 6-12. **(No. of citations = 998).**

3.Shea, L.E., McKittrick, J., Lopez, O.A., Sluzky, E., Synthesis of red-emitting, small particle size luminescent oxides using an optimized combustion process (1996) *Journal of the American Ceramic Society*, 79 (12), pp. 3257-3265. **(No. of citations=262).**

4. Ye, T., Guiwen, Z., Weiping, Z., Shangda, X., Combustion synthesis and photoluminescence of nanocrystalline Y_2O_3:Eu phosphors. (1997) *Materials Research Bulletin,* 32(5), pp. 501-506. **(No. of citations=270).**

*5.*Patil, K.C., Aruna, S.T., Ekambaram, S., Combustion synthesis (1997) *Current Opinion in Solid State and Materials Science*, 2 (2), pp. 158-165. **(No. of citations = 354).**

6. Mimani, T., Patil, K.C., Solution combustion synthesis of nanoscale oxides and their composites (2001) *Materials Physics and Mechanics,* 4, pp. 134-137. **(No. of citations = 275).**

*7.*Patil, K.C., Aruna, S.T., Mimani, T., Combustion synthesis: An update (2002) *Current Opinion in Solid State and Materials Science*, 6 (6), pp. 507-512. **(No. of citations = 611).**

8.Aruna, S.T., Mukasyan, A.S., Combustion synthesis and nanomaterials (2008) *Current Opinion in Solid State and Materials Science*, 12 (3-4), pp. 44-50. **(No. of citations = 483).**

9.K.C. Patil, M.S. Hegde, Tanu Rattan, S.T. Aruna, Chemistry of nanocrystalline oxide materials (combustion synthesis, properties and applications); World Scientific, Singapore (2008) **(No. of citations =335).**

10.Hegde, M.S., Madras, G., Patil, K.C., Noble metal ionic catalysts (2009) *Accounts of Chemical Research*, 42 (6), pp. 704-712. **(No. of citations =205).**

11.Rajeshwar, K., De Tacconi, N.R., Solution combustion synthesis of oxide semiconductors for solar energy conversion and environmental remediation (2009) *Chemical Society Reviews*, 38 (7), pp. 1984-1998. . **(No. of citations =148).**

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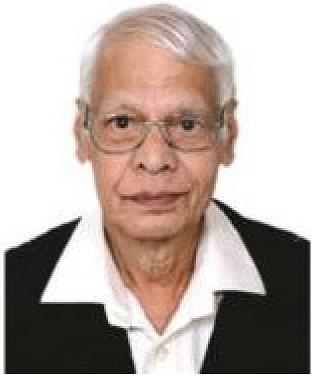
13. Li, F.-T., Ran, J., Jaroniec, M., Qiao, S.Z., Solution combustion synthesis of metal oxide nanomaterials for energy storage and conversion (2015) *Nanoscale*, 7 (42), pp. 17590-17610. **(No. of citations = 98).**

14. Varma, A., Mukasyan, A.S., Rogachev, A.S., Manukyan, K.V., Solution combustion synthesis of nanoscale materials (2016) *Chemical Reviews*, 116 (23), pp. 14493-14586. **(No. of citations = 141).**

15. Thoda, O., Xanthopoulou, G., Vekinis, G., Chroneos, A., Review of recent studies on solution combustion synthesis of nanostructured catalysts (2018) *Advanced Engineering Materials*, 20 (8), art. no. 1800047.

Please refer Part II for Bibliography of SCS of Nanoceramic Materials.

About the Authors



Prof. K.C. Patil is a Professor (Retd) in the Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bengaluru, India since 1972. He obtained his Bachelors and Master's Degrees from Karnatak University, Dharwad, Ph.D. from Indian Institute of Technology, Kanpur and D.Sc. from Indian Institute of Science, Bengaluru. In his long research career (over 50 years) he has contributed richly in the fields of solid state chemistry, inorganic synthesis, propellant chemistry, hydrazine chemistry and nanochemistry. His pioneering work on the solution combustion synthesis of nanoscale oxide materials has been widely recognized and practiced all over the world today.

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Dr. S.T. Aruna is currently working as a Principal Scientist at Council of Scientific and Industrial Research - National Aerospace Laboratories (CSIR-NAL), Bengaluru. She received her Bachelor's and Master's degrees from Bangalore University and Mysore University and Ph.D. from Indian Institute of Science. In her research career spanning two decades she has widely used solution combustion synthesized oxide materials for the development of electrodeposited wear and and corrosion resistant nanocomposite coatings and plasma sprayed functional coatings. She developed the concept of mixture of fuels approach and has tailored the solution combustion process for the preparation of flowable micron size particles for plasma spray application and nanosize powders suitable for suspension plasma spraying and tapecasting processes.

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