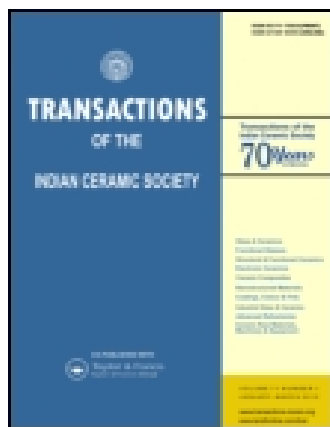


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Synthesis, Characterization and TL Studies of Porous CaSiO₃ Ceramic Powders

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Nanocrystalline porous CaSiO₃ ceramic powders have been synthesized by a novel low temperature initiated self-propagating, gas producing solution combustion process and characterized by XRD, SEM, EDS (energy dispersive spectroscopy), porosity, surface area and thermoluminescence (TL) studies. The effect of temperature on crystalline phase formation, amount of porogens and particle size of porous CaSiO₃ have been investigated. Single phase β-CaSiO₃ and α-CaSiO₃ were formed at 950° and 1200°C respectively. The phase transformation temperatures of combustion derived CaSiO₃ were found to be low compared to the powders obtained via solid state reaction method. The microstructure and morphology were studied by SEM and it was noted that with increase in calcination temperature, the samples became more porous and the pore diameter increased from 0.25 to 8 μm. The samples calcined at 950°C for 3 h had 17.5% porosity, however, the porosity increased to 31.6% on calcination at 1200°C for 3 h. The surface areas of the as-formed and calcined (at 950° and 1200°C) CaSiO₃ samples were found to be 31.93, 0.585 and 3.48 m².g⁻¹ respectively. The TL intensity in powder sample was more intense when compared to the pelletized CaSiO₃ and it was further observed that there was a shift in glow peak temperatures in pelletized sample. This is attributed to the interparticle spacing and pressure-induced defects.

[Keywords : Combustion synthesis, Macroporous CaSiO₃, Nanocrystalline, Ceramic powders, Thermoluminescence]

Introduction

In recent years materials with porous architecture and high surface area are being developed for numerous potential applications in nanotechnology and particular areas of interest including catalysis and separation science.¹⁻⁴ Traditionally, these solid materials with outstanding properties such as high porosity have been produced by discovery-based synthetic chemistry. The surface of the solid is the means by which it physically interacts with gases, fluids and other solids. Hence, the information on the textural features such as surface area and porosity of porous solids is essential. Porous ceramics have a wide range of applications in biomedical engineering, for example the fabrication of cranial plugs in bone reconstruction and the production of synthetic substrates for the fabrication of artificial corneas. There are various such applications, and various compounds that are in use: wollastonite (CaSiO₃) is one of them. In addition to this, CaSiO₃ has been traditionally used as a filler for resins or paper, as an alternative to asbestos,⁵ high frequency insulators⁶ and as machinable ceramics.

As a part of our programme on porous materials,⁷ we have synthesized a porous ceramic material (viz. CaSiO₃)

by a novel, low-temperature initiated, self-propagating, gas producing solution combustion process. This method involves an exothermic redox reaction of metal nitrates and an organic fuel. When comparing with other methods,⁸⁻¹¹ it is a simple, quick and inexpensive method involving a single-step reaction.

In this study, single phase β-CaSiO₃ and α-CaSiO₃ nanocrystalline ceramic powders have been prepared by solution combustion process and were characterized by XRD, SEM, Energy Dispersive Spectroscopy (EDS) and Thermoluminescence (TL) techniques. TL is a very common technique used for dosimetry of ionizing radiation for quite some time. Radiation processing using high doses, presents various advantages in industry (water purification, organic polymer materials crosslinking, pasteurization), medicine (radio-sterilization) and agriculture (disinfections, inhibition of sprouting). The increasing use of radiation in processes associated with industrial, medical and agricultural applications has motivated research on new materials with adequate dosimetric properties. To our knowledge, the dosimetric properties of nanocrystalline macroporous wollastonite have not been reported in literature. Therefore, the authors were interested to study the effect of radiation doses on the powder as well as on pelletized porous wollastonite samples.

Experimental

Calculation of Stoichiometry

The stoichiometry of the redox mixture used for the combustion process was calculated using the total oxidizing and reducing valencies of the ingredients which serve as

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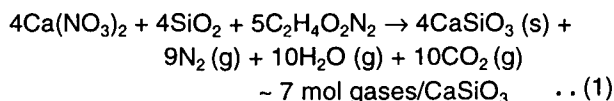
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numerical coefficients for the stoichiometric balance so that the equivalence ratio (ϕ_e) was unity and the energy released by the combustion was maximum.¹² According to the concept used in propellant chemistry the valence of C = +4, H = +1, divalent metal ions = +2, trivalent metal ions = +3 and so on and O = -2. The valence of nitrogen is considered to be zero. Based on these considerations, calcium nitrate has an oxidizing valence of -10 and diformyl hydrazine (DFH) a reducing valence of +8. The total valence of SiO₂ is zero. For the preparation of wollastonite, the required mole ratio of Ca(NO₃)₂·4H₂O : SiO₂ : DFH becomes 1 : 1 : 1.25.

CaSiO₃ ceramic powder was prepared by dissolving calcium nitrate and diformyl hydrazine (C₂H₄N₂O₂), a fuel for combustion synthesis, which was prepared in our lab by the reaction of formic acid and hydrazine hydrate, as described in the literature,¹³ in a minimum quantity of water in a cylindrical petri dish of 300 mL capacity. To this, fumed silica was added and dispersed well using a magnetic stirrer for half an hour. The heterogeneous redox mixture was rapidly heated in a muffle furnace maintained at 500° ± 10°C. DFH is a good fuel like other N-N bonded hydrazine derivatives, e.g. carbohydrazide (CH), tetraformal trisazine (TFTA), oxalyl dihydrazide (ODH), maleic hydrazide (MH) which are known¹⁴ to decompose to give NH₃, N₂, CO₂ and HNCO. These gaseous products along with oxides of nitrogen formed by the thermal decomposition of metal nitrates are hypergolic (spontaneously burst into flames) at 350°-500°C. The redox mixture when heated at 500°C boiled and thermally dehydrated forming a honeycomb like gel, which ignited to yield voluminous CaSiO₃ powder.

A theoretical equation assuming complete combustion of the redox mixture used for the synthesis of CaSiO₃ may be written as :



An X-ray powder diffractometer (Philips X'pert) using CuK_α radiation with a Ni filter was used to estimate the phase purity of the combustion derived CaSiO₃ powders. The surface morphology, size distribution of the grains and EDS of the sample were studied on a JEOL (JSM-840A) scanning electron microscope (SEM). The combustion products of CaSiO₃ (as-formed and heat-treated at 950°C) were ground well using a pestle and mortar. Pellets of 1 mm thickness and 6 mm diameter were prepared using polyvinyl alcohol as a binder and applying a pressure of 6 tons/ram of 6 mm diameter, using a home-made pelletizer at room temperature. The powder (1 mg) as well as the pellets (before and after heat-treated) were irradiated with gamma rays (Co⁶⁰ source) for a dose of 1-5 KGy. After irradiation TL studies have been carried out with a home-made TL setup described elsewhere.¹⁵

Results and Discussion

X-ray Diffraction

Figure 1 shows the phase evaluation of the solution combustion derived as-formed and calcined ceramic

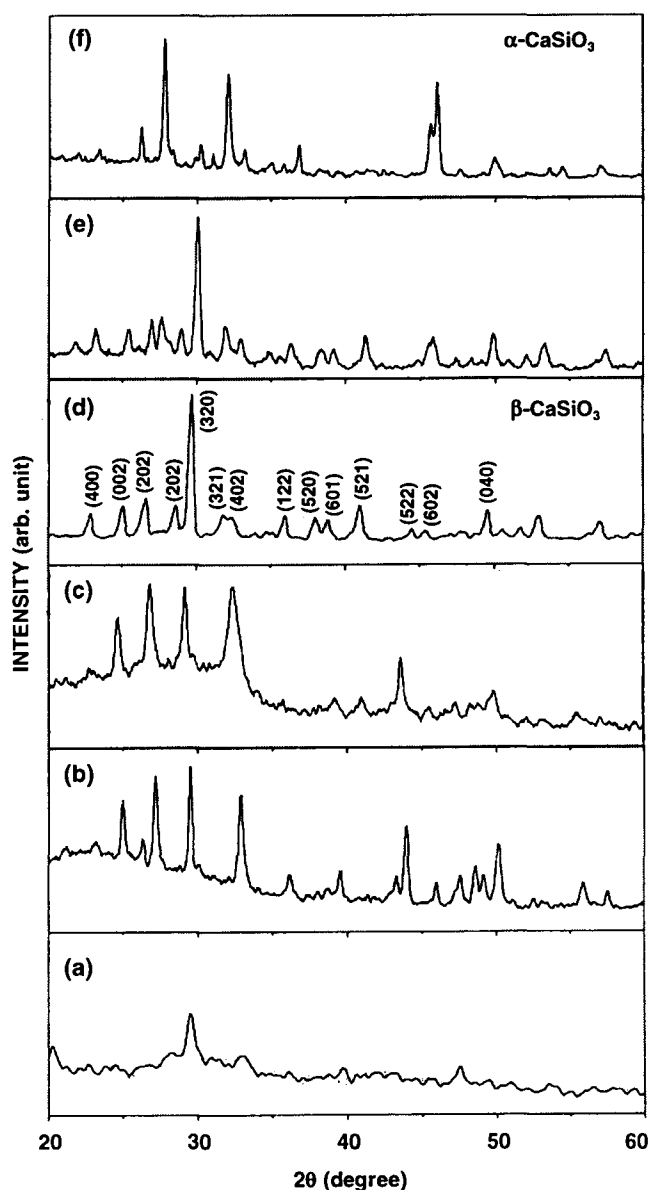


Fig. 1 – Powder XRD patterns of solution combustion derived wollastonite powders (a) as-formed (b) calcined at 500°C/3 h (c) 700°C/3 h (d) 950°C/3 h (e) 1100°C/3 h and (f) 1200°C/3 h.

powders of CaSiO₃ by XRD. As-formed CaSiO₃ powders show peak corresponding to mixtures of CaSiO₃ and CaO; on calcination the powders started crystallizing and gave rise to a single-phase β-CaSiO₃ (at 950°C for 3 h). The peak corresponding to CaO disappeared at 950°C. Therefore the samples calcined at 950°C indicated complete crystallization. No impurity could be detected, indicating a single phase of β-CaSiO₃ (JCPDS card-27-0088). It was also observed that the peaks of CaSiO₃ became more and more intense with increase in calcination temperature from 500° to 950°C. On further increase in calcination temperature from 950° to 1200°C, the simultaneous existence of CaSiO₃ in β and α phases could be observed at 1100°C. A complete phase transformation from β-CaSiO₃ to α-CaSiO₃ occurred at 1200°C. The peak positions of β and α phases are in

good agreement with those in the literature.^{10, 16} The phase transition temperatures of combustion derived CaSiO_3 powder were found to be low compared to the powder obtained in other methods,⁸⁻¹¹ for instance, about 600°C lower than the sample prepared by Kanzaki *et al.*⁸

The crystallite size of the ceramic powders was calculated from the full width at half-maximum (FWHM) of the diffraction peak of the powders from line broadening Scherrer's formula.¹⁷ The average crystallite size of the annealed CaSiO_3 samples was in the range 29-50 nm.

Scanning Electron Microscopy

The morphologies of the CaSiO_3 powders (a) as-formed (b) calcined at 950°C/3 h and (c) calcined at 1200°C/ 3 h, have been examined using SEM and are shown in Fig. 2. The SEM images exhibit some interesting results. It was observed that on calcination the hollow spherical CaSiO_3 powder underwent swelling and became more and more porous. The pore size of the wollastonite powders was in the range 0.25-1.5 μm for 950°C and 0.5-8 μm for 1200°C. The as-formed sample showed uniform distribution of nano particles except the samples calcined at 900°C/3 h and 1200°C/3 h which were quite agglomerated. Figure 3 shows the typical EDS of CaSiO_3 (950°C/3 h) and the nominal composition of the ceramic powders was confirmed by EDS.

Porosity Measurements

The bulk densities (D) of sintered (at 950°C/3 h and 1200°C/3 h) CaSiO_3 pellets were determined by the liquid displacement method. The percentage of porosity was calculated from the measured density according to the following equation:¹⁸

$$\text{Porosity} = (1 - D/D_T) \times 100 \quad \dots (2)$$

where D_T is the theoretical density (2.91 $\text{g}\cdot\text{cm}^{-3}$). It has been interesting to note that the porosity increased significantly with calcination temperature which could be seen clearly from the SEM images. The calculated porosity values were 17.5% and 31.6% at 950°C and 1200°C respectively. The powder densities were also measured by the standard procedure¹⁹ using a specific gravity bottle. The powder densities (1.73 $\text{g}\cdot\text{cm}^{-3}$ (as made), 0.89 $\text{g}\cdot\text{cm}^{-3}$ (calcined at 950°C/3 h) and 0.77 $\text{g}\cdot\text{cm}^{-3}$ (calcined at 1200°C/3 h)) of CaSiO_3 samples clearly exhibited the decreasing trend of density as calcination temperature increased. This might be due to expansion/swelling of the powders with calcination. This was also supported by the density measurements of pelletized samples (Table I).

Measurement of Surface Area

The surface area ($\text{m}^2\cdot\text{g}^{-1}$) of powder samples is related with other parameters such as particle size, shape, surface area textures. The surface areas of the CaSiO_3 samples prepared by combustion process were measured by a Quantachrome Corporation, NOVA 1000 Gas Sorption Analyzer using the Brunauer-Emmett-Teller (BET) equation.²⁰ The surface areas of as-formed CaSiO_3 and that calcined at 950°C/3 h and 1200°C/3 h were 31.93, 0.585

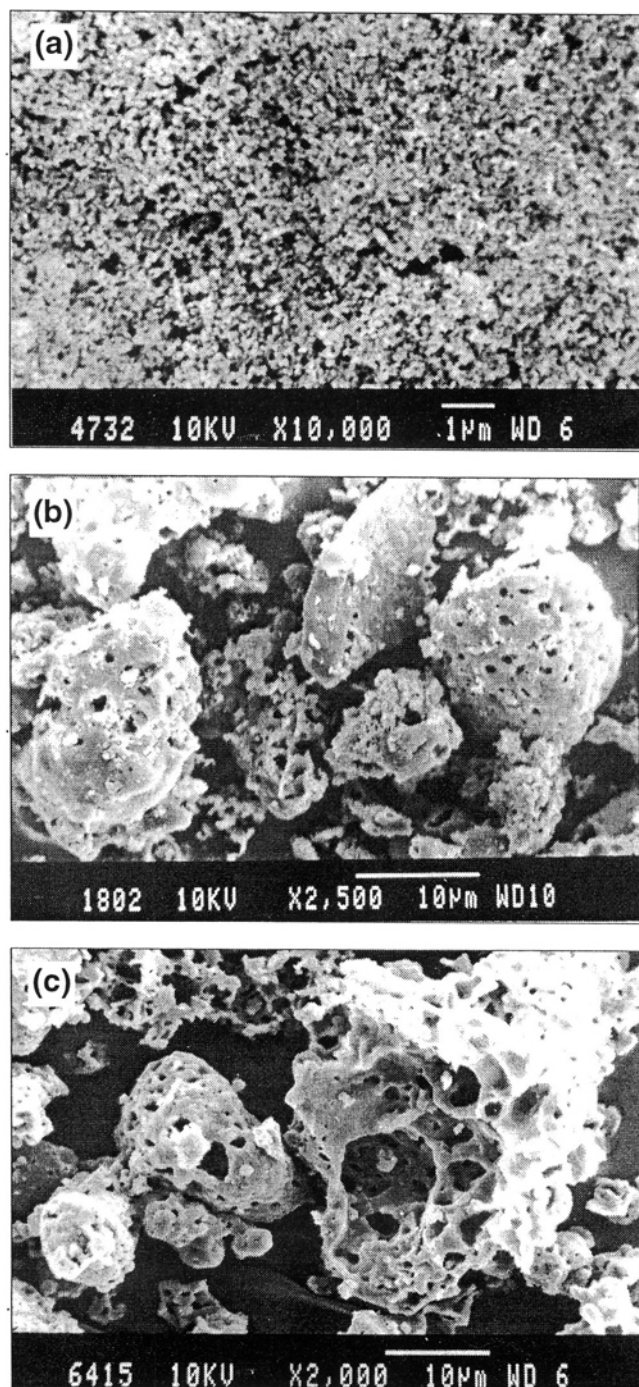


Fig. 2 – SEM images of solution combustion derived wollastonite powders (a) as-formed (b) calcined at 950°C for 3 h and (c) calcined at 1200°C for 3 h.

and 3.48 $\text{m}^2\cdot\text{g}^{-1}$ respectively. The large surface area (31.93 $\text{m}^2\cdot\text{g}^{-1}$) of as-made samples was due to uniform distribution of nano-sized particles as observed in SEM images and the same might be supported by data obtained from Scherrer's formula of XRD. On heating the sample at 950°C, the surface area decreased to 0.585 $\text{m}^2\cdot\text{g}^{-1}$; this might be due to the growth in particle size or agglomeration. On further calcination to 1200°C the surface area marginally

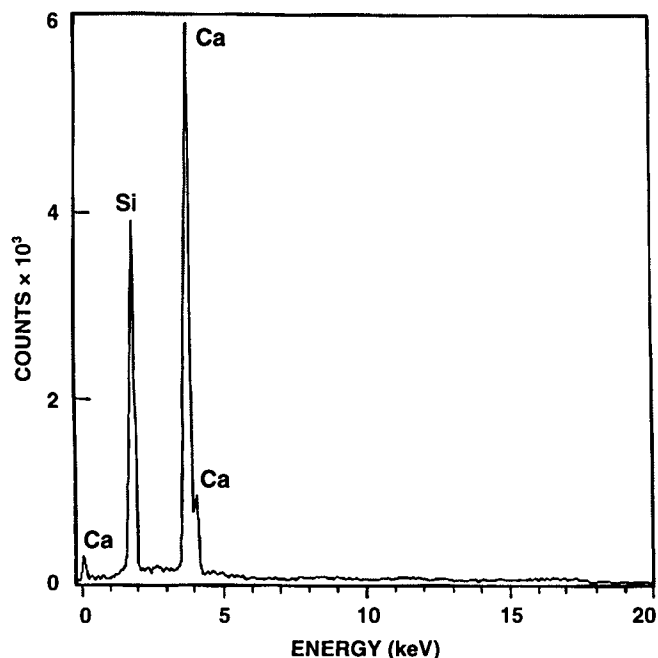


Fig. 3 – EDS of CaSiO_3 powder sample calcined at $950^\circ\text{C}/3\text{ h}$.

increased to $3.48\text{ m}^2\cdot\text{g}^{-1}$ which might be due to the swelling of agglomerated powder particles. These results are supported well by the density values as well as SEM images of the powders. The physical properties of wollastonite studied in the present work are given in Table I.

Thermoluminescence Studies

The values of TL intensity and the corresponding temperature were calculated from the experimentally recorded glow curves redrawn to the actual scale. Figure 4 shows the TL glow curves of as-prepared powder CaSiO_3 (1 mg)

Table I : Physical properties of wollastonite

Properties	Results
Powder density	
as formed	$1.73\text{ g}\cdot\text{cm}^{-3}$
at $950^\circ\text{C} / 3\text{ h}$	$0.89\text{ g}\cdot\text{cm}^{-3}$
at $1200^\circ\text{C} / 3\text{ h}$	$0.77\text{ g}\cdot\text{cm}^{-3}$
Bulk density	
at $950^\circ\text{C} / 3\text{ h}$	$2.41\text{ g}\cdot\text{cm}^{-3}$
at $1200^\circ\text{C} / 3\text{ h}$	$1.99\text{ g}\cdot\text{cm}^{-3}$
Surface Area	
as formed	$31.93\text{ m}^2\cdot\text{g}^{-1}$
at $950^\circ\text{C} / 3\text{ h}$	$0.585\text{ m}^2\cdot\text{g}^{-1}$
at $1200^\circ\text{C} / 3\text{ h}$	$3.48\text{ m}^2\cdot\text{g}^{-1}$
Particle size (by Scherrer's formula)	29-50 nm
Porosity	
at $950^\circ\text{C} / 3\text{ h}$	17.5%
at $1200^\circ\text{C} / 3\text{ h}$	31.6%

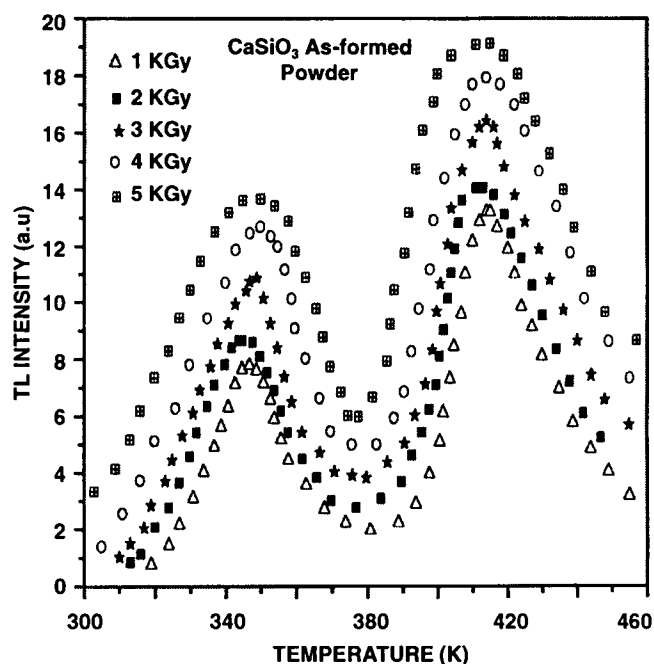


Fig. 4 – Thermoluminescence glow curve of γ -irradiated (1-5 KGy) as-formed CaSiO_3 powder sample.

sample irradiated with γ -rays for a dose of 1-5 KGy. Two well resolved TL glows, one at $\sim 349\text{ K}$ and another at $\sim 415\text{ K}$ were observed in all the samples. The effect of irradiation on any solid material was found to give rise to at least a pair of TL glow peaks arising due to recombination of two kinds of holes/electron deficiency trapping centres with at least one type of electron/electron donor centres. The TL glow curves of as-prepared pelletized CaSiO_3 samples irradiated for the same dose exhibited peaks at ~ 357 and $\sim 436\text{ K}$ at a warming rate of $20\text{ K}\cdot\text{min}^{-1}$ as shown in Fig. 5.

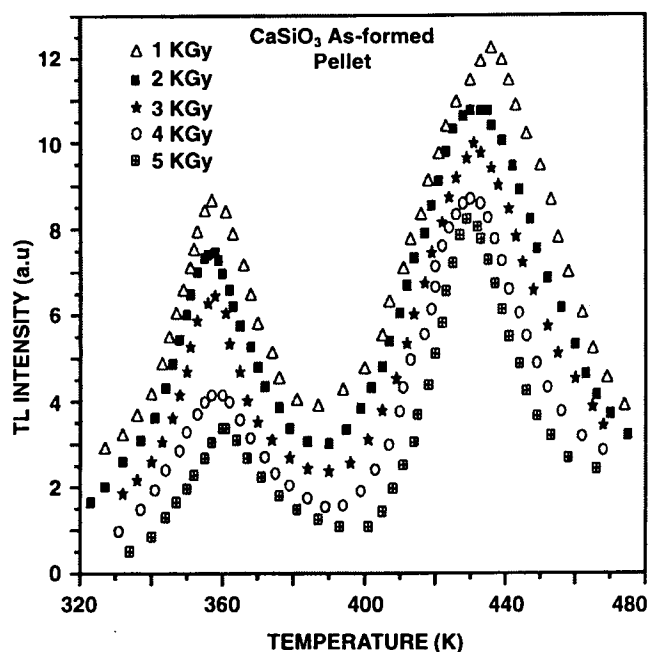


Fig. 5 – Thermoluminescence glow curve of γ -irradiated (1-5 KGy) as-formed pelletized CaSiO_3 sample.

The heat-treated CaSiO_3 powders ($950^\circ\text{C} / 3 \text{ h}$) irradiated with the γ -rays of 1-5 KGy exhibited two well resolved TL glow peaks at ~ 410 and ~ 479 K (Fig. 6). The same powder in pellet form also showed the peaks at ~ 454 and ~ 517 K (Fig. 7). It was observed that for powder samples, the TL intensity of both the glow peaks increased with increase of the dose of γ -irradiation. However, in the case of pelletized CaSiO_3 , the TL intensity of both the glow peaks decreased with increase of γ -irradiation dose. Further, the glow peaks in pelletized samples were found to be shifted towards higher temperature side. This is attributed to the interparticle spacing and pressure-induced defects.²¹⁻²³

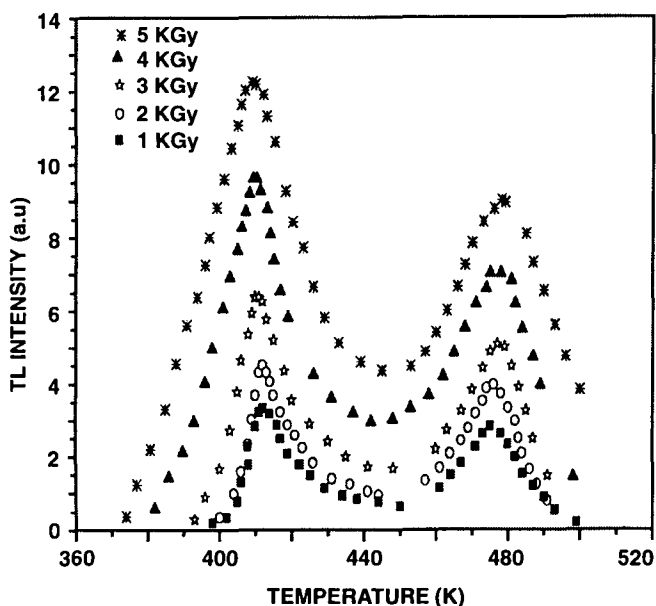


Fig. 6 – Thermoluminescence glow curve of CaSiO_3 heat-treated powder samples ($950^\circ\text{C}/3 \text{ h}$) and γ -irradiated for 1-5 KGy.

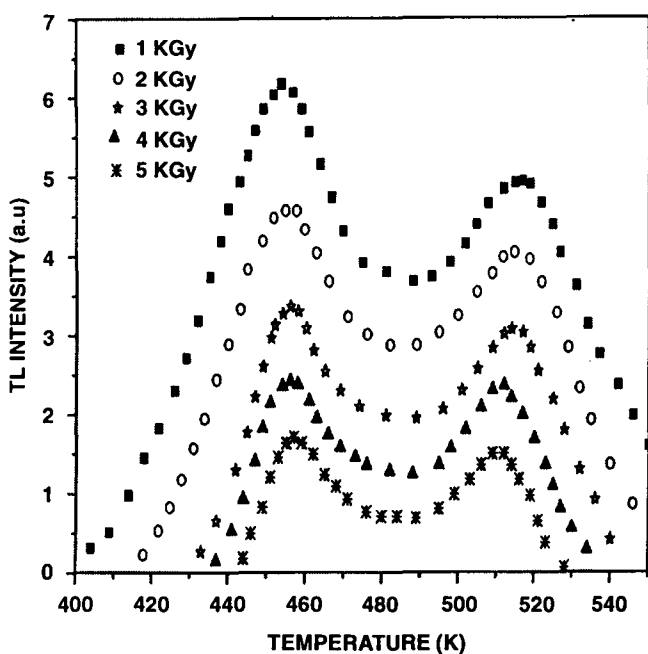


Fig. 7– Thermoluminescence glow curve of pelletized CaSiO_3 heat-treated ($950^\circ\text{C}/3 \text{ h}$) and γ -irradiated for 1-5 KGy.

The increase in TL intensity in powder samples might be attributed to the physical nature and structure of the samples.²¹⁻²³

Conclusions

Porous CaSiO_3 nanocrystalline ceramic powders have been synthesized by a novel low temperature initiated self-propagating, gas producing solution combustion process. Completely crystalline single phase β - CaSiO_3 and α - CaSiO_3 have been obtained at 950° and 1200°C respectively. The microstructure and morphology were studied by SEM and it is interesting to note that with increase of temperature the samples became more and more porous and the pore diameter increased from 0.25 to $8 \mu\text{m}$. The calculated porosity values were 17.5% and 31.6% at 950° and 1200°C respectively. The surface areas of as-formed CaSiO_3 and those calcined at $950^\circ/3 \text{ h}$ and $1200^\circ/3 \text{ h}$ were 31.93 , 0.585 and $3.48 \text{ m}^2.\text{g}^{-1}$ respectively. The large surface area ($31.93 \text{ m}^2.\text{g}^{-1}$) of as-made sample was due to uniform distribution of nano sized particles. On heating the sample at 950°C , the surface area decreased to $0.585 \text{ m}^2.\text{g}^{-1}$, which might be due to the growth in particle size or agglomeration. On further calcination to 1200°C the surface area increased to $3.48 \text{ m}^2.\text{g}^{-1}$, which might be due to the expansion/swelling of agglomerated powder particles. The TL intensity was more intense in powder sample compared to pelletized sample. This might be attributed to the physical nature and structure of the sample. The shifts in TL glow peaks as well as reduction in TL intensity of pelletized samples are attributed to inter particle spacing and pressure induced defects.

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