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EPR OF Cu^{2+} IONS IN MACRO POROUS NANOCRYSTALLINE WOLLASTONITE CERAMIC POWDERS

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In recent years materials with porous architecture and high surface area are being developed for numerous potential applications in nanotechnology, catalysis and separation science, molecular sieves, zeolites, hybrid optics, bio ceramics etc. We have synthesized porous wollastonite ($CaSiO_3$) and Cu^{2+} doped (0.1, 0.5, 1, 1.5, 2, 2.5, 3 and 5 mol%) ceramic powders by a novel low-temperature initiated, self-propagating, gas producing solution combustion process. The ceramic powders have been studied by XRD, TG/DTA, SEM, EDS and EPR spectroscopic techniques. The effect of temperature on crystalline phase formation, amount of porogens and particle size of porous wollastonite have been investigated. The XRD patterns confirms that the ceramic powders undergoes a clear single phase formation of β-$CaSiO_3$ and α-$CaSiO_3$ at 950 °C and 1200 °C respectively. These formation temperatures were lower than those using other methods by the conventional solid-state reaction method. It is observed that the average particle size of the annealed wollastonite samples are in the range 29-50 nm. The samples calcined to 950 °C has low porosity (17.5 %), however the porosity increases with calcination, and at 1200 °C has a large porosity of (31.6 %). The surface areas of as formed, 950 °C and 1200 °C calcined wollastonite samples were 31.93 m^2/g, 0.585m^2/g and 3.48 m^2/g respectively. The EPR spectra of all the investigated samples exhibit resonance signals characteristic of the Cu^{2+} ions. Various spin-Hamiltonian parameters have been evaluated from the EPR spectra and it is found that the ground state for paramagnetic electrons is d_{x2-y2} orbital ($^2B_{1g}$ state), implying that the Cu^{2+} ion being located in tetragonally distorted octahedral sites (D_{4h}) elongated along z-axis. The number of spins participating in resonance (N) and the paramagnetic susceptibilities (χ) have been evaluated from EPR data as a function of Cu^{2+} content. The effect of alkali ions (Li, Na and K) on the EPR spectra, have also been studied and it is interesting to observe that they exhibit a marked alkali effect. The results obtained from these studies have been discussed in detail.

Key words : Macro porous, nanocrystalline wollastonite, ceramic material, Electron Paramagnetic Resonance