Macroporous ceramics with pore size from 400 nm to 4 mm and porosity in the range 20%–97% are being used for a number of well-established and emerging applications, such as molten metal filtration, catalysis, refractory insulation and hot gas filtration. Such applications requiring porous ceramic structures have largely increased in the last decades, where high temperature, extensive wear and corrosive environment are involved. The advantages of using porous ceramics in these applications are their attractive properties such as high melting point, thermal stability, high corrosion resistance, good wear resistance and large surface area available on the surface and within porous structure to interact with atoms, ions and molecules therefore, can be used for multipurpose applications. Activated carbon, alumina, silicon carbide, zirconia and zeolites based foams fall in this category.

Keeping this in view a thorough literature survey was carried out on carbon and ceramic foams. It was found that most of researchers have used polyurethane (PU) foam as template. Two types of PU foams are available in the market. Therefore, it was thought of interest to analyze these two types of foams in order to develop reticulated carbon and silica foam with good permeability and strength. Also modeling of data was carried out to evolve the equation that will enable one to develop near net shape reticulated carbon and reticulated silica foam of required dimensions. The results of present exhaustive study are presented in the thesis. The thesis has been divided in to VII chapters.

Chapter I gives detailed introduction to the subject and related topics. It involves introduction to ceramics, their properties and applications. Also, brief introduction to various types of carbon materials and silica have been included. Various methods used for development of ceramic foams and their properties have been discussed in detail. Three procedures (i) replica, (ii) direct foaming and (iii) fugitive burn out have been used for developing foams. Early 1960s, Schwartzwalder and Somers [1] used polymeric sponges as template to prepare ceramic cellular structures of various pore sizes, porosities, and chemical
compositions. The replica technique is in fact considered as the first method used for production of macroporous ceramics. After that the sponge replica technique has become most popular method to produce macroporous ceramics due to simplicity, flexibility and control on pore shape and pore size for industrial applications i.e. as ceramic filters for molten metal and hot corrosive gases, light weight structural components, electrodes, heat exchangers, sensors, bioreactors, adsorbents, fuel cells, catalyst carriers, sound and thermal insulators, furnace door, roof and wall insulation, fire proof blocks, shields and coatings, safe rooms, radiant burners as well as porous implant in the area of biomaterials. [2-8]

Chapter II comprises of experimental procedures for development of carbon and functionalization of carbon foam and silica foam and the techniques used for their characterization.

Thermal stability of carbon foam was measured using thermo gravimetric analysis (TGA). The specific heat of carbon foam was measured using Differential Scanning Calorimetry (DSC). Co-efficient of thermal expansion (CTE) of prepared carbon foam was measured with Thermo Mechanical Analyzer (TMA) in nitrogen atmosphere up to 800 °C.

The surface area of carbon foam was measured with BET surface area analyzer (Micromeritics Gemini 2020). FTIR analysis was used to study functional groups present of carbon and silica foam at different heat treatment temperatures and change in surface oxygen functionalities on carbon foam surface with acid treatment time. A quantitative measurement of surface groups was carried out using Boehm’s titration method. The main intrinsic properties of carbon foam e.g. surface morphology like cell structure, size and shape of cell, wall thickness, etc. were studied by scanning electron microscope (SEM).

Porosity of carbon and silica foams was determined by kerosene porosity method. The gas permeability of carbon and silica foam heat treated at different temperatures was measured by an indigenous set up fabricated in the lab. The permeability studies were made
for different gases e.g. nitrogen, oxygen, helium, argon using different carrier gases and TCD
(thermal conductivity detector) as detector.

X – Ray diffraction pattern were taken using Philips X-ray diffractometer (X’pert model). The crystalline phases, crystallite size, degree of graphitization and La (length in a
direction) were determined by analyzing the X-ray diffraction pattern. The compressive
strength of foams was measured by INSTRON 5500R.

The results of characteristic properties of polyurethane (PU) foams and phenolic resin
used are given in the chapter III. Different types of PU foam available were characterized for
their density. High density foam was selected for further processing to carbon foam as to
have good mechanical strength. Two types of PU foam viz. polyether and polyester available
were also analyzed for their properties. The detailed results of their analysis are given in this
chapter. Phenolic resin was used as carbon precursor for impregnation of PU foam. The
carbonization behaviour of phenolic resin and its yield percentage were studied.

Chapter IV includes results of characteristic properties to enable further processing of
impregnated foam heat treated at different temperatures e.g. 200, 400, 600, 800, 1000, 1200,
1400, 1600 °C etc. During carbonization of impregnated foam, non carbon elements such as
nitrogen, hydrogen and oxygen present in the resin get removed as volatiles, such as H2O,
CO, CO2, H2. Due to decomposition of resin, density of carbon foam decreases as the heat
treatment temperature is raised. On the other hand it was found that porosity of carbon foam
decreases with increase in the heat treatment temperature.

Specific heat of carbon foam heat treated at 1000 °C was 0.3 cal/g.°C. CTE
(co-efficient of thermal expansion) of carbon foam at 800 °C in nitrogen atmosphere was
1.41 ppm/°C. These properties make carbon foams suitable for utilization for many
engineering applications i.e. power electronics, phase change devices, etc.
SEM micrographs taken for carbon foam show well organized cell structure without any cracks and stress-induced failure in the carbon foam. The pore wall thickness varies between 60 µm to 100 µm. The diameter of pores of carbon foam decreases with increase in heat treatment temperatures. The pore diameter varies from approximately 200 µm to 500 µm.

The measured BET surface area of heat treated carbon foam at 1000 °C was 30 m²/g and at 1600 °C was 23 m²/g. Since size of nitrogen molecule is 0.162 nm² which is much smaller as compared to the size of foam pores. Therefore during adsorption of nitrogen no condensation take place in macropores, which account for smaller value of surface area.

Gas permeability was measured using an indigenous set up. Different gas samples like nitrogen, helium, argon, hydrogen, air, etc. were used to determine permeability of porous samples. Results show that percentage permeability through carbon foam varies from 99.6 to 97.6 % for different gases thus showing interconnected porosity.

XRD pattern of carbon foam heat treated at 1000 and 1600 °C shows amorphous nature of carbon foam. Reticulated vitreous carbon (RVC) structures, also called turbostract carbon (TC), have their microstructure formed by disordered, no planar and confluent graphitic ribbons. Above 1000 °C the crystalline structure was lengthened and broadened. Therefore, the Lc and La value increased with an increase in heat treatment temperature.

Compressive strength measurement show higher value of compressive strength as 0.61 MPa for carbon foam heat treated at 1000 °C. It is seen that compressive strength of carbon foam increases with increase in the relative density. It was seen that carbon foam shows a brittle failure mode undergoing successive cell wall fractures during testing of compressive strength.
The oxidation resistance of carbon foam was found to increase with increase in heat treatment temperature. This shows that these foams can be used up to temperature of 550 °C in air.

The percentage resin impregnation into PU foam was also varied to optimize the overall volume shrinkage, change in density, porosity and mechanical properties of carbon foam. Carbon foam of various densities (e.g. 0.04 to 0.26 g/cc) was prepared. Reaction parameters were optimized to get desired density carbon foam.

Statistical analysis was carried out by using SPSS software for optimization of parameters such as dimensional changes and controlled density carbon foam.

Functionalization of prepared carbon foam and their characterization has been described in chapter V. The functionalization was done by oxidation with nitric acid. The surface oxygen complexes developed were characterized by FTIR analysis and their quantitative determination was made by Boehm’s titration. The presence of groups such as –COOH, -OH, C=O and C-O-C were found on surface of carbon foam. Oxidation of carbon foam beyond nine hours with nitric acid resulted in loosening of structure of foam and cracks formation within.

Chapter VI describes method for development of silica foams and their characterization. Silica foams were prepared by using PU foam as template and silica sol as precursor for silica. Sol-gel route was used to prepare silica sol. Silica sol was impregnated in PU template to prepare silica foam. Sol impregnated dried foams were heat treated at 1000 °C at slow heating rate to retain the porous structure of silica foam. Density of silica foam obtained was 0.13 g/cc. It was seen that density can be controlled by amount of silica sol amount impregnation. On heat treatment of foam at high temperatures, porosity as well as permeability decreases.
SEM micrograph showed obtained silica foam has open cell and well organized cell structure without any cracks and stress-induced failure. The diameter of strut varied from 60 to 120 μm. Pore size varies from 100 to 450 μm.

The measured BET surface area of silica foam heat treated at 550 °C was 101 m²/g with 4.56 % microporosity. XRD results shows that as heat treatment temperature increases, the tridymite phase is transformed into cristobalite phase. Compressive modulus of silica foam sintered at 1000 °C was 6 MPa.

The silica foam showed excellent thermal stability as there was no weight loss on heating in air. Silica foam though porous, showed very good insulation properties. The extent of insulation depends on thickness of silica foam. It was seen that 25 mm thick silica foam when exposed to 1100 °C from one side, temperature on the other side was 170/175 °C. Studies on corrosion resistance of prepared silica foam showed very good resistance against concentrated acids and dilute bases for more than 6 months without losing their structural stability and weight.

Statistical analysis was carried out by using MINITAB software for optimization of parameters such as dimensional changes and controlled density silica foam.

Important conclusions drawn from the present studies and about future scope of work is given in chapter VII. It can be concluded that both carbon foam and silica foam having highly interconnected porosity can be developed by using template method. The chemical composition and pore structure of template used is very important and play a vital role. Near net shape foams having desired porosity can be prepared by controlling parameters i.e. concentration of resin, heating rate and number of impregnation cycles. It is seen that ester based PU foam and ether based PU foam provide better properties in terms of permeability and compressive strength of carbon foam while for silica foam ether based PU template provide better strength.
References: