
Chapter 2

Literature Review – Processing of Porous Ceramics

Porous ceramics are being fabricated via different processing routes, depending on the requirements of the properties and corresponding applications. Properties can be tailored for each specific application by controlling the composition and microstructure of the porous ceramics. Changes in open and closed porosity, pore size distribution, and pore morphology can have a major effect on a materials' properties. All these microstructural features are in turn highly influenced by the type of processing route used for production of the porous material.

Generally, the microstructure and associated properties of porous ceramics are largely dependent on the type of pore forming processes. The common approach of all fabrication techniques is to incorporate air or gas phase intentionally in to the material either in the liquid state or in the solid state of the material. Preparation of porous ceramics through entrapment of air or gas in the molten state is an uncommon option unlike processing of metals, considering the tremendous difficulties as well as high cost involved during handling and processing. Thus, the only option towards preparation of porous ceramics is through powder processing route. The same is true for fabrication of dense ceramic components.

In this chapter, we will review the main fabrication techniques through powder processing routes that are being used or can be used for fabrication of porous ceramics with controlled microstructure. These ultimately determine the properties of the porous ceramics. Fabrication of ceramics through powder processing essentially involves packing of and compaction of powder particles which are eventually sintered. Since the particles can never be compacted to full density even after considering the possibility of maximum achievable packing of the green products, the powder processing techniques result in green bodies typically with 30-40% porosity and these porosities are eliminated during firing.

Thus, the most straight forward processing route for preparation of porous ceramics is the partial/incomplete sintering of the initially porous green powder

compacts. Also, the sintering of powder mixtures which undergo solid state reactions which lead to pore formation is another method.¹¹ In this process a part of the porosity is retained through partial sintering. This method often results in a relatively low porosity (< 60%), with pores homogeneously distributed within the microstructure. The limitation of such a process is that there is not much choice over the pore size range as well as the percent porosity.

In addition to such a straight forward approach, many novel methods for the preparation of porous ceramics with controlled microstructure have been developed in response to the increasing number of new potential applications for porous ceramics.¹² Versatile techniques that allow one to deliberately tune the porosity, pore morphology, pore size distribution, and which can additionally be applied to ceramic materials of many different chemical compositions are especially demanded.

Table 2.1 Processes for making ceramic foams

Processes	Classification		Porosity (vol%)	Pore size (μm)
Partial sintering	-		60	0.5-5
Solid state particle bonding	Reaction bonding		50	0.1-10
	Oxidation bonding		50	0.1-10
	Bonding with fibrous precursors		50	0.1-10
Replication of 3-D preforms	Slurry infiltration	Synthetic templates	60 – 96	1-5000
		Natural templates	43 – 92	10-500
		Other methods	94-97	50-350
	Chemical vapour infiltration		70 – 95	10-5000
Sacrificial template method	Natural organics		20-90	1-700
Direct foaming	Stabilization with surfactants		40-97	1.2-10
	Stabilization with particles		40-93	10-300
Other methods	Sol-gel		10-90	0.15-2
	Freeze drying		20-90	2-200

Various processing routes can be classified into main categories like solid state particle bonding, replica method through 3-D preforms, sacrificial template,

incorporation of fugitive method, direct foaming method, etc. Table 2.1 gives a brief classification of various processes adopted for making porous ceramics.

2.1. Partial Sintering

Partial sintering is one of the easiest methods for preparation of porous ceramics. The percent porosity, pore size and pore morphology change continuously with the extent of sintering. In this case the pore size cannot be altered (Fig. 2.1).¹³⁻¹⁴

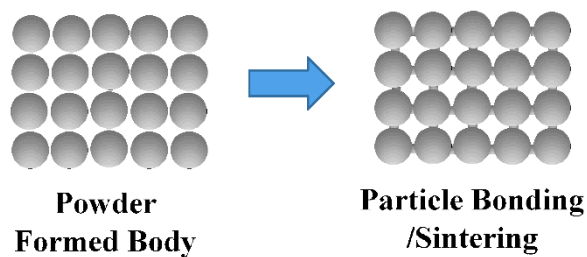


Fig. 2.1 Representative fabrication process of porous ceramics¹¹¹

Another innovative processing technique demonstrated by Deng et al.¹⁵, combines partial sintering with use of a corresponding hydrated oxide that undergoes a volume contraction during heat treatment leaving additional porosity in the partially sintered compact. Compacts made of alumina powder and $\text{Al}(\text{OH})_3$, during heat treatment resulted in porous alumina having porosity up to 62%. $\text{Al}(\text{OH})_3$ powder was mainly composed of various minerals like bayerite ($\alpha\text{-Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) in major amount and boehemite ($\gamma\text{-Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$), nordstrandite ($\text{Al}(\text{OH})_3$) in minor quantity. A phase transformation accompanied by 60% volume contraction occurred during heat treatment of the sample and finally at around 1300°C , a stable α -alumina phase is formed. Porosity of samples increased with increase in volume fraction of $\text{Al}(\text{OH})_3$ powder in the composition and decreased with increase in sintering temperature. Further increase in porosity was obtained by suppressing densification through addition of ZrO_2 powder in the composition.¹⁶

Another process similar to the above produces porous ceramics up to 30 % porosity and pore size in the range $0.5\text{-}5\ \mu\text{m}$.¹⁷ In this process seeded boehemite ($\gamma\text{-Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) sol was mixed with coarse $\alpha\text{-Al}_2\text{O}_3$ and gelled by dehydration. The

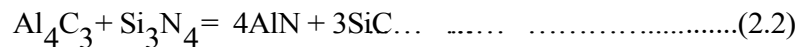
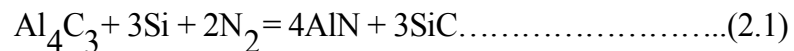
dried gel was ground into powders. The dried powders were dispersed in ethanol followed by drying and then grinding. The ground powders were compacted and sintered in the temperature range 1200-1600°C. In this case also, during sintering, boehmite undergoes a phase transformation accompanied by 36% volume shrinkage and finally forms a porous structure with fine α -Al₂O₃.

2.2. Solid State Bonding

2.2.1. Reaction bonding

This is a new technique based on low temperature bonding of silicon carbide powder particles through an in situ generated chemical bonding agent.¹⁸ The bonding agent is derived from pyrolysis of a pre-ceramic polymer. The complete processing can be carried out at comparatively low temperature (1100°C) in contrast to the high temperature (>1800°C) that is generally required for processing of silicon carbide. The pyrolysis products formed at 1100°C in nitrogen atmosphere were responsible for bonding the particles. The percentage porosity ranged from 45 – 52 %. Initial size of the starting powder played a key role in controlling the pore size of these materials.

Another process¹⁹ where porous AlN-SiC composites were prepared using Al₄C₃ and either Si or Si₃N₄ in nitrogen atmosphere. The following two reactions take place:



The compacts undergo complete conversion to AlN-SiC in the temperature range 1700-1800°C. Samples with porosity in the range 36-42% were obtained in this process.

2.2.2. Oxidation bonding

She et al. developed a simple technique for preparation of porous silicon carbide.²⁰⁻²¹ The basic idea was to develop bonding between silicon carbide particles through formation of a surface oxide (grown in a controlled manner by

heat treatment in air at 1100-1400°C). Initial particle size of the powder and processing temperature control the porosity and pore size in this technique. The porosity decrease with increase in processing temperature and with the use of coarser particles. Use of graphite in this process improved the properties of final compacts.

2.2.3. Bonding of fibrous precursor

A novel technique for fabrication of porous hydroxyapatite (HA) is based on bonding of HA whiskers.²² In this process porosity is incorporated into the sample because of incomplete packing of the whiskers. The porous microstructure is obtained simply by hot pressing the HA whiskers in the temperature range 800-900°C. The porous structure obtained is more tough due to the presence of whiskers in comparison to the powder based compacts.

2.3. Replication of 3-D Preforms

The replica method is based on the impregnation/infiltration of a porous structure with a ceramic suspension or precursor solution in order to produce a porous ceramic exhibiting the same morphology as the original porous material (Fig. 2.2). The method can be carried out either through slurry infiltration or through chemical vapour infiltration. Many synthetic and natural porous structures can be used as templates to fabricate porous ceramics through the replica technique.

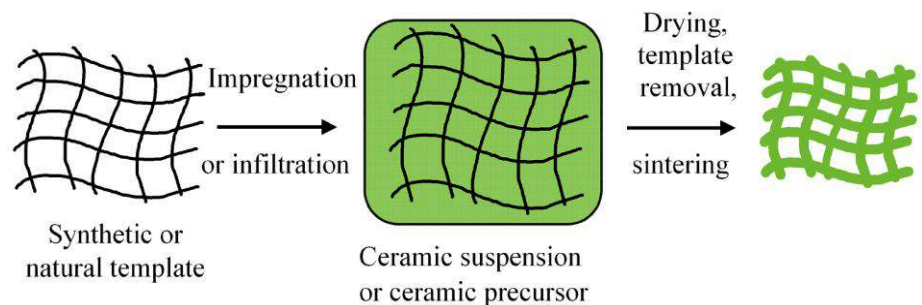


Fig. 2.2 Replica processing routes used for the production of porous ceramics¹¹

2.3.1. Slurry infiltration

A. Synthetic templates

The original invention was based on using polymeric sponges as templates to prepare ceramic porous structures of various pore sizes, porosities, and chemical compositions. Since then the sponge replica technique has become the most popular method to produce porous ceramics and today this technique is extensively used in industry to prepare ceramic filters for molten metal filtration and other applications.²³

In polymer replica approach, a highly porous polymeric sponge (typically polyurethane) is initially soaked or infiltrated with a ceramic suspension until the internal pores are filled with the ceramic material. The impregnated sponge is then passed through rollers to remove the excess suspension and enable the formation of a thin ceramic coating over the pores of the original porous structure and open pore channels were left in between. The ceramic-coated polymeric template is subsequently dried and pyrolysed through careful heating between 300-800°C.¹¹ After removal of the polymeric template, the ceramic coating is finally densified by sintering in an appropriate atmosphere at temperatures ranging from 1100-1700°C depending on the material type. The great flexibility of the method is partly because of the fact that it is applicable to any ceramic material that can be appropriately dispersed into a suspension. Various SiC based porous ceramics have been produced by replacing the ceramic suspension with preceramic polymers.²⁴

Porous ceramics obtained in this method can reach total open porosity in the range 40-95% and are characterized by a reticulated structure of highly interconnected pores with size between 200 μm and 3 mm. The ratio of open to closed pores in the final porous ceramics may be adjusted to a certain extent by controlling the suspension viscosity. A disadvantage of the sponge replica method is the fact that the struts of the reticulated structure are often cracked during pyrolysis of the polymeric template, degrading the final strength of the porous ceramics.²⁵ Many attempts have been made to avoid this shortcoming by improving the wetting of the suspension on the sponge with the help of additives,²⁶⁻²⁷

performing a second impregnation step to fill the cracks in the ceramic struts,²⁸ and introducing fibers or reactive compounds²⁹ to enhance the materials integrity. In contrast to ceramic-suspension derived reticulated structures, porous ceramics obtained from preceramic polymers have crack-free struts due to most likely to the improved wetting on the sponge.³⁰

B. Natural templates

In addition to synthetic polymer templates, other porous structures have been used as templates for the fabrication of porous ceramics through the replica approach. Porous/cellular structures available in nature are particularly interesting as natural replica templates mainly due to their special pore morphology and intricate microstructures, which may be difficult to produce artificially.

Corals have long been applied as a natural template for the preparation of scaffolds for tissue engineering and bone replacement.³¹ The cellular structure of corals has also been directly converted into porous hydroxyapatite scaffolds by submitting the marine skeleton to hydrothermal treatments at high temperature and pressure (Fig. 2.3).¹¹ This treatment is performed in a phosphate solution so that the carbonate ions from the aragonite material (CaCO_3) originally present in the coral are partially or totally replaced by phosphate ions to form hydroxyapatite.³²

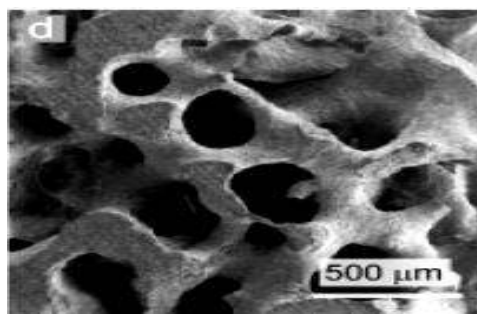


Fig. 2.3 Porous hydroxyapatite obtained from the coral structure¹¹

Many investigators have studied the transformation of wood cellular structures into porous ceramics (Fig. 2.4). The presence of oriented vessels in the structure of wood enables the preparation of porous ceramics with highly anisotropic aligned pores which cannot be achieved with other replica techniques.³³ Fig. 2.4 illustrates the processing routes that can be applied to transform wood

structures into cellular ceramics. The most common approach is to first prepare a carbon cellular preform by heat treating the wood structure under an inert atmosphere at temperature within 600-1800°C. Carbon preform is subsequently infiltrated with gases or liquids at high temperature to obtain the porous ceramic. Alternatively, carbon preform is infiltrated at room temperature with liquid sols and afterwards oxidized to give cellular ceramics (Fig. 2.4).

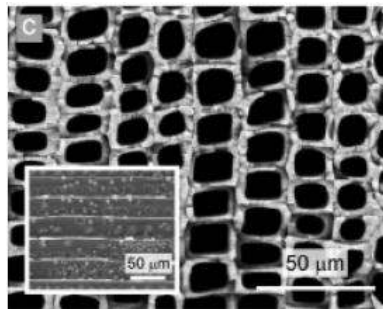


Fig. 2.4 Transversal view of a highly oriented SiC porous ceramic obtained after infiltration of a wood template with Si gas¹⁰

Pore size distribution of wood-derived porous ceramics is determined by the microstructure of the original wood template used. As wood structures may exhibit cell size down to a few micrometer, porous ceramics obtained from wood templates display pore size markedly lower than those achieved with the polymer replica technique, typically within the range 10-300 μm . Porosity of these structures is predominately open and can vary from 25% to 95% depending on the amount of material impregnated into the template. The anisotropic nature of porous ceramics produced using wood as template is advantageous in applications that require open and highly oriented porous structures such as in catalysis and in the filtration of liquids and hot gases. In the case of sponge replica method, the walls of the wood-derived structures might contain flaws/pores generated from the pyrolysis of residual carbon in oxidizing environments. Such pores degrade the mechanical strength of the porous ceramics. Another disadvantage of the wood-replica approach is the several time-consuming steps involved which might add considerable cost to the process.

C. Other methods

Numerous other approaches using synthetic³⁴ or natural biomorphic templates such as plants³⁵, egg shell membrane³⁶⁻³⁷, bacteria³⁸ and bioclastic structures formed by aquatic organisms(e.g., diatoms)³⁹ have also been applied in the last few years to fabricate porous particles, films and unique 3D porous structures up to 100 μm in size.

2.3.2. Chemical vapor infiltration

Porous ceramics have also been prepared from carbon preforms derived from synthetic porous structures produced from thermosetting polymers. Different hybrid cellular structures have been produced by directly depositing ceramic materials (oxides, carbides, borides, silicides) on the carbon skeleton using the chemical vapour deposition/chemical vapour infiltration process (CVD/CVI). Deposited material densities of up to 50% of theoretical values have been achieved.

Sherman et al.⁴⁰ developed a process which is similar to the original replica method where the polymeric sponge was first converted into a vitreous skeleton and subsequently infiltrated with reactive gaseous species to form porous ceramics of many different materials. SiC based porous ceramics of diverse compositions were fabricated by Greil and colleagues⁴¹⁻⁴² through the infiltration and reaction of gaseous silicon metal with the carbon preform.

2.4. Sacrificial Template Method

This technique usually consists of preparation of a biphasic composite comprising a continuous matrix of ceramic particles or ceramic precursors and a dispersed sacrificial/organic phase (pore forming agent) that is initially homogeneously distributed throughout the matrix and is ultimately extracted to generate pores within the microstructure (Fig. 2.5).

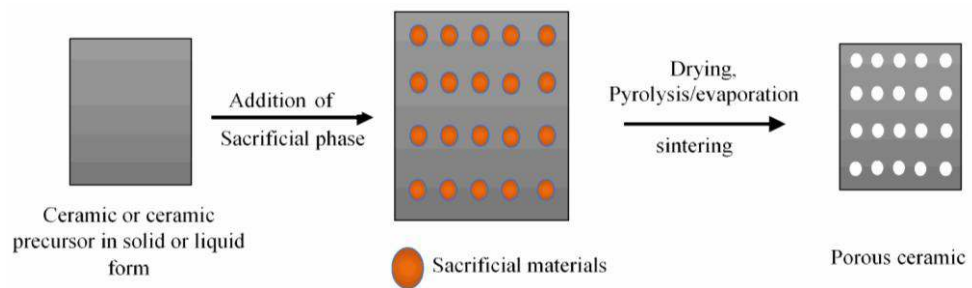


Fig. 2.5 Sacrificial template processing route used for the production of porous ceramics.¹¹

This method leads to porous materials displaying a negative replica of the original sacrificial template as opposed to the positive negative replica of the original sacrificial template as opposed to the positive morphology obtained from the replica technique described above. The biphasic composite is commonly prepared by various methods such as: (a) pressing a powder mixture of the two components⁴³ (b) forming a two phase suspension that is subsequently processed by wet processing and casting¹² or impregnating previously consolidated preforms of the sacrificial material with a preceramic polymer or ceramic suspension.⁴⁴ The way that the sacrificial material is extracted from the consolidated composite depends primarily on the type of pore former employed. A wide variety of sacrificial materials have been used as pore formers including natural and synthetic organics, salts, liquids, metals, and ceramic compounds.

2.4.1. Synthetic and natural organic pore formers

Organic pore formers are often extracted through pyrolysis by applying controlled thermal treatments at temperature between 200 to 600°C.⁴⁵⁻⁴⁶ The heat treatment schedule should be properly defined in order to avoid formation of defects. The long periods required for complete pyrolysis of the organic pore formers is one of the main disadvantage. The burnout rate of organic pore formers also plays an important role in order to avoid cracking of relatively thin samples during heat treatment.

A wide number of different oxides have been used to fabricate porous ceramics using starch particles, saw dust, wood dust, polymeric beads etc. as sacrificial template. The main advantage of this approach are its simplicity and the fact is that it can be applied to any material.^{12,47}

2.4.2. Liquid pore formers

Examples of liquid pore formers are water, oil or a solid phase that can be easily sublimated (e.g., naphthalene). In these cases, even though the extraction process is also time consuming, liquids and volatile oils can be evaporated or sublimated under milder conditions without generating toxic gases and excessive stresses during removal of the pore former.⁴⁸⁻⁵⁰

2.4.3. Salts, ceramics and metallic particles as pore former

Sacrificial pore formers such as salts, ceramics and metallic particles, are usually extracted by chemical rather than thermal means. The extraction of salts has been easily accomplished by repeatedly washing the composite with water.^{44,51} Ceramic and metallic particles require more aggressive agents and are in most cases removed by acidic leaching.⁵²⁻⁵³

In all of these processes, the continuous matrix phase has to be partially consolidated before removal of the sacrificial template so that the porous structure does not collapse during the extraction step. The consolidation of the continuous ceramic phase is very important. It differs from process to process.

The sacrificial template technique is quite flexible with respect to the possible chemical compositions that can be used in the fabrication process. One of the main advantages of the sacrificial template method in comparison to other fabrication routes is the possibility of deliberately tailor the porosity, pore size, distribution and pore morphology of the final ceramic component through the appropriate choice of the sacrificial material. The range of porosity and pore size that can be achieved with this technique is very broad (20-90% and 1-700 μm , respectively) as they only depend on the volume fraction and size of the sacrificial template used. Since in this method the ceramic component corresponds to the negative of the original template, the removal of the sacrificial phase does not lead to flaws in the struts as in the case of positive replica techniques described earlier.

2.5. Direct Foaming Method

In direct foaming methods, porous materials are produced by incorporating air in a suspension or liquid media. This is subsequently set in order to keep the

structure of air bubbles created (Fig. 2.6). In most cases, the consolidated foams are afterwards sintered at high temperatures to develop strength of porous ceramics. The total porosity of directly foamed ceramics is proportional to the amount of gas incorporated into the suspension or liquid medium during the foaming process.

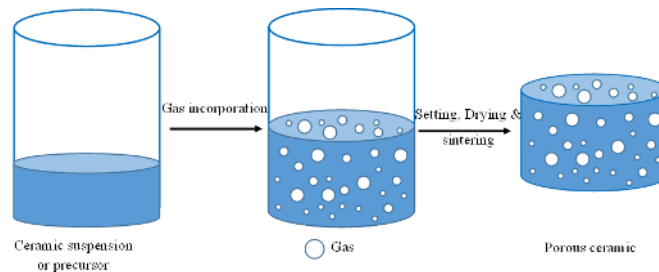


Fig. 2.6 Direct foaming processing routes used for the production of porous ceramics¹¹

The pore size is determined by the stability of the wet foam before setting takes place. Wet foams are thermodynamically unstable systems which undergo continuous Ostwald ripening and coalescence processes in order to decrease the overall free energy of the foam. These stabilization processes significantly increase the size of the incorporated bubbles resulting in large pores in the final microstructure. Therefore, stabilization of incorporated air bubbles in the suspension is the most critical controlling parameter. The foam stabilization can be achieved either by use of surfactants or by the use of particle.

2.5.1. Foam stabilization with surfactants

Surface active agents such as long-chain amphiphilic molecules and biomolecules like lipids and proteins are used to stabilize wet aqueous foams. These molecules adsorb on the air bubble surface and reduce the air-water interfacial energy, slowing down the coalescence of bubbles. However, due to low adsorption energy of such surfactants, they cannot prevent long term destabilization of foams and collapse within a few minutes after foaming. Therefore, a setting agent is required to consolidate the foam structure. The ultimate pore size of the porous ceramic depends on a balance between the kinetics of bubble disproportionation and the speed of liquid/suspension setting. Various surfactants like nonionic, anionic, cationic, zwitterionic surfactants are used for stabilization of foams in direct foaming methods.

Numerous processing routes have been developed in the last few decades to prepare porous ceramics using direct foaming methods based on surfactants. Various processes can be categorized in to (i) in situ polymer blowing/setting (ii) sol-gel setting (iii) gel cast setting etc.^{25,28,29} By controlling the foam stability and setting kinetics, pore size within the range 35 μm to 1.2 mm have been achieved using the above mentioned surfactant based direct foaming methods. The porosity can vary in the range 40-90%. The pores obtained with this method are typically spherical and can be either closed or open depending on the foam wet processing. By controlling the foam stability, air content, particle concentration, and setting kinetics, the microstructures can be altered. As opposed to the replica techniques, the direct foaming methods usually lead to dense flaw less struts after sintering.

2.5.2. Stabilization with particles

Solid particles with tailored surface chemistry have lately been shown to efficiently stabilize gas bubbles upon adsorption at the air-water interface.⁵⁴⁻⁵⁸ In this method colloidal particles are used to stabilize the foams and to produce smaller pore sizes than those obtained with surfactant based foaming techniques.⁵⁸⁻⁵⁹ In this method, the attachment of colloidal particles at the air-water interface is promoted by deliberately changing the wettability of the particle upon adsorption of short-chain amphiphilic molecules on the surface. A minimum amphiphile concentration is required to induce their attachment at the air-water interface. After the surface modification of particles, air can be easily incorporated by mechanical frothing, injection of gas stream directly into the suspension.

The particle stabilized foam is completely stable against drainage, coalescence and disproportionation for more than 4 days. The outstanding stability achieved with this direct foaming technique relies on the irreversible adsorption of colloidal particles at the air-water interface of the gas bubbles, as opposed to the continuous adsorption-desorption experienced by conventional surfactant molecules.⁵⁴⁻⁵⁸ Because of its remarkable stability, particle-stabilized foams do not require a setting step and thus can be directly dried and sintered to obtain the porous ceramics. The porosity of foams produced with this method vary typically between

40-93%, whereas the average pore size can be from approximately 10 to 300 μm . Porous ceramics with closed pores can be easily prepared with this method.

2.6. Other Methods

2.6.1. Sol-gel method

Sol-gel route for fabrication of porous ceramics was developed by Velvec et al., which produces uniform and controllable pore size in the micrometer range. In this method modified colloidal silica was used as the template for silica polymerization.^{28,60} Colloidal crystals were formed by filtering the suspension of polystyrene latex microspheres (200-1000 nm) carrying either negative (sulphate) or positive surface charge (amidine) through a smooth surfaced narrow-pore membrane. The deposited particulates on the surface of the membrane build up a closely packed ordered layer of about 10 μm . This ordered layer was separated from the membrane and soaked with 0.02 M hexa decyltrimethyl ammonium bromide (HTAB) surfactant solution for 20 minutes. The unadsorbed HTAB solution was removed by washing with water and then 0.5 M silica sol was allowed to penetrate into the layer formed by the microspheres. The insitu polymerization took place while the sol was padded through the layer formed by the microspheres. This resulted in a decrease in rate of penetration of silica sol through the latex layer and then finally stopped within a minute. Then the latex/silica composites were dried in vacuum followed by removal of polymer template at around 450°C for one hour. In this method, pore size could be controlled from 150 nm to 1 μm by varying the size of the microspheres.

2.6.2. Freeze drying

Freeze drying is a simple method for forming of porous ceramics. This neither requires use of pore forming additives nor any organic materials to produce porosity. In this process, aqueous ceramic suspensions with no other organic additives, except dispersant, are made. The aqueous suspensions are frozen to promote directional growth of ice. Ethanol is used as a refrigerant and the process temperature is controlled between -20°C to -80°C . During freezing, while the ice growth occurs vertically, the ceramic particles are concentrated in regions between

the columns of ice. The frozen suspension is dried at a reduced pressure allowing ice to sublime leaving pores in the ceramic body. Finally, the green alumina bodies were sintered in the temperature range of 1400-1600°C. This process resulted in a porosity of 50% and the pore morphology is altered by changing the solid loading in the aqueous suspension and the freezing temperature.^{48,61}

Each method has its own advantages and potential uses. The control of processing steps and consequently the ultimate material properties in terms of pore microstructure is a common requirement for all fabrication processes. In this regard, selection of fabrication process plays an important role in controlling the microstructure and corresponding properties of porous ceramics. Among various forming methods, as stated above, use of sacrificial template (pore formers) which burn out during heat treatment leaving porosity in the sample, has been a very common approach for fabrication of porous ceramics. While several pore forming agents have been used in ceramic processing, those of biological origin are especially popular due to the fact that their burnout is usually harmless from ecological and hygienic point of view. Their ash content producing inorganic salts is mostly low enough to be neglected with respect to the purity of the initial raw materials used in formulating the composition.

A wide variety of organic pore formers such as starch, saw dust, cotton thread, wheat flour etc., have been successfully used in fabrication of porous ceramics. Ferreira et al. have reported use of starch as both binder and pore former during forming of porous ceramics.¹² Ducman et al. have used saw dust and paper making sludge as pore forming agent for light weight clay bricks.⁶² Zhang et al. have studied fabrication of porous ceramics with unidirectionally aligned continuous pores using cotton thread.⁶³ Pravakaran et al. fabricated macroporous alumina ceramics using wheat particles as gelling and pore forming agent.⁶⁴

Several authors have reported use of rice husk, an agricultural waste, for making refractory bricks and porous ceramics, though the porosity and pore sizes are, however, limited within a narrow range. Wei et al. reported preparation of porous cordierite with porosity 42.4 to 45.02%, using rice husk where rice husk was used as silica source as well as pore forming agent.⁶⁵ Watari et al. have reported

fabrication of (10-30 μm pore size) SiO_2/C composite from rice husks.⁶⁶ Makovska et al. have used rice husk and waste aluminium oxide to fabricate porous mullite ceramics with porosity 52.23 to 58.31%.⁶⁷ Torikai et al. reported preparation of porous ceramics containing Al or Al_2O_3 using rice husk.⁶⁸ The pore diameters were 28 μm and 94 μm for (RH/Al) and (RH/ Al_2O_3) respectively. The reported literature has not emphasized on control and analysis of pore microstructure of the developed rice husk based porous ceramics.

In this study, therefore, attempts were made to fabricate porous ceramics with wide range of pore microstructure with tailored porosity, pore size, pore connectivity etc., using rice husk as fugitive pore former. The study has primarily focused on microstructure tailoring with special emphasis on volume fraction porosity, pores size and pores connectivity of the developed porous ceramics, by variation in RH content and particle size of RH powder in the composition. The objectives of the present research work are outlined in details in the next chapter.