CHAPTER II
Experimental and Characterization Techniques

II.1 Introduction:

Carbon and ceramic foams are of great interest, because of their properties like light weight, low density, high chemical resistance, good thermal shock resistance and potential tailor-ability of their physical properties from insulator to conducting material. Numbers of methods used for development of carbon and silica foam are known. In the present work, template method has been used for development of carbon and silica foams. The chapter includes:

1. Development of carbon foam
2. Functionalization of carbon foam
3. Development of silica foam
4. Characterization techniques

The carbon and silica foams have been characterized for their composition, physical, thermal, microstructure and mechanical properties. The characterization techniques used for analyses have been described in this chapter.

Prepared carbon and silica foam were also analyzed using statistical data analysis software (SPSS and MINITAB) to estimate dimensional shrinkage observed after heat treatment at 1000 °C. Linear equations were obtained for estimation of linear shrinkage observed in length, breadth and height.

II.2 Development of carbon foam:

Carbon foams were developed by using template route. Commercially available open cell polyurethane (PU) foam was used as template and phenolic resin was used as carbon precursor. PU foams were impregnated with phenolic resin solution and cured. Carbonization of cured foam was carried out up to 1000 °C in an inert atmosphere to make carbon foam.
The carbon foams were characterized for their volume shrinkage, change in density, crystallinity, porosity, permeability, specific heat, co-efficient of thermal expansion, surface morphology, mechanical properties, etc.

II.2.1 Materials used:

- Commercially available open cell polyurethane foams
- Phenolic resin, Kemrock Industries and Export Limited.
- Methanol, [99.5%]

II.2.2 Characteristics of Polyurethane foam:

Open cell polyurethane (PU) foams, commercially available were collected having different densities shown in figure II.1. There were of different colours.

![PU foam having different density](image)

**Figure II.1**: PU foam having different density

Open cell PU foams used were characterized for their density. The densities of different foams were calculated and table II.1 gives density of different types of foam. The density varies from 0.02 to 0.04 g/cc.
Table II.1: Density of different PU foams

<table>
<thead>
<tr>
<th>No.</th>
<th>Colour</th>
<th>PU foam density, gm/cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>i</td>
<td>White</td>
<td>0.020</td>
</tr>
<tr>
<td>ii</td>
<td>Yellow</td>
<td>0.024</td>
</tr>
<tr>
<td>iii</td>
<td>Dark grey</td>
<td>0.026</td>
</tr>
<tr>
<td>iv</td>
<td>Orange</td>
<td>0.036</td>
</tr>
<tr>
<td>v</td>
<td>Pink</td>
<td>0.040</td>
</tr>
</tbody>
</table>

II.2.3 Properties of phenolic resin:

Phenolic resin was used as carbon precursor for making carbon foam. The specifications of phenolic resin used are given in table II.2:

Table II.2: Specification of phenolic resin

<table>
<thead>
<tr>
<th></th>
<th>: Clear brownish viscous liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td></td>
</tr>
<tr>
<td>Viscosity @ 25 °C</td>
<td>: 2008 cp</td>
</tr>
<tr>
<td>Specific gravity @ 25 °C</td>
<td>: 1.23</td>
</tr>
<tr>
<td>Moisture content</td>
<td>: 11 %</td>
</tr>
<tr>
<td>pH</td>
<td>: 6.4</td>
</tr>
<tr>
<td>Refractive index</td>
<td>: 1.570</td>
</tr>
<tr>
<td>Non-volatile content</td>
<td>: 76 %</td>
</tr>
<tr>
<td>Resin/catalyst ratio</td>
<td>: 100/7 part</td>
</tr>
</tbody>
</table>

II.2.4 Preparation of foams:

The open cell PU foams having density of 0.04 g/cc were selected for development of foam. The foams were washed in distilled water and dried in oven at 100 °C. The colour of PU foam was pink. Cleaned PU foams were cut into small rectangular and square pieces. Dimensions of PU foam pieces were taken using vernier callipers and mass by using electronic balance LIBROR AGE-220 (Shimadzu co. Japan).
II.2.5 Preparation of resin solution:

The resin solution was made by using methanol as a solvent. Mixture of methanol and phenolic resin were prepared in different ratio and change in viscosity of these mixtures of phenolic resin and methanol was measured using Brookfield viscometer. Calibration curve was prepared for resin to methanol ratio from 10 : 90 to 100 : 0. Change in density of mixture of phenolic resin and methanol were measured using weight to volume ratio.

II.2.6 Impregnation of phenolic resin

Cleaned PU foam was impregnated with phenolic resin. For that PU foam was dipped into beaker filled with phenolic resin. The excess resin was removed from time to time to achieve uniform impregnation. Figure II.2 shows physical observation of resin impregnated cured foam from inside. Incomplete impregnation results into the voids or vacant space in final carbon foam. The presence of extra resin in the sample may close some pores or having dead end at the bottom.

![Image](image.jpg)

**Figure II.2:** Cross-section image of incomplete (left) and complete impregnated (right) foam

By carrying out multiple impregnations, it was found that 13 ml resin solution was sufficient for impregnation of 50 cc PU foam.
II.2.7 Drying and curing of foams:

Drying of resin impregnated foam was carried out in oven at 150 °C. To enhance the degree of cross linking between phenolic chains, impregnated PU foams were cured at 150 °C overnight. The change in color of foam appeared to be dark brown as shown in figure II.2 and II.4.

II.2.8 Carbonization:

The cured foams were carbonized at 1000 °C in nitrogen atmosphere. The carbonization assembly used for making carbon foam is shown in figure II.3. Carbonization was carried out in carbonization reactor having gas outlet and inlet on same side. The reactor was made up of stainless steel material. The S.S. container was placed in a muffle furnace which was heated electrically. Heating and cooling rate of the furnace was controlled by temperature programmer. The temperature of furnace was measured using chromel-alumel thermocouple and CHINO-KP1000 temperature programmer/controller was used to maintain the temperature of furnace. High purity Nitrogen gas was used during carbonization as an inert gas.

Figure II.3: Set up for carbonization
Schematic diagram for development of carbon foam is shown in figure II.4.

**Figure II.4**: Schematic diagram for development of carbon foam

II.2.9 Heat treatments:

Carbon foams heat treated at 1000 °C were heated at different temperature as given below:

1) In nitrogen atmosphere at 1000 °C
2) In argon atmosphere at 1200, 1400 and 1600 °C.

High temperature heat treatment was carried out in graphite boat in alumina tubular furnace. Figure II.5 shows furnace used for high temperature heat treatment. Heating and cooling rate was 300 °C/ hr. Samples were soaked for 1 hour at 1200, 1400 and 1600 °C temperature.
II.2.10 Functionalization of carbon foam:

Carbon foams developed by using template route were chemically functionalized by treatment with concentrated nitric acid at 100 °C. Figure II.6 shows schematic diagram for functionalization of carbon foam. Figure II.7 shows set up used for functionalization of carbon foam.

Weighed carbon foam samples were placed in 4 N nitric acid solution in volumetric flask and refluxed for different time intervals, of 3, 6, 7, 8 and 9 hours. The acid treated samples were washed thoroughly with distilled water to free them of nitrate ions till neutral pH was achieved. These were dried in oven at 150 °C. The type and amount of surface oxygen functionalities formed on the surface were determined using Boehm’s titration and FTIR analysis.
II.3 Development of silica foam:

Silica foam was developed by using template method. Polyurethane foams of lower density were used as template to get highly interconnected porous network. Silica sol was prepared using sol gel route and it was impregnated into the PU template. Sintering of sol impregnated dried samples were carried out at 1000 °C in air, to make silica foam.
foams were characterized for different properties e.g. crystallinity, acid/base resistance, thermal, compressive strength.

II.3.1 Materials used:

- Open cell PU foam, (density: 0.02 g/cc)
- Tetraethyl orthosilicate (TEOS, Si(OC₂H₅)₄), Sigma-Aldrich
- Ethanol (C₂H₅OH) (99.5 %)
- Hydrochloric acid [HCl], (36 %)
- Distilled water [H₂O]
- Nitric acid, [HNO₃], (70 %)
- Sulphuric acid, [H₂SO₄], (98 %)
- Sodium hydroxide, [NaOH], (98 %)
- Potassium hydroxide, [KOH], (85 %)
- Sodium bicarbonate, [Na₂CO₃], (100 %)

II.3.2 Preparation of silica sol:

Silica sol was prepared by hydrolysis of Tetraethoxysilane, Si(OC₂H₅)₄, with acidified water. Ethanol was used as a solvent for TEOS. 2 moles of ethanol and 1 mole of TEOS were mixed in a flask using magnetic stirrer (shown in figure II.8). The hydrolysis of TEOS was carried with acidified water using TEOS : H₂O from 1 : 2 to 1 : 6. Acidified water was added drop wise into solution of TEOS and ethanol. Hydrolysis reaction was carried out for 70 hrs.
Kinetics of hydrolysis of silica sol was studied using UV-VIS spectrophotometer by measuring $\lambda_{\text{max}}$ of TEOS, ethanol and water mixture at different time interval. Figure II.9 shows the UV-VIS spectrophotometer. (Shimadzu UV1601, Double Beam Optics with Halogen Light Source, Spectral Resolution Power: 2 nm)
II.3.3 Impregnation of silica sol:

PU foams were cleaned, cut into small rectangular pieces and dried at 100 °C in oven. PU foam having 0.02 g/cc density (white in colour) was used as template. Silica sol was impregnated in PU foam by dipping and drying. By number of experiments it was concluded that 40 cc volume PU foam was required for 25 ml silica sol.

II.3.4 Drying of foams:

Drying was carried out in conventional oven as well as in microwave oven. The conventional vacuum oven used is shown in figure II.10. The drying of foam was carried out at 100 °C. The uneven shrinkage and small deformation were seen as shown in figure II.11.
To overcome these uneven shrinkage and deformation, microwave drying was carried out. The microwave oven used is shown in figure II.12. Silica sol impregnated PU foam was dried using microwave oven at different power levels as shown in table II.3 and up to 800 watt power level was used for drying foams. During drying, the sample was overturned as to have uniform drying of sol in the sample.

![Microwave oven](image1.png)

**Figure II.12: Microwave oven (Whirlpool, Model: JT 368)**

**Table II.3:** Steps for microwave drying of sol impregnated PU sample

<table>
<thead>
<tr>
<th>Power level</th>
<th>I side</th>
<th>II side</th>
<th>III side</th>
<th>IV side</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>500 watt</strong></td>
<td>15 s</td>
<td>15 s</td>
<td>15 s</td>
<td>15 s</td>
</tr>
<tr>
<td><strong>800 watt</strong></td>
<td>15 s</td>
<td>15 s</td>
<td>15 s</td>
<td>15 s</td>
</tr>
</tbody>
</table>

![Drying of silica sol impregnated PU foam](image2.png)

**Figure II.13:** Drying of silica sol impregnated PU foam using Microwave oven
Chapter II

The pink and white colour PU foams were used as template shown in figure II.13. Microwave drying of silica sol impregnated PU foams show uniform drying without warping at edges and in the centre.

II.3.5 Heat treatment of foams:

The silica impregnated dried foams were sintered at 1000°C in furnace, shown in figure II.4. Silica foams were further heat treated at high temperature in furnace shown in figure II.5. Various heating rates were used during heat treatment of silica impregnated dried foam to obtain good strength. Hold time was varied from 1 to 5 hours at 570, 650, 870 and 1000 °C. Maximum strength of silica foam was obtained using heating rate profile given in figure II.14. Silica foams were further heat treated up to 1600°C at 300°C/hr heating and cooling rate with 1 hour hold time.

![Figure II.14: Heating rate profile used for sintering](image)

Schematic diagram for development of silica foam is shown in figure II.15. The prepared silica foams were characterized for different properties.
II.4 Characterization of foams:

II.4.1 Physical properties:

II.4.1.1 Volume shrinkage:

The reduction in length, breadth and thickness during carbonization and sintering was measured by measuring dimensions both before and after heat treatment using vernier callipers. The shrinkage in all direction was calculated by using formula:

\[
\text{% Shrinkage in length} = \left( \frac{L_{\text{before}} - L_{\text{after}}}{L_{\text{before}}} \right) \times 100
\]

where,

L\text{-before} – Length before heat treatment, mm

L\text{-after} – Length after heat treatment, mm

Likewise, shrinkage in breadth and thickness was determined by above formula.
II.4.1.2 Carbon yield, (%):

Carbon yield was calculated from the initial weight of materials (W1) before carbonization and final weight of carbon (W2) obtained after carbonization using following relation.

\[
\% \text{ carbon yield} = \frac{W2}{W1} \times 100
\]

II.4.1.3. Density Measurement:

Bulk density of the samples was theoretically calculated. Geometric volume of sample was calculated by measuring length, breadth and thickness of foam with the help of vernier calipers.

\[
\text{Volume of sample (V)} = l \times b \times t
\]

Where,

\[ l = \text{length of the sample} \]
\[ b = \text{breadth of the sample} \]
\[ t = \text{thickness of the sample} \]

Density of sample was determined using following formula:

\[
\text{Density (D)} = \frac{\text{Mass of sample (W)}}{\text{Volume of sample (V)}}
\]

II.4.1.4 Porosity:

II.4.1.4.1 Kerosene porosity:

The kerosene pick-up method was used for determination of open porosity of developed carbon and silica foams. Figure II.16 shows set-up used for determination of kerosene porosity of foams. It consists of a glass flask connected to a kerosene reservoir and vacuum system with stopcocks. One stopcock was joined in between flask and kerosene reservoir and the second stop cork was joined in between flask and vacuum system.

A small piece of foam of known measured dimensions was taken and weighed. It was placed in the round bottom flask. Flask was connected with vacuum system by opening
stopcock 2. The sample was evacuated at $10^{-3}$ torr for one hour with the help of vacuum pump to clean the sample. The stopcock -2 was closed after evacuation for one hour and kerosene was allowed to flow by opening stopcock- 1. Kerosene was allowed to flow into open pores of sample. After equilibrium was attained, sample was taken out and excess of kerosene was wiped out. Sample was weighed using electronic balance. The density of kerosene was determined using specific gravity bottle. The kerosene porosity of sample was calculated using following reaction.

$$\% \text{Porosity} = \frac{[W_b - W_a]}{V_S \times D_k} \times 100 \quad \text{......( i )}$$

Where

$W_a =$ weight of sample before adding kerosene/water

$W_b =$ weight of sample after adding kerosene/water

$V_s =$ volume of sample

$D_k =$ Density of Kerosene/water

\[\text{Figure II.16: Set up for measurement of kerosene porosity}\]
II.4.1.4.2 Water porosity:

![Figure II.17: Set up used for measurement of water porosity](image)

Figure II.17 shows the set-up for determination of water porosity of foams. A small piece of foam of known accurate dimensions was weighed and placed in the beaker having boiling water. It was allowed to remain in boiling water for 5 hours so that all pores were filled with water. Water porosity was determined by using equation (i).

II.4.1.5 Permeability measurement:

Permeability describes how easily a fluid is able to flow through the porous material. Thus, it is related to the connectedness of void spaces and to pore size of the material. It is calculated using Darcy’s Law. In the present study, permeability was measured using set up designed and fabricated indigenously as shown in figure II.19. The set was designed on the basis of Darcy’s principal i.e. pressure drop occurs when porous media is placed in the constant gas flow.

The leak proof sample box made up of stainless steel having 5 x 5 x 5 cm of sample loading facility was fabricated is shown in figure II.18. From both sides it was attached with control valves and pressure gauges. Pneumatic joints and polymeric pipes were used to avoid leakage of gas. Inlet was attached with gas flow meter, non-return valve and sample gas
cylinder while outlet was directly connected to gas chromatograph (GC) as shown in figure II.20.

![Sample box designed for permeability measurement](image)

**Figure II.18**: Sample box designed for permeability measurement

The sample was mounted in the holder and attached to the GC. In GC, sample gas was transported through the column by flow of inert, gaseous mobile phase. The gaseous compounds being analyzed interact with walls of column, which is coated with different stationary phases. The GC analysis was carried out using TCD (thermal conductivity detector). A TCD detector is used to monitor outlet stream from the column; thus, time at which each component reaches outlet and amount of that component can be determined. This causes each compound to elute at a different time, known as the *retention time* of the compound. Area under the peak gives amount of gas passing through the column of GC.

Flow of gases through the sample, both carrier and test gas was controlled by using flowmeter and could be quantified with the help of GC. The gases flowing from the sample were passed into GC for quantitative analysis.
The following conditions were monitored during the permeability test.

- Injector temperature : 100 °C
- Column/oven temperature : 60 °C
- Detector temperature : 60 °C
- Column : 13X Molecular sieves

Retention times for different gases were measured on the above mentioned conditions and retention time for different gases in presence of argon/nitrogen carrier gases are given in table II.4.

Sample size: Carbon foam: (47.45 X 38.17 X 23.46 mm)

Table II.4: Retention time for different gases

<table>
<thead>
<tr>
<th>Sample Gas</th>
<th>Retention time</th>
<th>Sample Gas</th>
<th>Retention time</th>
</tr>
</thead>
<tbody>
<tr>
<td>N₂</td>
<td>1.46/1.47</td>
<td>Ar</td>
<td>0.83/0.82</td>
</tr>
<tr>
<td>O₂</td>
<td>1.30/1.31</td>
<td>O₂</td>
<td>1.21/1.20</td>
</tr>
<tr>
<td>H₂</td>
<td>1.14/1.15</td>
<td>H₂</td>
<td>1.05/1.03</td>
</tr>
<tr>
<td>He</td>
<td>1.12/1.13</td>
<td>He</td>
<td>0.99/1.0</td>
</tr>
</tbody>
</table>
Permeability of different gases i.e. nitrogen, oxygen, hydrogen and helium through carbon foam was measured. Permeability was calculated by following formula:

\[
\% \text{ Permeability} = \frac{A_{\text{Sample}}}{A_{\text{Blank}}} \times 100
\]

Where,

\(A_{\text{Sample}}\) – Area under the peak with sample

\(A_{\text{Blank}}\) – Area under the peak without sample

II.4.2 Surface characteristics

II.4.2.1 Surface characteristics:

The surface area of carbon and silica foam was determined by BET surface area analyzer (Micromeritics Gemini 2375) using nitrogen as adsorbent at 77 K shown in figure II.20. Prior to adsorption, samples were degassed initially at 100°C for one hour in presence of argon gas and at 250°C for 14 - 15 hours. After degassing sample was weighed and attached to the surface area analyzer instrument. The nitrogen adsorption was studied at liquid nitrogen temperature.

Figure II.20: BET Surface Area Analyzer (Micromeritics Gemini 2375).
II.4.2.2 Surface oxygen complexes:

The Boehm titration was used as a chemical method to quantitatively measure surface oxygen groups present on carbon materials. It underlying theory says that bases of different strength react with acidic surface functionalities present on the carbon samples. [1-3]

Accurately weighed 0.5 gm of the functionalized carbon foam samples was added to 250 ml Erlenmeyer flasks containing 50 ml of 0.1N NaHCO₃, Na₂CO₃, NaOH and HCl solution respectively. The flask was stoppered and stirred for 24 hours. After 24 hours of stirring, solutions were filtered through a Whatman filter paper and excess of acid/base were titrated with 0.1N base/acid solution.

The volume required for neutralization for a particular surface group was calculated as follow:

- NaHCO₃ neutralizes carboxylic functionalities
- Na₂CO₃ neutralizes carboxylic and lactone groups
- NaOH neutralizes carboxylic, lactone and phenolic functionalities
- HCl neutralizes basic groups present on surface

Total concentration of surface functionalities ($Q_e$) available on carbon foam was calculated by:

$$Q_e \text{ (milimole/gm)} = \frac{C_i - C_e}{M_s} V$$

Where,

- $C_i$ = initial concentration (milimole)
- $C_e$ = final concentration (milimole) of corresponding acid/base,
- $V$ = volume of solution (ml),
- $M_s$ = weight of adsorbent (gm).
II.4.2.3 Surface groups (FTIR):

Functional groups present on foams at different heat treatment temperatures were studied using Shimadzu FTIR-8300 Spectrophotometer shown in figure II.21. KBr was used as reference material. Ratio between KBr to sample was kept 100:1. Before analysis KBr was kept in oven at 110°C to remove the moisture. The fine ground sample was mixed with KBr in agate mortar for uniform mixing. The sample was placed on sample holder of FTIR instrument and spectra were obtained in range of 4000 to 400 cm⁻¹. The absorption peaks observed on spectra were compared with standard literature data and analyzed for functional groups. Area under the peak was calculated for different peaks using software provided with the FTIR instrument.

![Figure II.21: Infrared spectrophotometer (Shimadzu FTIR-8300)](image)

II.4.3 Thermal properties

II.4.3.1 Thermal Gravimetric Analysis:

Thermal gravimetric analysis (TGA) was carried out using TGA, METTLER TG/SDTA 851 as shown in figure II.22. Small piece of sample was cut and placed in vertical furnace of TGA. Heating rate used in TGA was 15 °C / min.
II.4.3.2 Specific Heat:

Specific heat was measured by using DSC, METTLER DSC20 along with Console TC11 shown in figure II.23. Small piece of carbon foam was placed in crucible of differential scanning calorimetric (DSC). The sample was heated from 80ºC to 600 ºC. Heating rate of carbon foam during analysis was maintained at 15 ºC/min. The value of specific heat was obtained from software available with instrument.

II.4.3.3 Co-efficient of thermal expansion:

Coefficient of thermal expansion (CTE) was measured by thermo mechanical analyser (TMA), METTLER TA40 along with Console TC11 as shown in figure II.24. Carbon foam sample was sandwiched between S.S. plate and heat treated at 800 ºC. Heating rate during testing was maintained at 15 ºC/min with nitrogen environment. CTE of carbon foam was measured both cross sectional and transverse direction. Blank run was also performed using standard S.S. plates.

Figure II.22: Thermo Gravimetric Analyzer (TGA), METTLER TG/SDTA851
Figure II.23: Differential Scanning Calorimetric (DSC), METTLER DSC20

Figure II.24: Thermo Mechanical Analyzer (TMA), METTLER TA40
II.4.4 Mechanical properties:

II.4.4.1 Compressive Strength:

Compressive strength is a measure of compressive load per unit area. Compressive strength of carbon and silica foam was measured using Universal Testing Machine (UTM) Shimadzu AG-100kNG shown in figure II.25. Load cell of 100 KN and crosshead speed of 1.5 mm/min were used. The sample having 25 x 25 x 25 dimensions were used for measurement.

Figure II.25: Universal Testing Machine
II.4.5 Surface morphology

II.4.5.1 Scanning electron microscopy:

Surface morphology of PU foam, carbonized carbon foam and silica foam samples were observed using Hitachi S-3000N as shown in figure II.26 Scanning Electron Microscope. These samples were mounted on sample holder with the help of graphite tape. The tape acts as both adhesive and conducting material. Specimens were scanned at different magnifications. Non-conducting samples were coated with Pt-Pd in order to make conductive samples.

Figure II.26: S-3000N Hitachi scanning electron microscope
II.4.5.2 Optical microscopy

Optical microscope was used to study the microstructure of PU foam for average pore size, strut length and strut thickness having different density. The PU foams having different densities were cleaned with distilled water, and cut into thin slice. The cleaned samples were dried in oven at 100 °C. These dried samples were viewed under Leitz Optical Microscope at different magnifications using polarized light as shown in figure II.27. The average pore size, strut length and strut thickness was measured using LEICA image analysis software provided with optical microscope.

Figure II.27: Leitz Optical Microscope
II.4.6 X-Ray Diffraction Analysis:

XRD analysis of carbon and silica foam were carried out by powder diffraction method using X-Ray Diffractometer Philips, X’pert model shown in figure II.28 having Cu Kα1 (λ=1.54056 Å) source. Fine powder of foam was pressed in stainless steel sample holder. Wide angle X-ray diffraction patterns were recorded for analysis.

Figure II.28: X – Ray diffrectometer
II.4.7 Statistical analysis

Statistical analysis of carbon and silica foam was carried out using SPSS and MINITAB data analysis software. Samples having different sizes were fabricated and multiple regression tests were performed to generate linear equation to get idea of the optimum shrinkage occurred in the sample after heat treatment.

Multiple linear regression (MLR) method is used to model linear relationship between dependent variable and one or more independent variables. The dependent variable is sometimes called predictand, and the independent variables as predictors. MLR is based on least squares model. In the process of fitting or estimating model, statistics are computed that summarize accuracy of regression model based upon selected condition. [4-10]

II.4.7.1 Model description:

In present work shrinkage was observed in length, breadth and thickness on heat treatment of foam sample at 1000 °C in cured foam. Therefore, to carry out statistical analysis and for modelling, data base was generated. For this, sample of foam of known dimensions were taken and heat treated at 1000 °C under identical condition. The dimensional shrinkage was measured for each and every sample. Using this data base Multiple regression tests were performed on SPSS software to generate linear equation which decides the optimum shrinkage for sample having any size.

Model equation: The model expresses values of predicted variable as a linear function of three predictor variables (i.e. shrinkage observed in length, breadth and thickness after heat treatments shown in figure II.29) and an error term

\[ Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + e \]

Where,

\[ X_1, X_2 \text{ and } X_3 = \text{value of predictor (length, breadth and thickness respectively)} \]

\[ b_0, b_1, b_2 \text{ and } b_3 = \text{regression constant (i.e. intercept)} \]
y = predictand (response variable)

e = error term

\[ Y = \hat{b}_0 + \hat{b}_1 \sum_{i=1}^{n} X_i + \hat{b}_2 \sum_{i=1}^{n} X_i + \hat{b}_3 \sum_{i=1}^{n} X_i \]

Three different equations for shrinkage observed in length, breadth and thickness were obtained using multiple regression analysis.

II.4.8 References:


