# CHAPTER – 4

(Synthesis and Characterisation of Porous Spinel by Porous MgO)

### 4.1 Synthesis of Spongy spinel by Spongy MgO

This chapter deals the Preparation of Porous Spinel by Porous MgO by Solid state sintering template method .The functional views of preparation are given below. MgO ,Starch, PVA are mixed in ratio after that different solution treatment result are examined.

| MgO | Starch ( Potato) | PVA |
|---|---|---|
| Mixing (60:30:10) | | |
| Milled (3h) | | |
| Pressing ( 8 tonn) | | |
| Drying (120°C) | | |
| Firing at Temperature 1100ºC (SP 1h) | | |
| Dipped in first 6 mol %Aluminium nitrate elucidation (t=24 h) | | |
| Firing at temperature 1300ºC | | |
| Study the Spinel conversion Ratio by SEM and XRD | | |
| Find the porosity ,density ,thermal conductivity ,permeability, chemical resistance ,compressive strength Specific heat of sample after first elucidation treatments | | |
| Dipped in second aluminium nitrate elucidation t=48 hour | | |
| Firing 1300ºC | | |

| |
|---|
| Study the Spinel conversion Ratio by SEM and XRD |
| Find the porosity ,density ,thermal conductivity ,permeability, chemical resistance ,compressive strength Specific heat of sample after 2 elucidation treatments |
| Dipped in third nitrate elucidation t=72 hour |
| Firing 1300ºC |
| Study the Spinel conversion Ratio by SEM and XRD |
| Find the porosity ,density ,thermal conductivity ,permeability, chemical resistance ,compressive strength Specific heat of sample after 3 elucidation treatments |
| Dipped in fourth aluminium nitrate elucidation t=96 hour |
| Firing 1300ºC |
| Find the porosity ,density ,thermal conductivity ,permeability, chemical resistance ,compressive strength Specific heat of sample after 4 elucidation treatments |
| Dipped in fifth aluminium nitrate elucidation t=120 hour |
| Firing 1300ºC |
| Find the porosity ,density ,thermal conductivity ,permeability, chemical resistance ,compressive strength Specific heat of sample after 5 elucidation treatments |

## 4.2 Characteristics of Spongy spinel by Spongy MgO

Table 4.1 gives information at different elucidation treatment at different concentration ,P1 for 6 mol%,P2 for 8 mol%, P3 for 10mol %used. From the figure 4.1 It is clear that thermal conductivity is high for fifth elucidation treatment where low for zero elucidation treatment [Latanic *et al* (1991)] .

**Table 4.1 Measurement and Result of Spongy spinel firing temp 1300 ºC soaking period 1 hour with different mole concentration.**

| Measurement | **(P1)** | **(P2)** | **(P3)** |
|---|---|---|---|
| Bulk Density (gm/cc) | 2.4 | 2.32 | 2.3 |
| Apparent Porosity (%) | 48.3 | 51 | 51 |
| Compressive Strength (MPa) | 17 | 15.3 | 15 |
| Thermal Conductivity (w/mk) | 4.8 | 4.1 | 3.9 |
| Corrosion Resistance (%) | 3.08 | 3.08 | 3.08 |
| Pearmeability (cm/sec) | 2.81 | 2.8 | 2.7 |
| IL water (%) | 51 | 51 | 48 |
| NIL | ------ | | |

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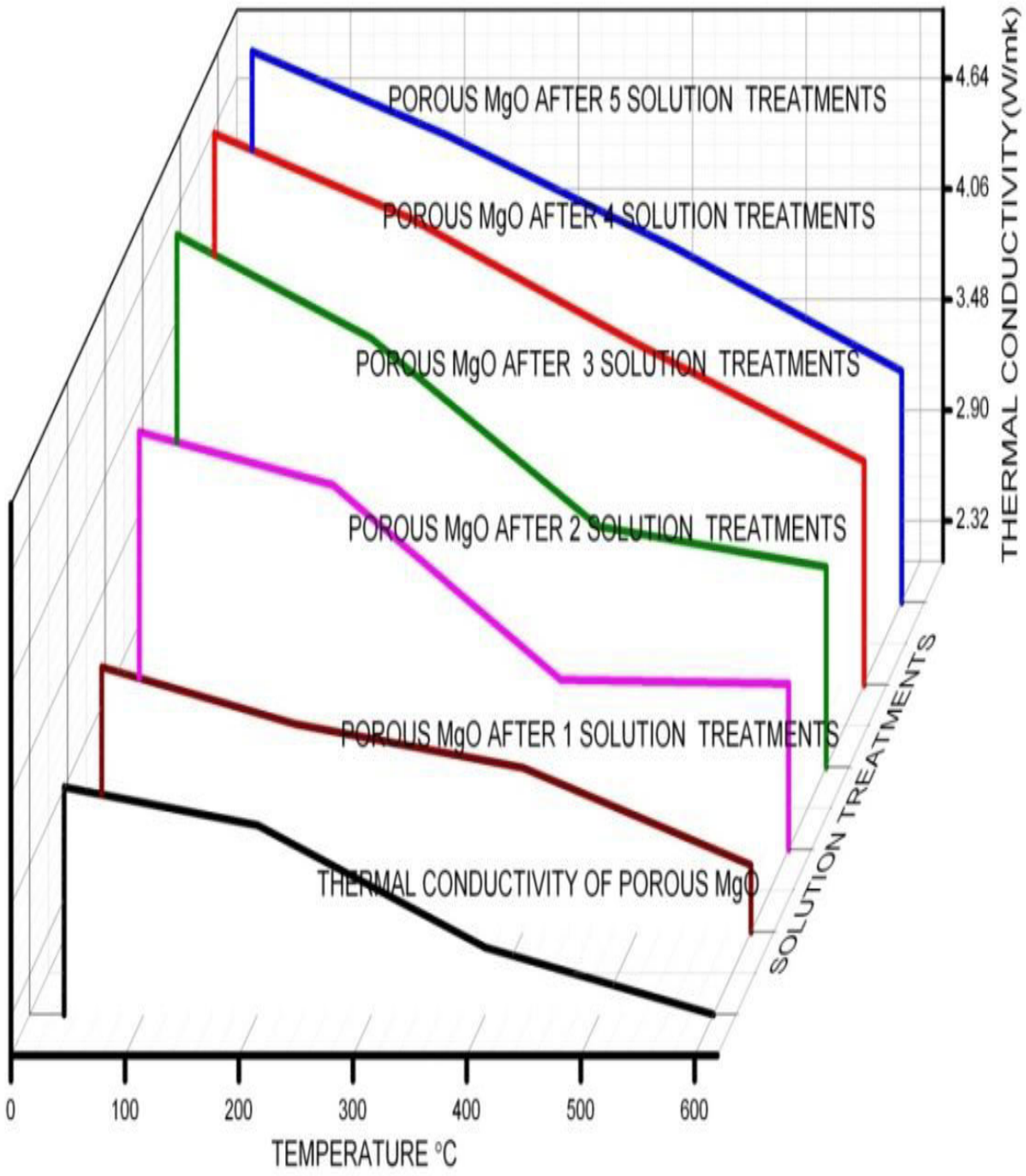


**Figure 4.1 Thermal conductivity of Spongy MgO with different number of elucidation treatment.**

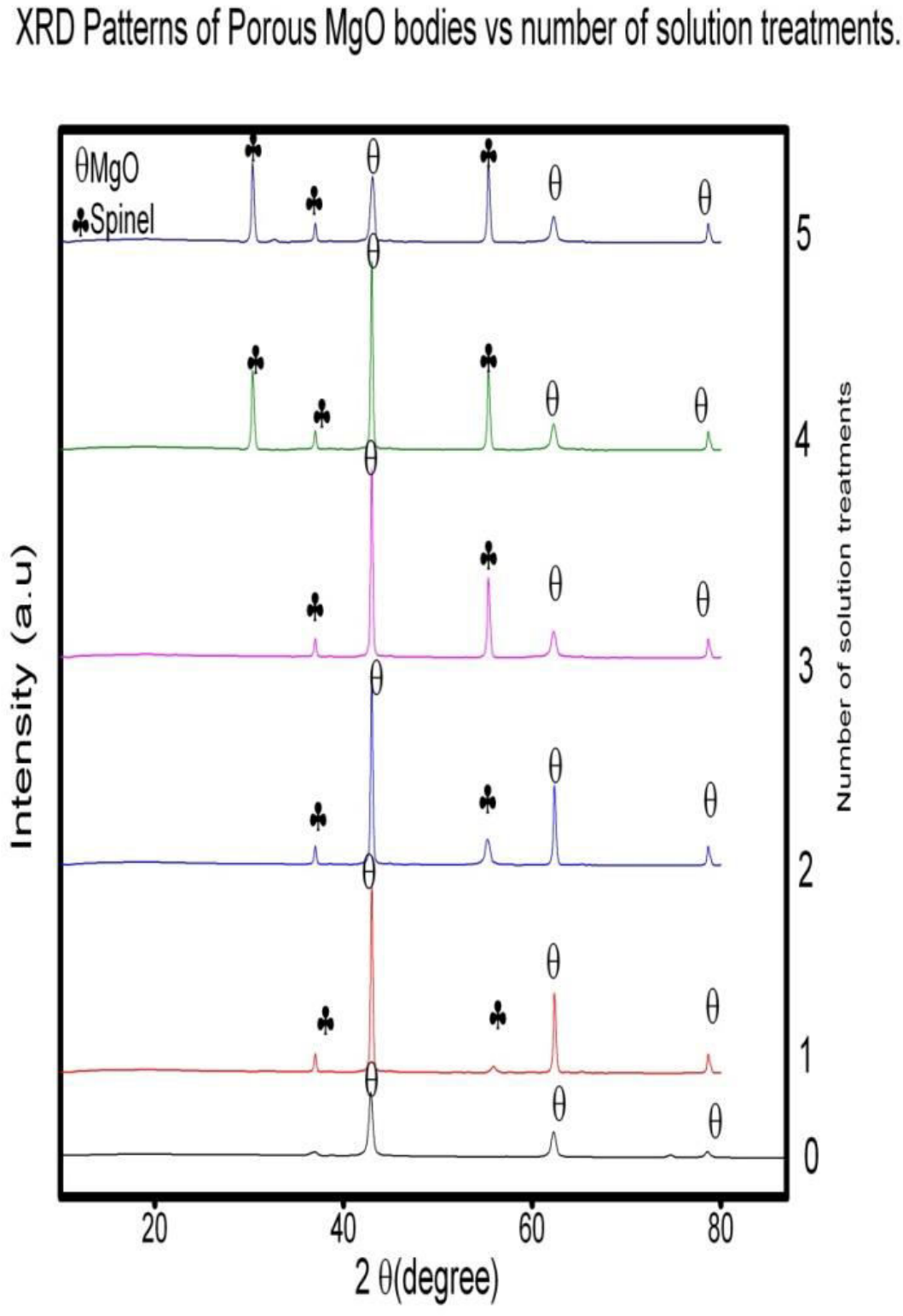


**Figure 4.2 XRD Pattern of Spongy MgO with different number of elucidation treatment**. [Cullity (2001)]

From the figure 4.1 It shows that with escalating elucidation treatments, the thermal conductivity of the illustration enlarged finally triumph 4.8W/m1K1. Subsequent to five exacerbate [Latanic *et al,* (1991)] . From the Figure 4.2 shows ,In these absorbent samples, the minute opening span be alive quite a lot of micrometers outstanding to the extent of the platelets From the XRD [Cullity (2001)]. It is understandable with the intention of veto Alumina are become aware of in any taster which shows that primarily Precipitated alumina absolutely Reacts in the midst of MgO to appearance spinel [ West , (2003) ] .There front ratio of adaptation to Spinel may perhaps be premeditated from the mock-up weight expand according to following element reaction .From the figure 4.3 Spongy MgO having a greater percentage of corrosion as compare to fifth elucidation treatments.

$$MgO+Al_2O_3=MgAl_2O_4$$

5th elucidation treatments colossal quantity of spinel are distinguish [Salvini, (2003)].

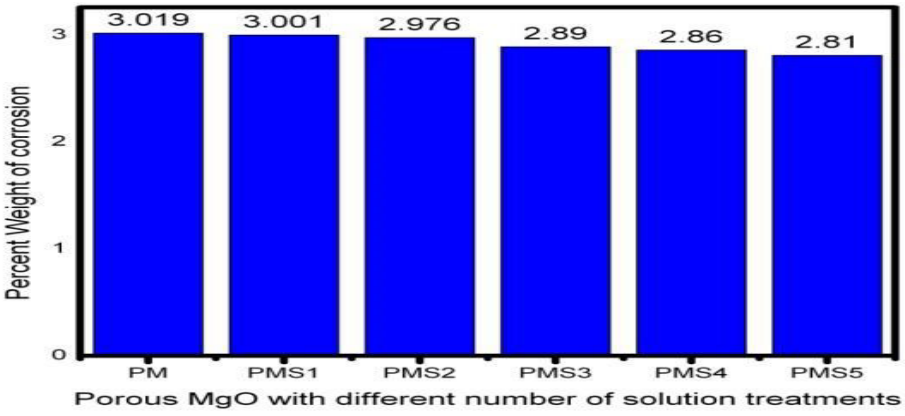


**Figure 4.3 Percentage corrosion of Spongy MgO with diverse number of elucidation treatment.**

**Table 4.2 Measurement and Result of Spongy spinel firing temp 1400 ºC soaking period 1 hour.**

| Measurement | **(P1)** | **(P2)** | **(P3)** |
|---|---|---|---|
| Bulk Density (gm/cc) | 2.4 2 | 2.45 | 2.49 |
| Apparent Porosity (%) | 48.1 | 45.3 | 45.3 |
| Compressive Strength (MPa) | 17 .1 | 17.3 | 18 |
| Thermal Conductivity (w/mk) | 4.8 | 4.9 | 4.91 |
| Corrosion Resistance (%) | 3.08 | 3.08 | 3.08 |
| Pearmeability (cm/sec) | 2.71 | 2.8 | 2.7 |
| IL water (%) | 50 | 51 | 51 |
| NIL | ------ | | |

**Table 4.3 Measurement and Result of Spongy Spinel Firing temp 1400 ºC SP 2 h**

| Measurement | **(P1)** | **(P2)** | **(P3)** |
|---|---|---|---|
| Bulk Density (gm/cc) | 2.5 | 2.51 | 2.55 |
| Apparent Porosity (%) | 48.3 | 47.9 | 46.9 |
| Compressive Strength (MPa) | 17 | 16.9 | 16.5 |
| Thermal Conductivity (w/mk) | 5.1 | 5.05 | 5.03 |
| Corrosion Resistance (%) | 3.06 | 3.07 | 3.07 |
| Pearmeability (cm/sec) | 2.67 | 2.71 | 2.3 |
| IL water (%) | 49 | 51 | 51 |
| NIL | ------ | | |

From the Table 4.1 to 4.4 it is clear that 8 mol% solution treatment gives a better response in terms of physical and mechanical Charesteristics as camparison to 6 mol% and 10 mol%.

**Table 4.4 Measurement and Result of Spongy spinel firing temp 1300 ºC soaking period 2 h**

| Measurement | **(P1)** | **(P2)** | **(P3)** |
|---|---|---|---|
| Bulk Density (gm/cc) | 2.5 | 2.51 | 2.55 |
| Apparent Porosity (%) | 48.3 | 47.9 | 46.9 |
| Compressive Strength (MPa) | 17 | 16.9 | 16.5 |
| Thermal Conductivity (w/mk) | 5.1 | 5.05 | 5.03 |
| Corrosion Resistance (%) | 3.06 | 3.07 | 3.07 |
| Pearmeability (cm/sec) | 2.67 | 2.71 | 2.3 |
| IL water (%) | 49 | 51 | 51 |
| NIL | ------ | | |

Figure 4.4 shows Archemedies Principle for bulk density Measurement .The following formula are used for measurement of bulk density and apparent porosity.

**BD = Dry Weight / (Soaked Weight –Suspended Weight) [Nandi ,(1987)].**

**AP=Soaked weight-Dry Weight / (Soaked Weight –Suspended Weight) *100 %** [Nandi ,(1987)].

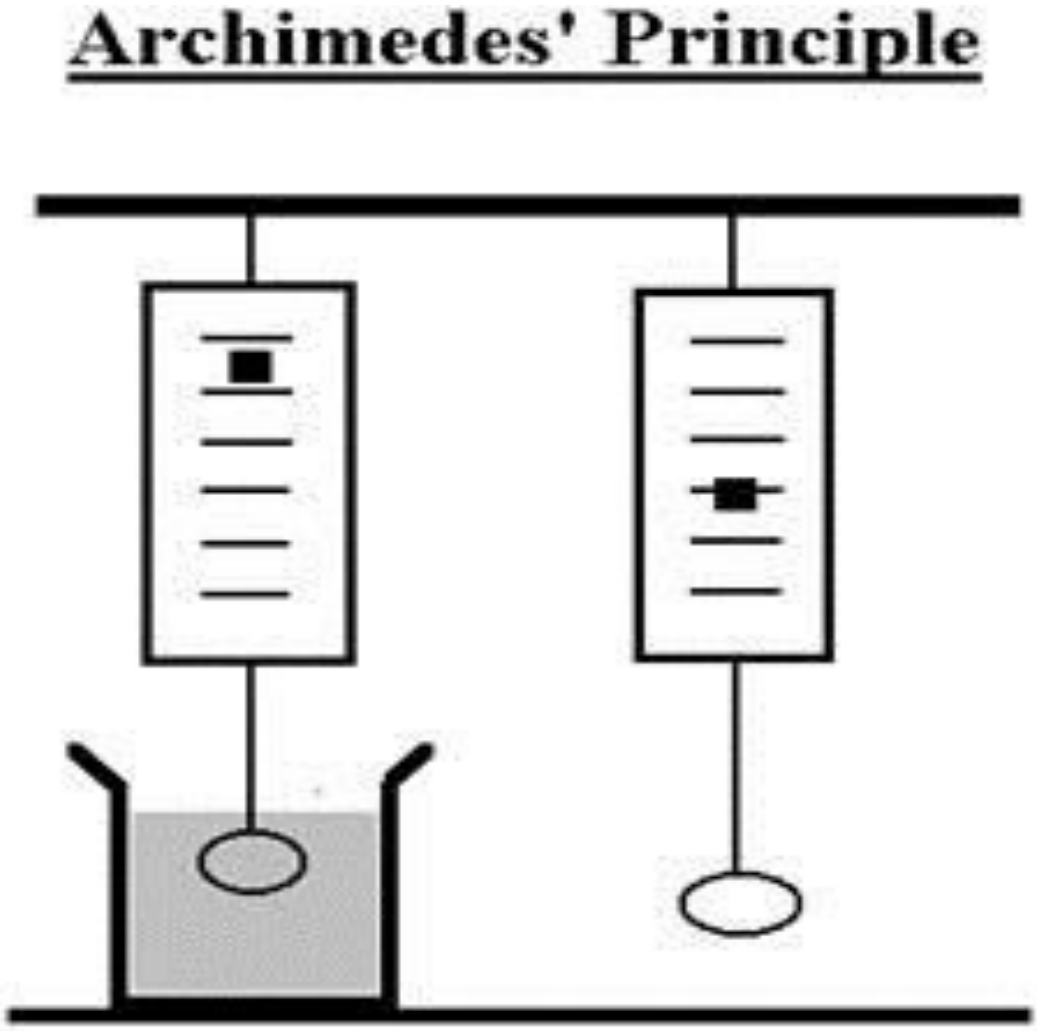


**Figure 4.4 Archimedes principle used for bulk density measurements .**

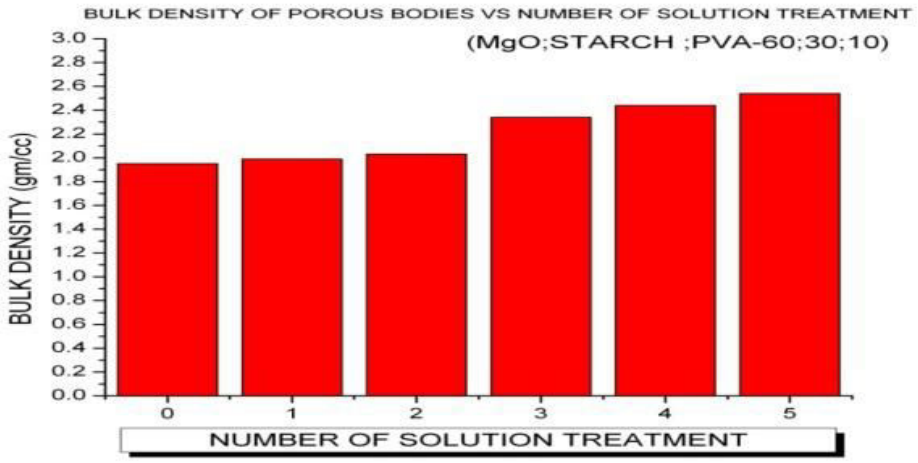


**Figure 4.5 Bulk density of Spongy MgO with different number of elucidation treatment.**

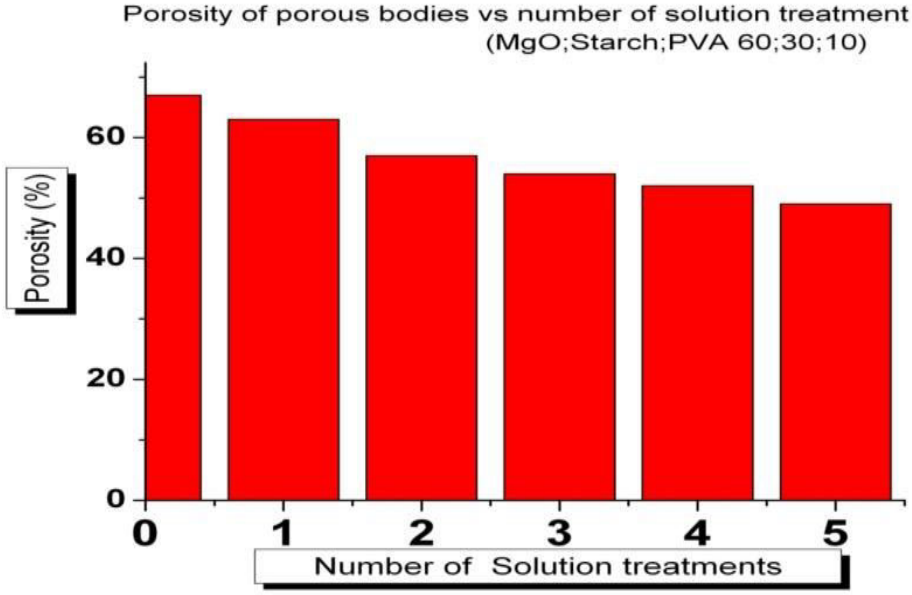


**Figure 4.6 Bulk density of Spongy MgO with different number of elucidation treatment.**

From the figure 4.5 Shows that density is maximum for fifth elucidation treatments and figure 4.6 Shows Porosity is minimum for fifth elucidation treatments.

## SEM analysis of Porous MgO during 1,2,3,4 solution treatments

From the figure 4.7 and 4.8 it was observed that a pallid facade are misshapen in sphere appearance in succeeding elucidation dealing ,then rehabilitated in opaque form in third explanation trea- tme nt. White surface shows spinel formation after elucidation treatment . Where as subsequent elucidation treatment this surface are change in dense ball form.

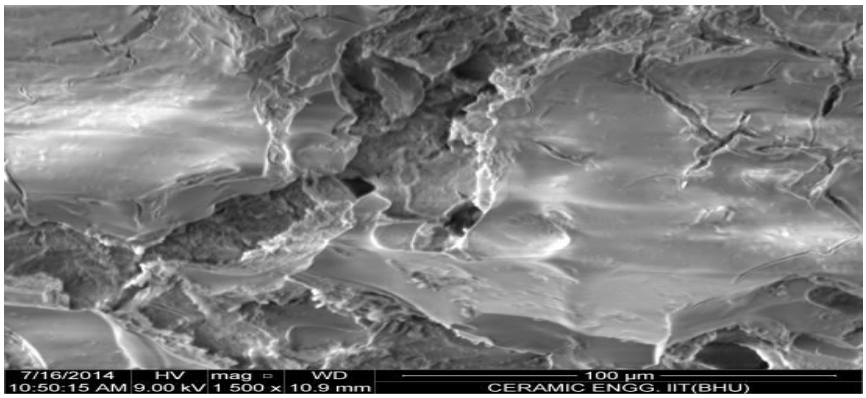


**Figure 4.7 SEM of of Spongy MgO with first number of elucidation treatment**

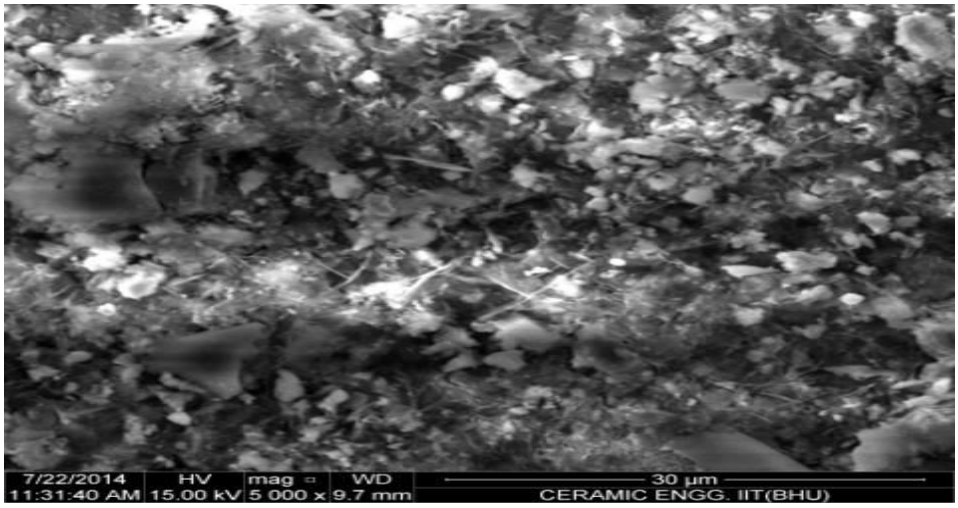


**Figure 4.8 SEM of of Spongy MgO with 5 number of elucidation treatment**

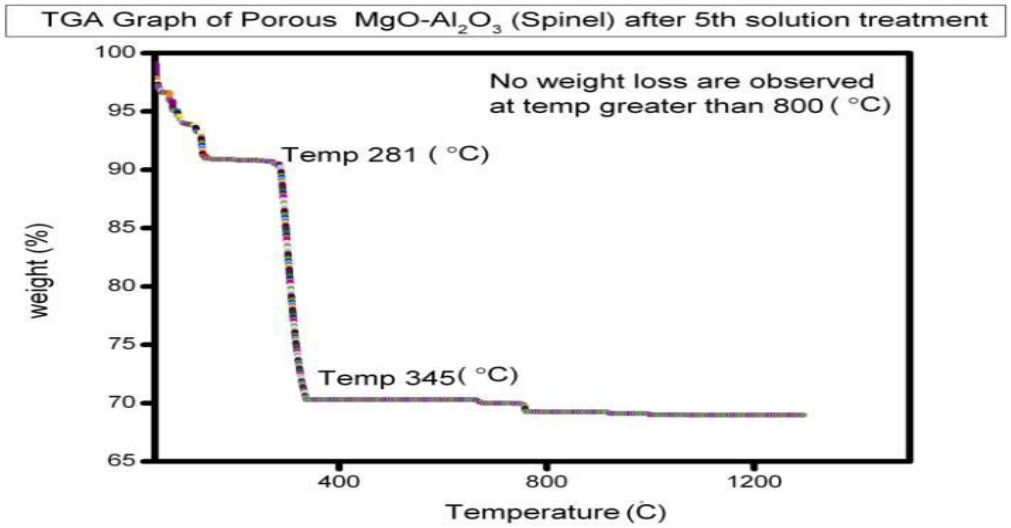


**Figure 4.9 Thermal gravimetric analysis curve of PMS5**

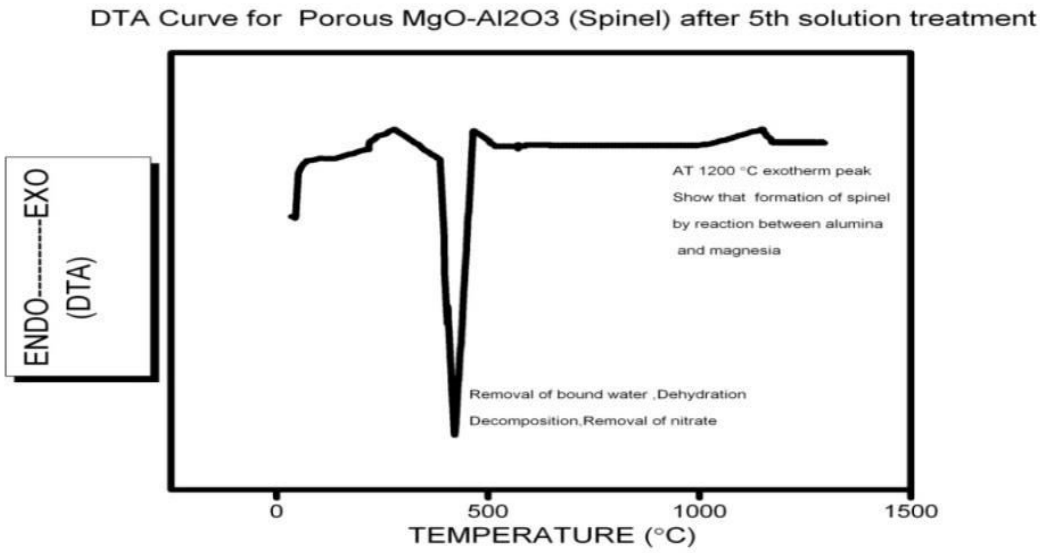


**Figure 4.10 DTA of Spongy MgO with 5 number of elucidation healing**

From the figure 4.9 and 4.10 Shows ,no credence loss were found subsequent to a temp 800 ℃ , Endothermic peaks shows elimination of unboun water in a material where as exothermic paeks demonstrate Spinel configuration at 1200 °C.

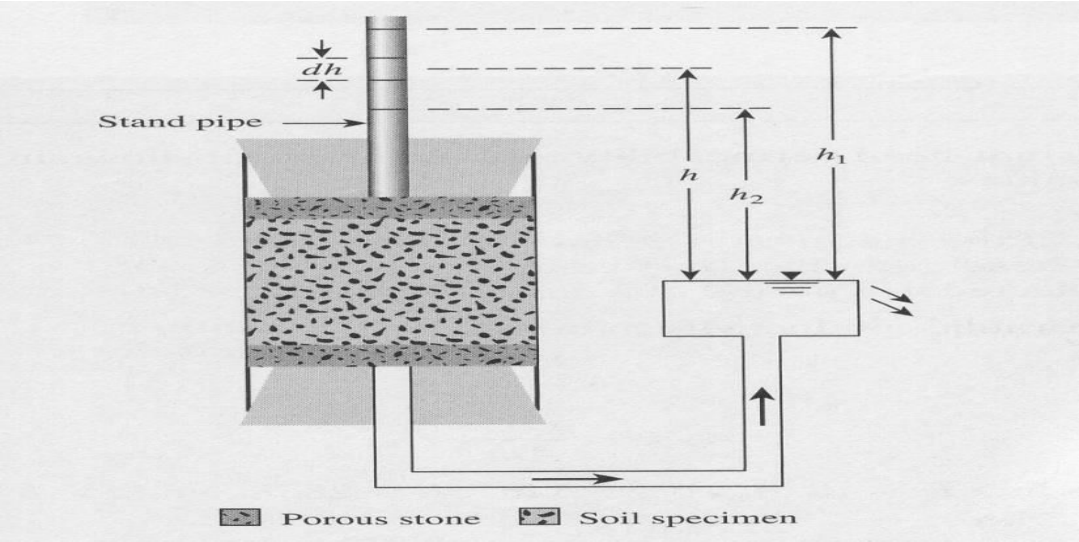


**Figure 4.11 Water Falling head test method**

The assess of gush from side to side porosity in the midst of in a body propensity to authorize irrigate to surge during its minute opening or holes [ West , (2003) ] . Figure 4.11 shows pearmebility by Darcy law.The limitation q during Darcy's decree is permit the surge tempo or merely the gush (unsteadiness It portray in a entity moment in time greater than a entity side view area, how much dampen in requisites of capacity has be alive gush through**.** Permeability unhurried of crowded refractories confer an suggestion of how in good health that refractory spirit locate up to slag, liquefy or chatter dissemination for the duration of once-over (squat permeability = worse slag, liquefy or chatter penetration resistance) [Salvini, (2003)] .This technique are worn in the direction of determine the permeability [Nandi ,(1987)].

$$K=k\ 1\ \ln (h1/h2)$$

everywhere k1 = proportion of fractious sectional vicinity of reputation pipes and water tank From the figure 4.12 shows pearmeabilty is maximum for Spongy MgO with no elucidation treatment

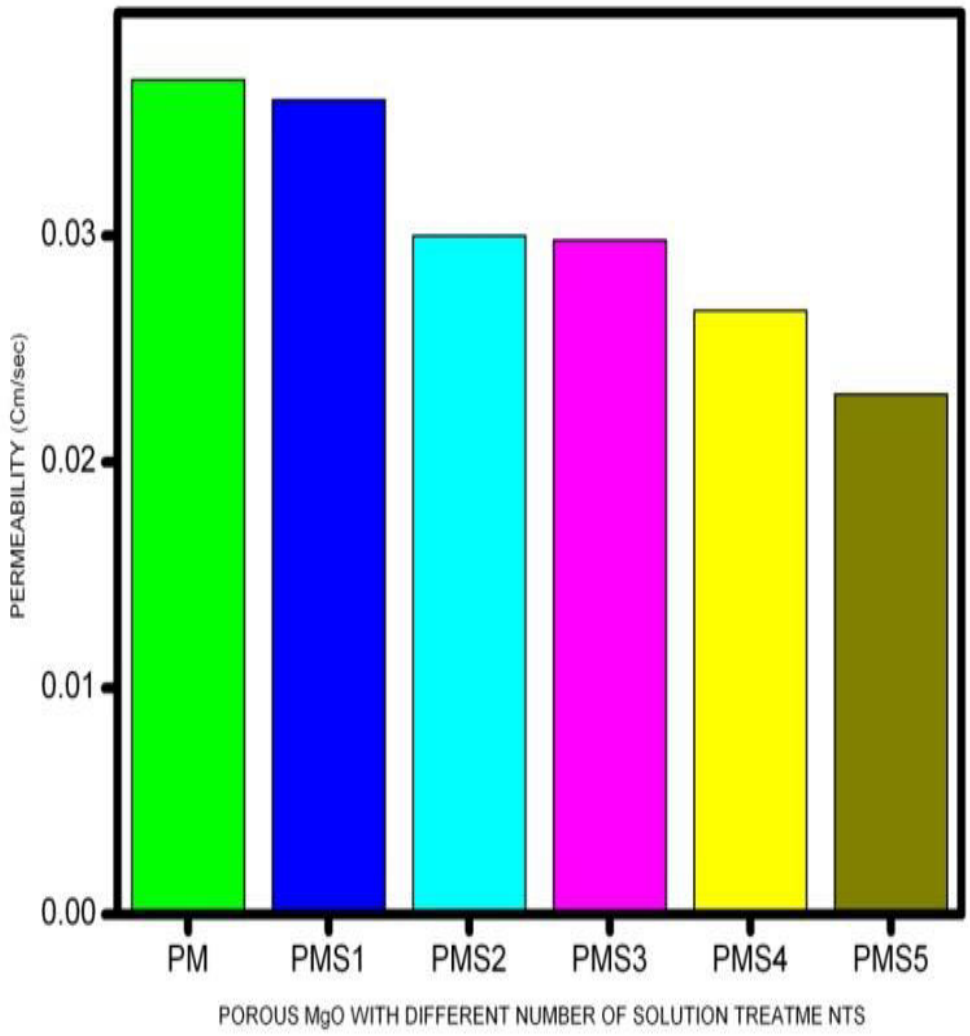


**Figure 4.12 Pearmeabilty of Spongy MgO with different number of elucidation treatment.**

From the figure 4.13 Shows that Relative density , is maximum for fifth elucidation treatments in terms of quotient of the compactness (gathering of a unit dimensions of a stuff to the compactness of a kerosene orientation material. Specific gravity regularly means comparative density with esteem to water/kerosene. The tenure "relative density" is often favored in contemporary precise usage [Salvini, (2003)]. It is defined as a ratio of density of fastidious stuff with that of dampen /kerosene. Bulk density is the bunch in excess of the entire capacity density is the mass greater than the dimensions not counting the emptiness [ West , (2003) ].From Figure 4.14 True porosity of Spongy MgO with no elucidation treatment are maximum.

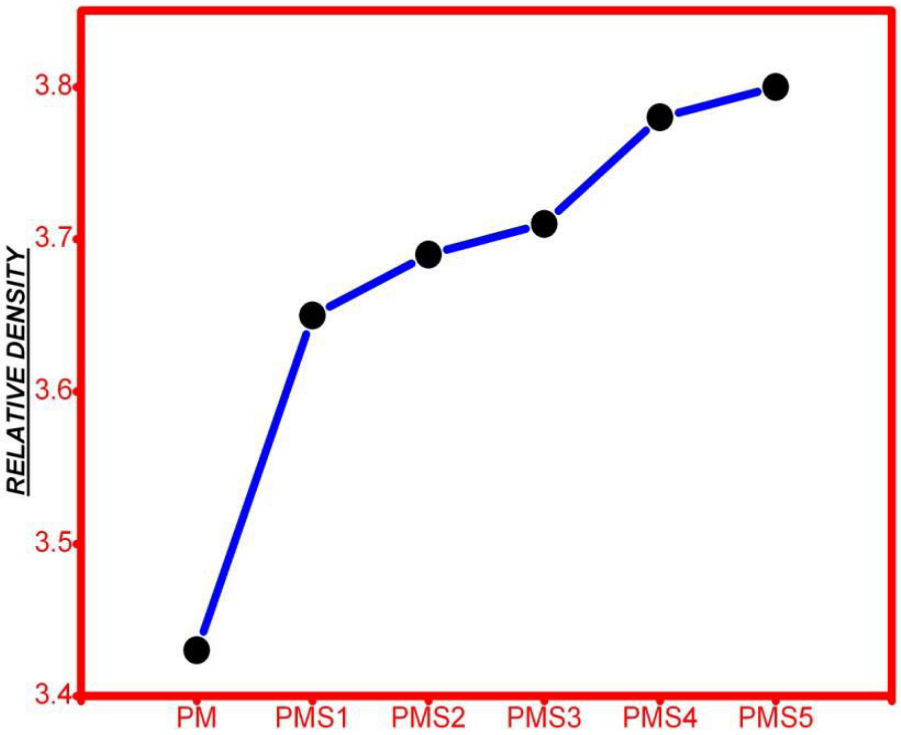


**Figure 4.13 relative density of Spongy MgO with diverse number of elucidation dealing.**

True porosity = ( 1 - ( Ga / Gt ) ) * 100 )

**where,**
Ga - Apparent solid specific gravity
Gt - True specific gravity

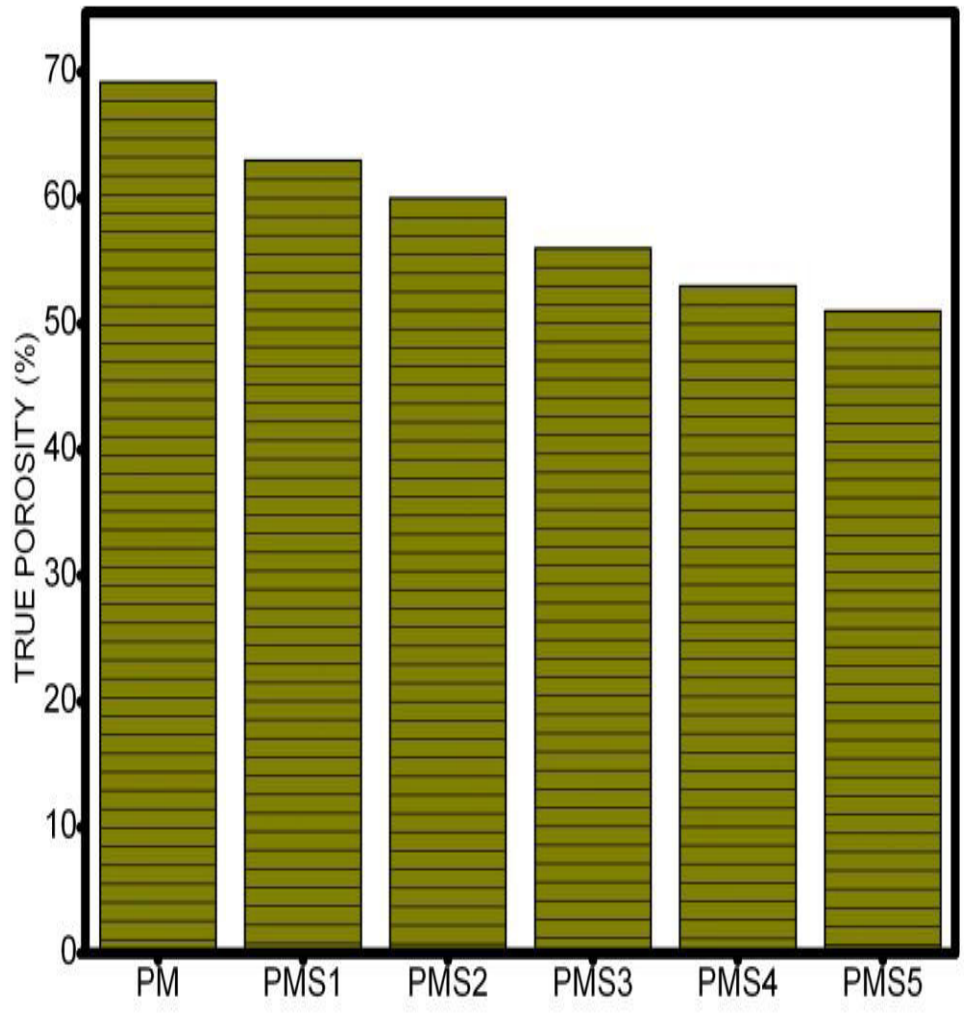


**Figure 4.14 True porosity of Spongy MgO with diverse number of elucidation treatment.**

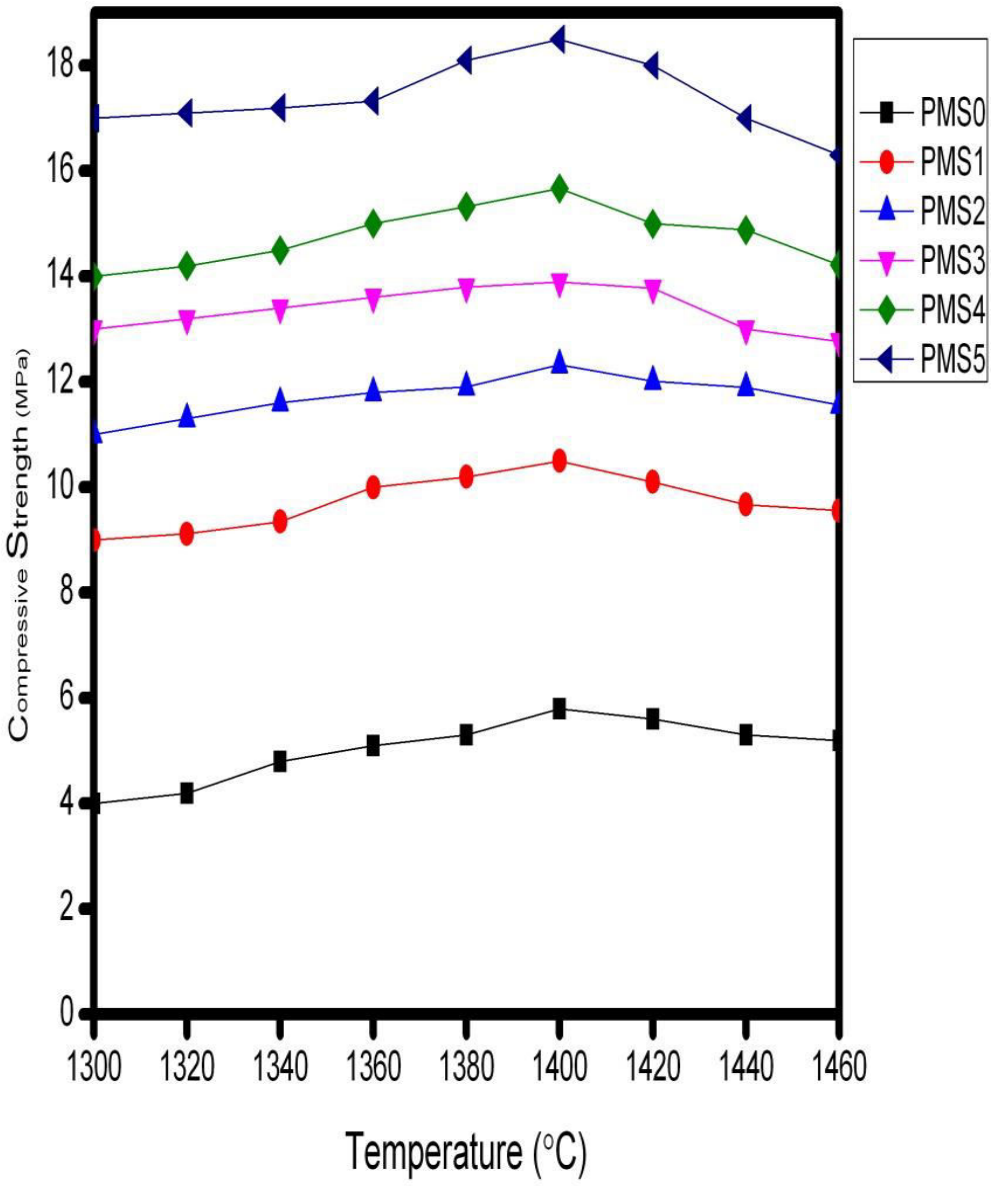


**Figure 4.15 Thermal shock Measurement of Spongy MgO with different number of elucidation treatment by compressive strength.**

From the figure 4.15 It is lucid that compressive strength are stridently decreases at the temperature 1400 to 1420 ℃ ,this is due to rising of thermal shock in a different solution treatments.

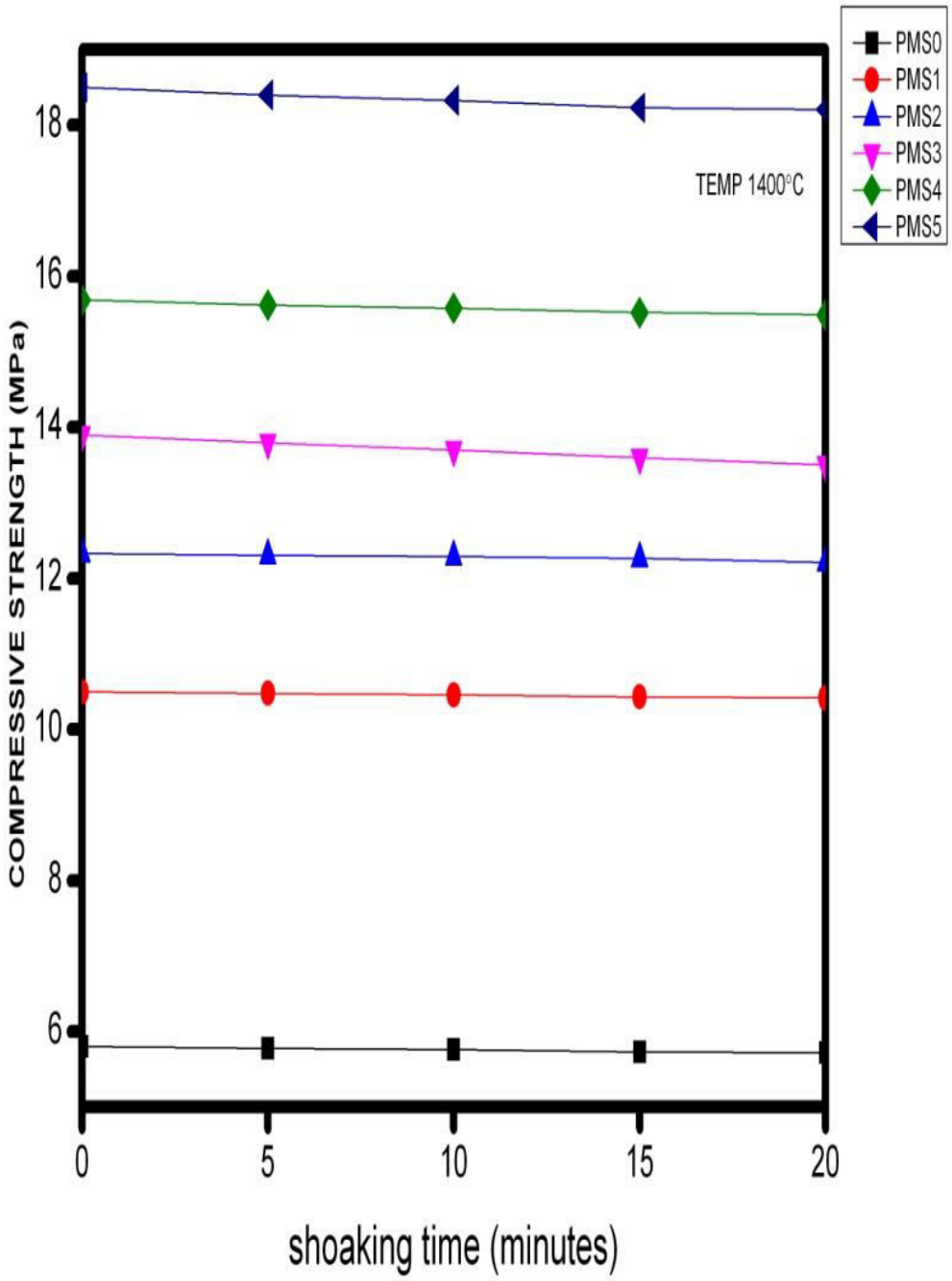


**Figure 4.16 Thermal shock Measurement of Spongy MgO with different number of elucidation treatment at temp 1400◦C SP 5 min.**

From the figure 4.16 it is clear that compressive strength are decreases at soaking time due to material swelling and thermal shock rising in a material.

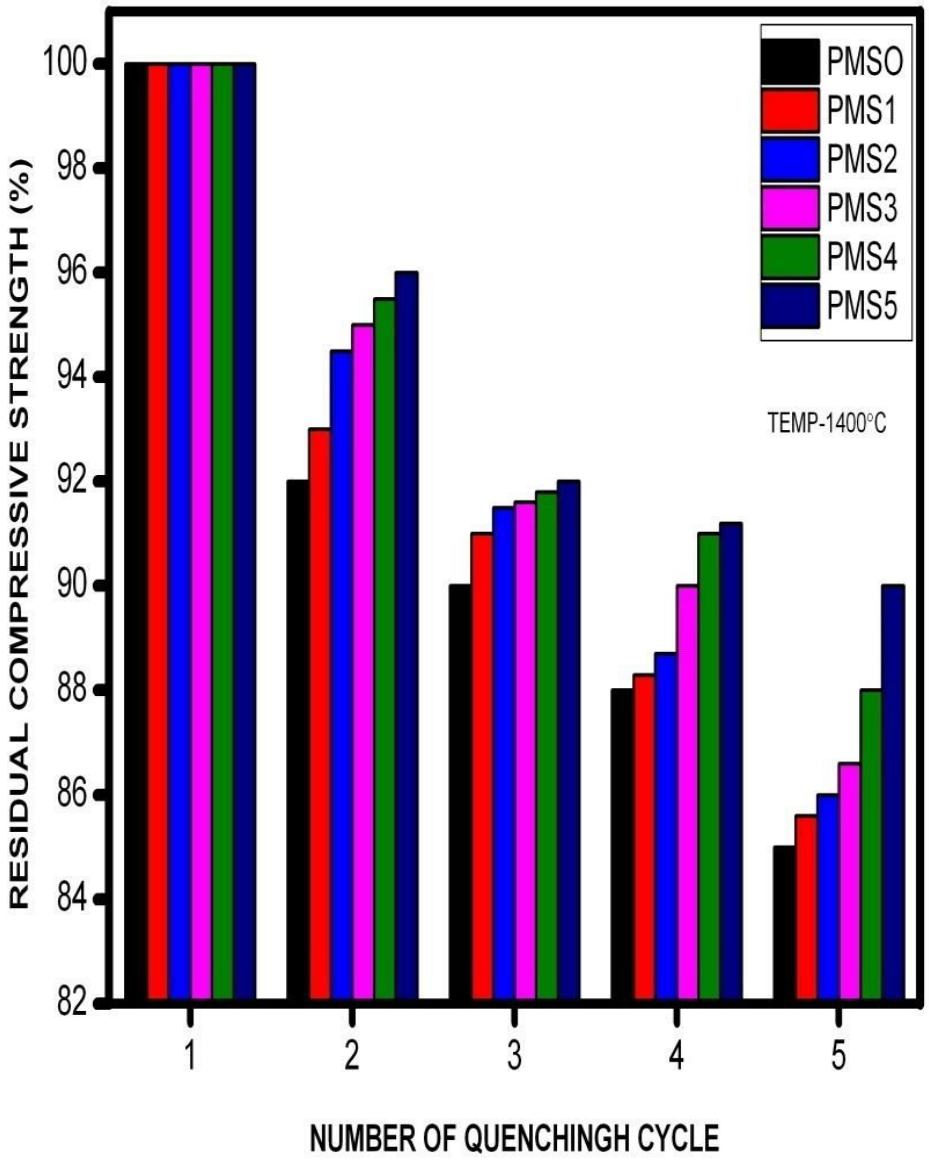


Figure 4.17 Residual compressive strength of Spongy MgO with diverse number of elucidation treatment at temp 1400◦C

From the figure 4.17 Residual Compressive strength is minimum for no elucidation treatments due to high porosity and low density its compactness are very low.

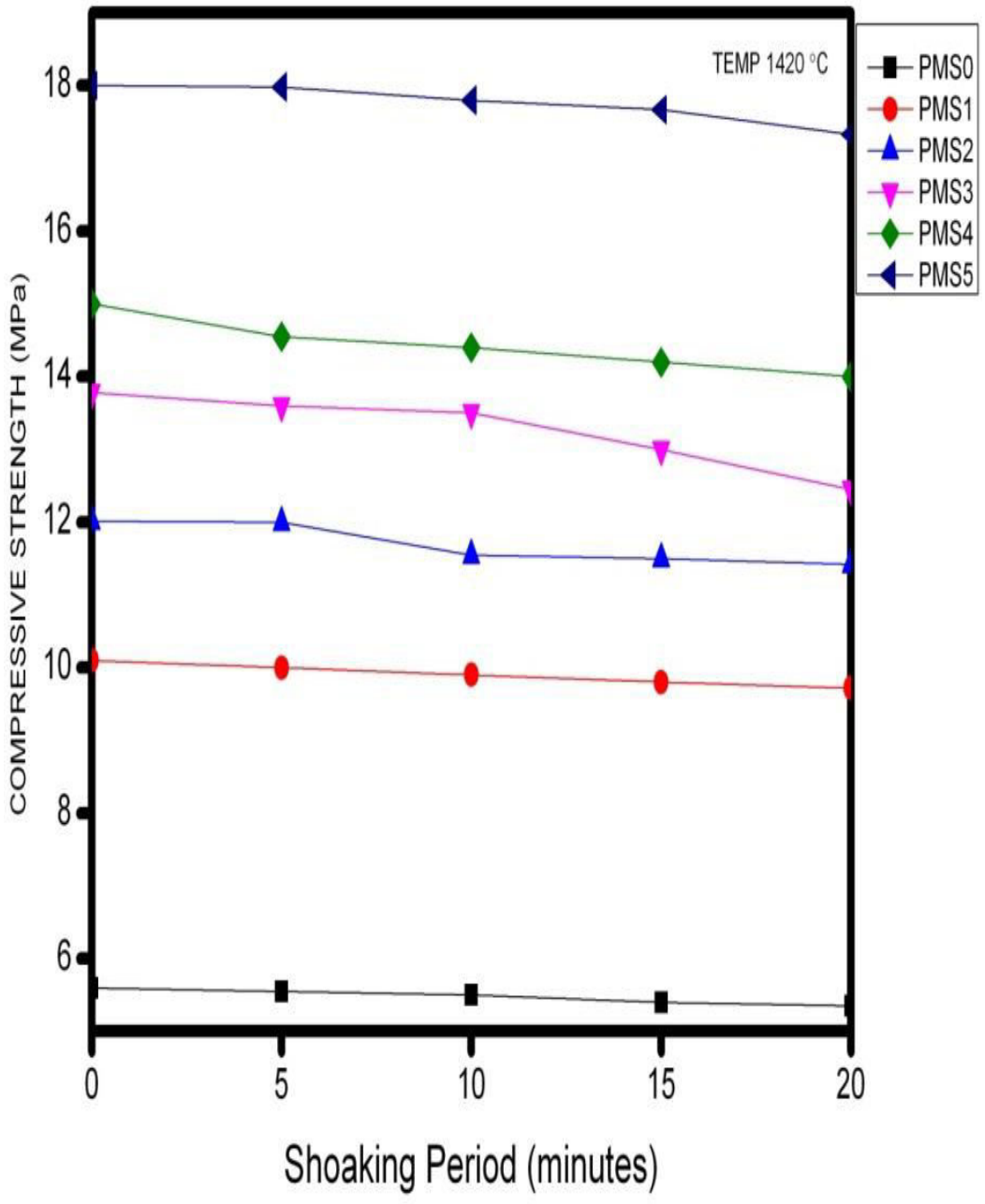


**Figure 4.18 Thermal shock Measurement of Spongy MgO with dissimilar number of elucidation treatment at temp 1420◦C**

From the figure 4.18 A Sharp decrease in Compressive strength at temp 1420°C for PMSO PMS2 ,PMS3,PMS4,PMS5 this is due to rising of thermal shock in a different solution treatments.

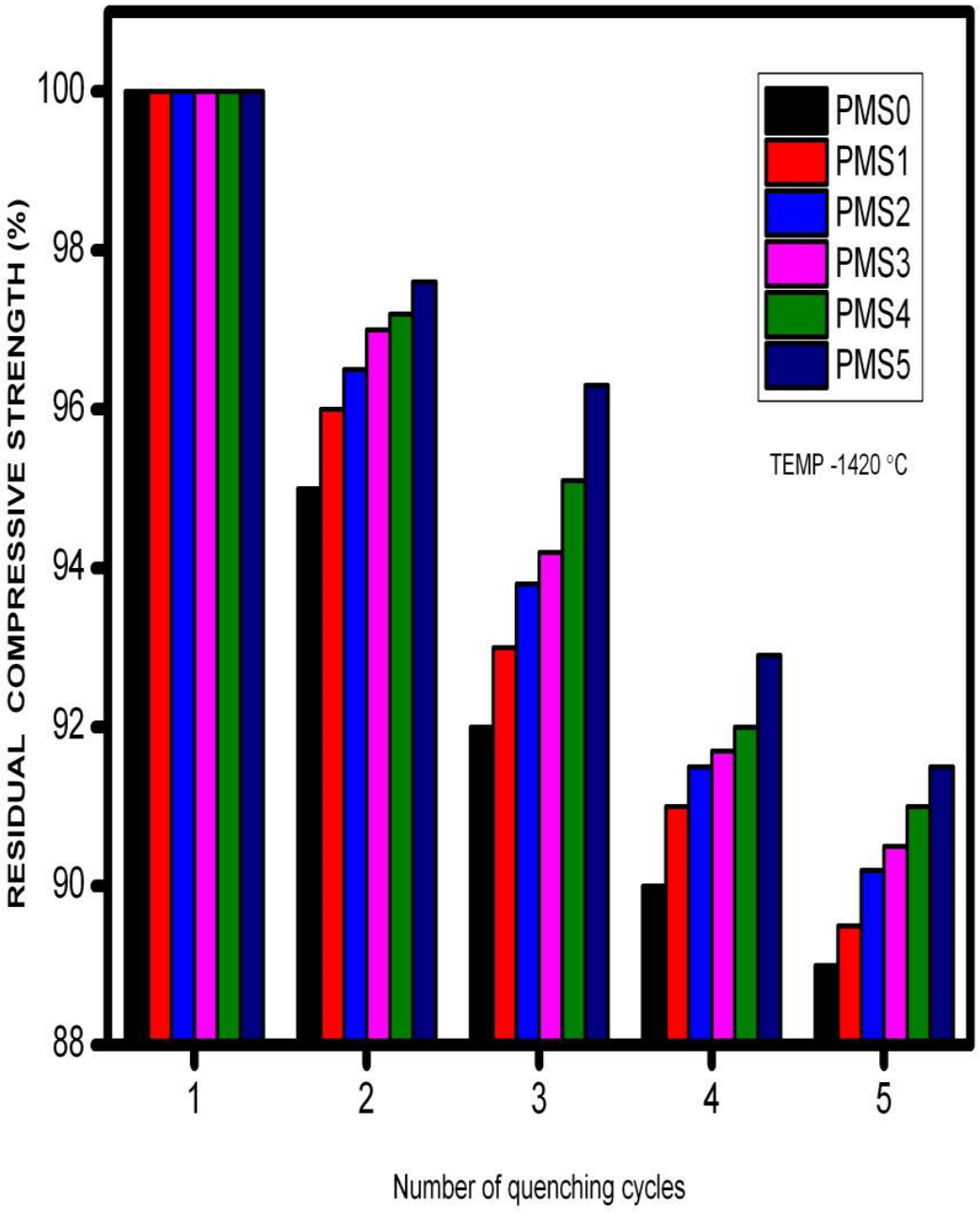


**Figure 4.19 Residual compressive strength of Spongy MgO with different number of elucidation treatment at temp 1420◦C**

From the Figure 4.19 Residual Compressive strength is is minimum for no elucidation treatments.

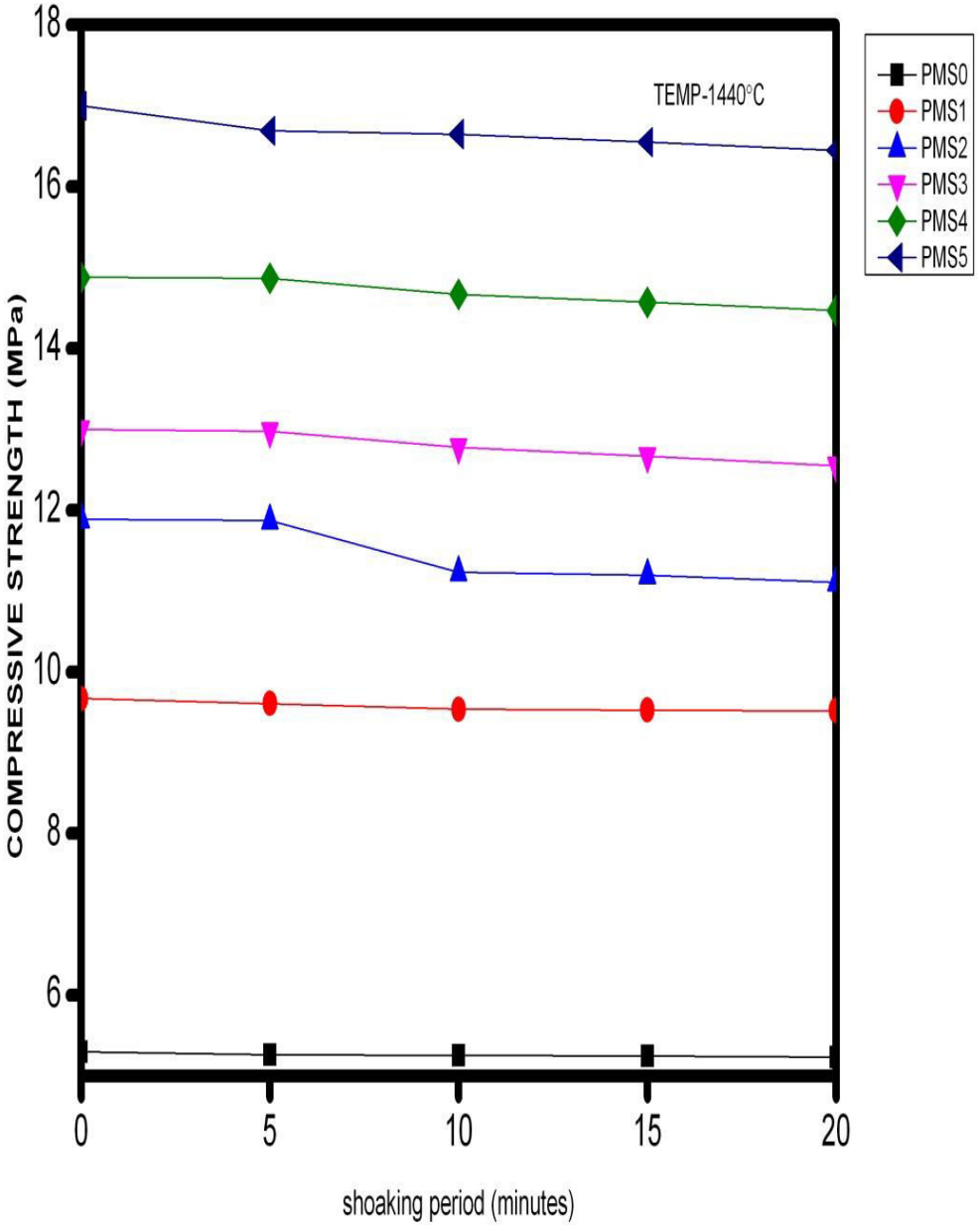


**Figure 4.20 Thermal shock Measurement of Spongy MgO with diverse quantity of elucidation treatment at temp 1440◦C**

From the figure 4.20 A Sharp decrease in Compressive strength at temp 1440°C for PMS5 and PMS2

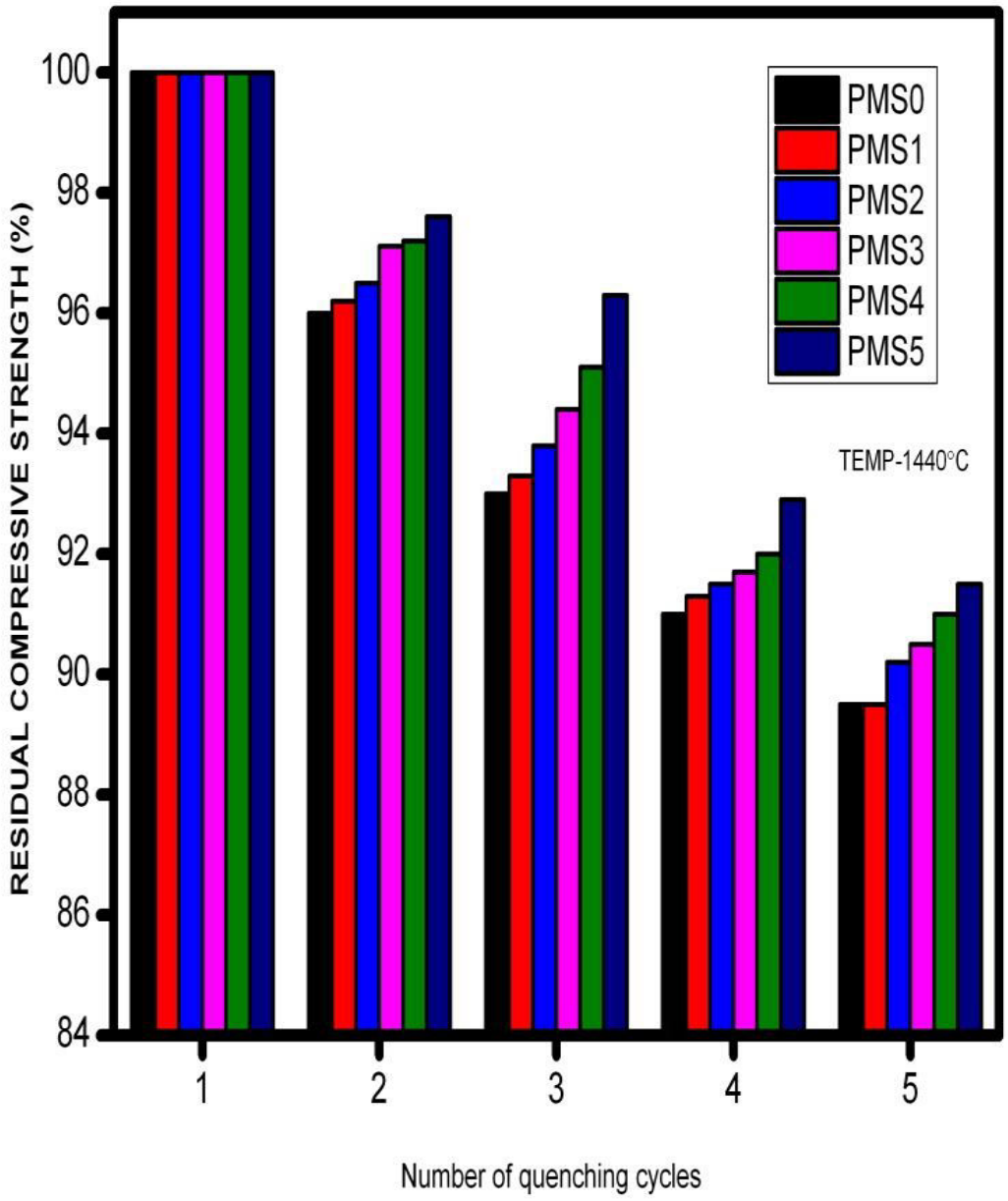


**Figure 4.21 Residual Compressive Strength of Spongy MgO with diverse number of elucidation treatment at temp 1440◦C**

From the figure 4.21 Residual Compressive strength is is minimum for n elucidation treatments due to high porosity and low density its compactness are very low.

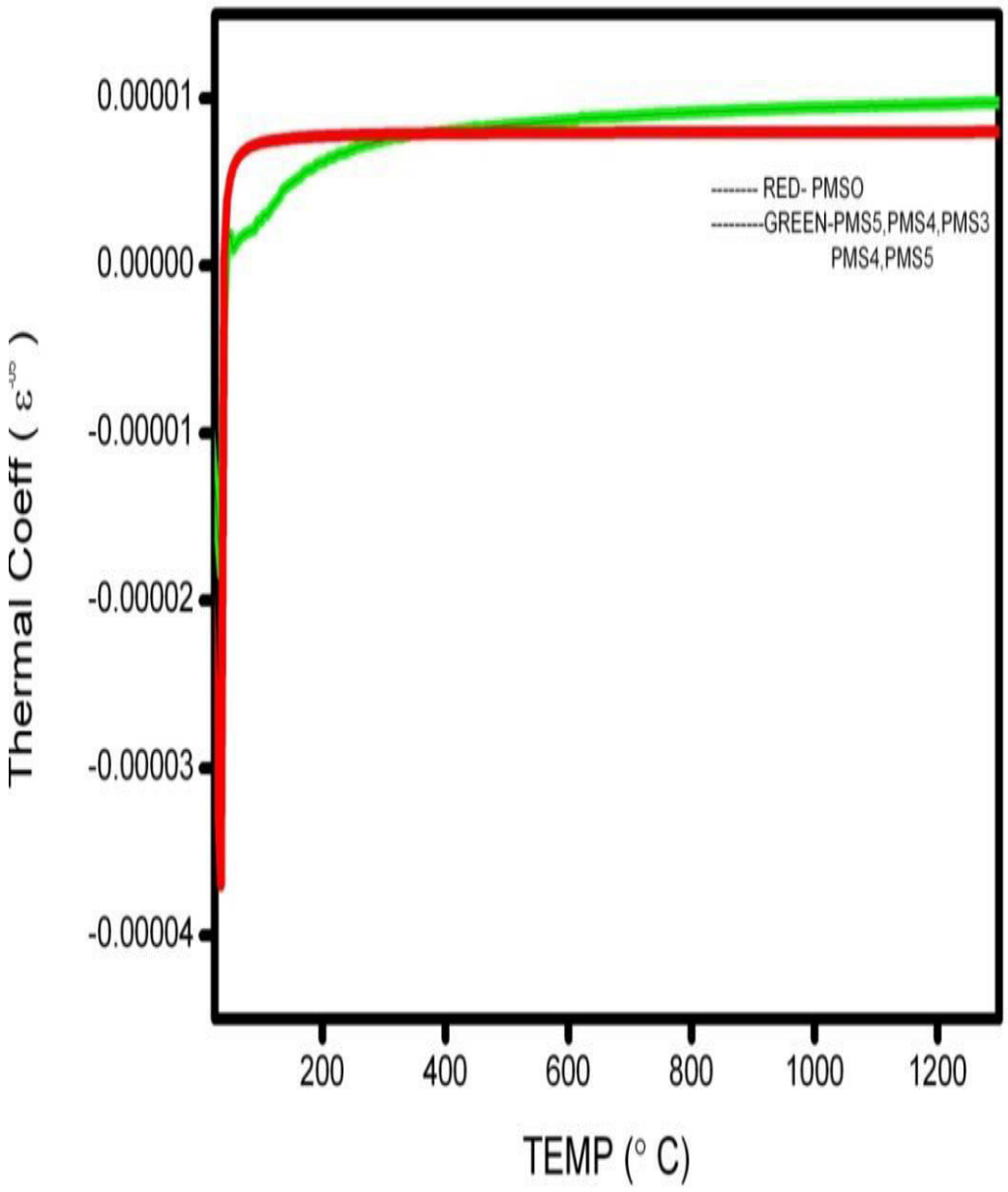


**Figure 4.22 Thermal Exp -- of Spongy MgO with different number of elucidation treatment at temp 1440◦C**

From the figure 4.22 Shows that thermal co-eff were constant after a temperature 300 °C.It is very good indication shows less impurity present in a material and material shows constant value at a particular > 300 °C fixed temperature.