

Effect of Colloids Impregnation on Permeability Reduction of Corundum Bricks

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ABSTRACT

Refractory bricks have higher permeability than monolithics. From discoloration and crack formation it is likely that massive penetration of impurities in the bulk of the refractory is taking place. Permeability of the refractory is an important characteristic in that case, and impregnation of the refractory to make a barrier may increase lining life. In order to get lower permeability of corundum bricks, several SiO_2 and Al_2O_3 colloids are applied to impregnate the bricks. The permeability are tested by home-made equipment and calculated through Darcy's Law for a linear flow. The results show Al_2O_3 colloid can make permeability much dropper, compared with SiO_2 colloid. Vacuum sol impregnation processing is an effective method to decrease permeability. This paper discusses the application of Darcy's law and variation of permeability with pressure gradients at different treatment temperatures (110°C, 1200°C and 1600°C).

INTRODUCTION

Corundum bricks were installed as work lining in a furnace for a while. The maximum service temperature is 1600°C. From discoloration and crack formation it is likely that massive penetration of impurities in the bulk of the refractory is taking place. Permeability of the refractory is an important characteristic in this case, and impregnation of the refractory to make a barrier may increase lining life. Small samples that were impregnated are scheduled to be tested in laboratory. Several SiO_2 and Al_2O_3 colloid sols are prepared to impregnate samples of corundum bricks. A home-made equipment is used to get data of pressure and flow rate. Permeability are calculated through Darcy's Law.

Darcy's Law

Permeability is part of the proportionality constant in Darcy's law which relates discharge (flow rate) and fluid physical properties (e.g. viscosity), to a pressure gradient applied to the porous media.

Darcy's Law for incompressible fluids:

$$v = \frac{k}{\mu} \frac{\Delta P}{\Delta x} \quad (\text{for linear flow})$$

Therefore:

$$k = v \frac{\mu \Delta x}{\Delta P} \quad \text{where:}$$

v is the superficial fluid flow velocity through the medium (i.e., the average velocity calculated as if the fluid were the only phase present in the porous medium) (m/s)

k is the permeability of a medium (m^2)

μ is the dynamic viscosity of the fluid ($\text{Pa}\cdot\text{s}$)

ΔP is the applied pressure difference (Pa)

Δx is the thickness of the bed of the porous medium (m)

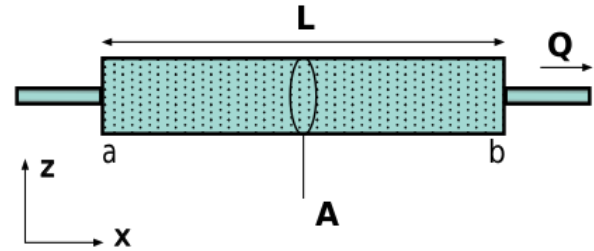


Figure 1: Diagram showing definitions and directions for Darcy's law

In naturally occurring materials, the permeability values range over many orders of magnitude

Deviating from Darcy's linear law, the porous flow characteristics obey a nonlinear law in a low-permeability porous medium, the viscosity of the porous flow fluid and the permeability values of fluid are not constants^[1-8].

An incompressible fluid cannot be compressed and has relatively constant density throughout. Liquid is an incompressible fluid. A gaseous fluid such as air, on the other hand, can be either compressible or incompressible. Generally, for theoretical and experimental purposes, gases are assumed to be incompressible when they are moving at low speeds--under approximately 220 miles per hour (98,3 m/s).

Darcy's law, which states that a fluid flow rate is directly proportional to the pressure gradient, is shown to be accurate only at low flow velocities, both in liquid and gas phase systems. At higher flow rates, Darcy's law is usually replaced by the Forchheimer equation, which includes a term that is quadratic in the flow rate.

In sum, Darcy's Law is valid for incompressible fluids, linear flow, and low flow velocities.

Permeability measurement

The apparatus fabricated for measuring permeability is presented in Figure 2. Cylindrical samples of 50 mm x 50 mm, fired at different temperature are coated with a thermo-setting resin on the side surface and cured for overnight at 50°C. This is to prevent the gas leak though side surfaces while measuring permeability. Compressed air at a pressure of 0.5 to 6.5 bars was passed through the sample to measure the flow rate of gas (ml/min or l/min). The Darcy's Law is applied to evaluate the permeability of material.

Darcy's law, as refined by Morris Muskat, in the absence of gravitational forces and in a homogeneously permeable medium, is given by a simple proportionality relationship between the instantaneous flow rate through a porous medium, the permeability of the medium, the dynamic viscosity of the fluid, and the pressure drop over a given distance.

Fundamental assumptions inherent in these test methods are defined by linear flow and without velocity decay along the porous media samples.

When the law is applied to porous media such as refractory, following conditions are neglected.

Non-linear flow: Flow in porous media is non-linear in nature in that the shear factor, the dispersion coefficient and the macroscopic viscosity are functions of flow velocity.

Velocity decay: flow velocity is fluctuant and dispersive in a simple porous medium.

The viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress. Air dynamic viscosity at given temperatures and atmospheric pressure can be found by www.engineeringtoolbox.com.

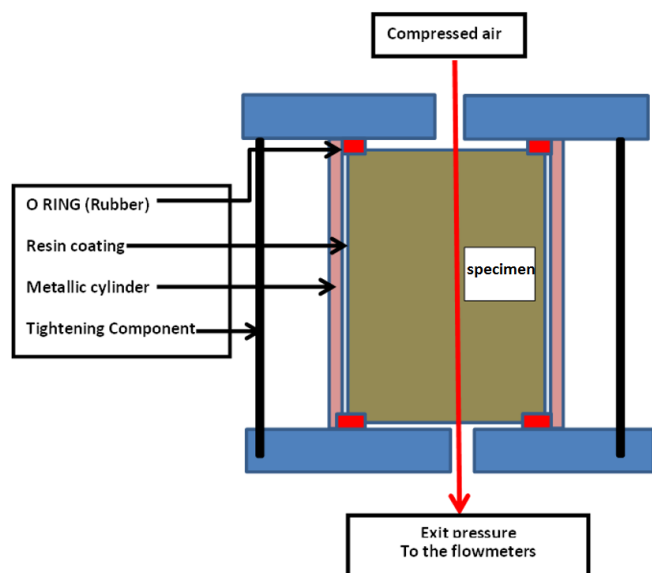


Figure 2: Schematic representation of permeability tester fabricated in laboratory

In this study, the exit pressure is supposed as a standard atmosphere and omitted. Therefore, compressed air at a pressure of 0.5 to 6.5 bars could be considered as pressure gradient or difference.

CYLINDER SPECIMEN IMPREGNATION PREPARATION AND PERMEABILITY TESTING

1. Corundum Brick Technical Information

Table 1 Corundum Bricks Technical Information

High alumina brick based on sinter alumina	
Bulk density [g/cm ³]	3,05
Apparent porosity [%]	12
Cold crushing strength [MPa]	175
Permanent linear change [%] at 1600°C/5h	0,4
Linear thermal expansion [%] at 1000°C	0,7
Aluminium Oxide, Al ₂ O ₃ [%]	90
Silicon Dioxide, SiO ₂ [%]	9

2. Corundum Brick Cylinder Specimens Impregnation Preparation

Ø50 mm x 50 mm cylindrical specimens are drilled through corundum bricks, and drying at 110°C for 24 hours. Multi-colloid sols are ready for impregnation, details are shown in Table 2.

Apparent porosity testing vacuum-pressure vessel is hired for impregnating vacuum procedure.

Table 2 Corundum Cylinder Specimens Impregnation Preparation

No.	Sol	Concentration	Impregnation	Vacuum	Soaking
S1	SiO ₂	20%	Soaking	n/a	68 hr
S2	SiO ₂	30%	Soaking	n/a	68 hr
S3	SiO ₂	40%	Soaking	n/a	68 hr
S4	SiO ₂	30%	Vacuum	1hr	1hr
S5	reference	Corundum brick specimen without impregnation			
S6	Al ₂ O ₃	25%	Soaking	n/a	68 hr
S7	Al ₂ O ₃ -1	50%	Soaking	n/a	72hr
S8	Al ₂ O ₃ -2	50%	Soaking	n/a	72hr
S9	Al ₂ O ₃ -1	50%	Vacuum	1 hr	1 hr
S10	Al ₂ O ₃ -2	50%	Vacuum	1 hr	1 hr

Note: S1 and S6 are dilute solutions of S2 and S7 respectively.

2. Permeability Testing

Compressed air at a pressure of 0.5 to 6.5 bars is passed through the specimen to measure the flow rate of air (ml/min or l/min). Applying Darcy's Law to calculate the permeability of each specimen.

TEST RESULTS AND DISCUSSION

1. Effect of SiO₂ sol impregnation on permeability of corundum bricks

There are three silica sols are used as impregnating solutions in this study, S1, S2 and S3 respectively. S1 is a dilution of S3. Tables 3 shows the typical properties.

Table 3 Silica Sols Typical Properties

Sol	S2	S3
Silica, wt%:	30	40
Specific surface area, m ² /g	360	220
pH:	10,1	9,8
Viscosity, cP	6	15
Density, g/cm ³	1,2	1,3
Na ₂ O, wt%	0,6	0,42

The testing results are graphically shown in Figure 3, 4 and 5. S5 is a reference specimen which is corundum brick without any impregnation.

Permeability of reference specimen increases with pressure gradient, which is common as regular refractory samples. However, permeabilities decline with pressure gradient for silica sols except S1.

Permeabilities decline at lower pressure gradient. Explanation of this phenomenon could be related to initiative capillary hysteresis and chemical incompatibility.

S1, water diluted solution shows a constant permeability comparatively. Water has a higher wettability to alumina base, that could relief chemical incompatibility and even capillary

hysteresis. Water's compatibility to alumina base can also explain S1's permeability drops with treatment temperature.

S3 has a lower permeability than S2 duo to a lower pH value, which is easy to wet to neutral alumina base.

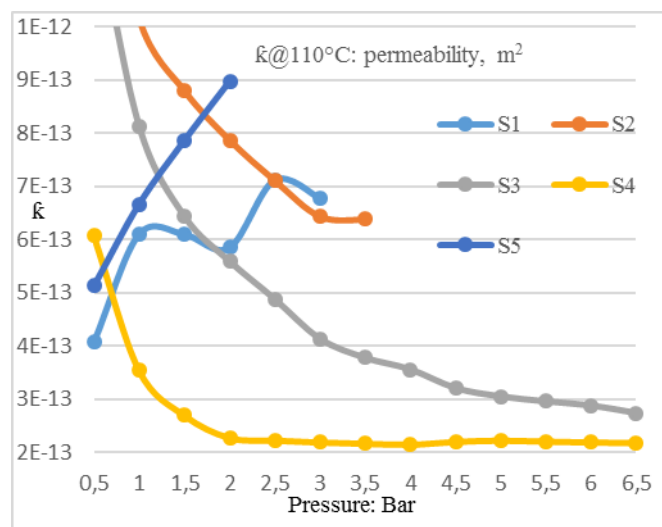


Figure 3: Permeability vs. Pressure gradient: corundum cylinder specimen dried at 110°C after silica sol impregnation

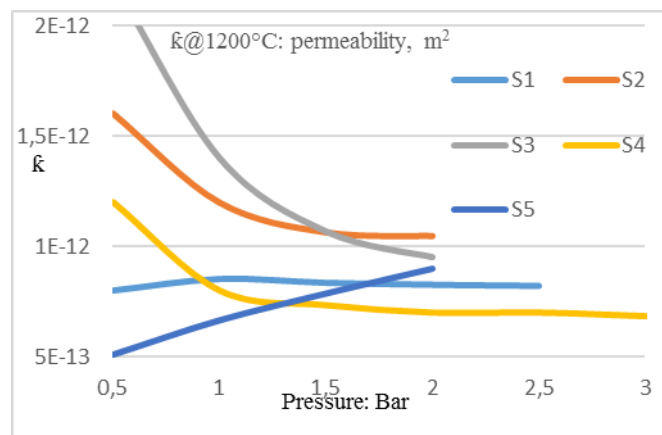


Figure 4: Permeability vs. Pressure gradient: corundum cylinder specimen fired at 1200°C after silica sol impregnation

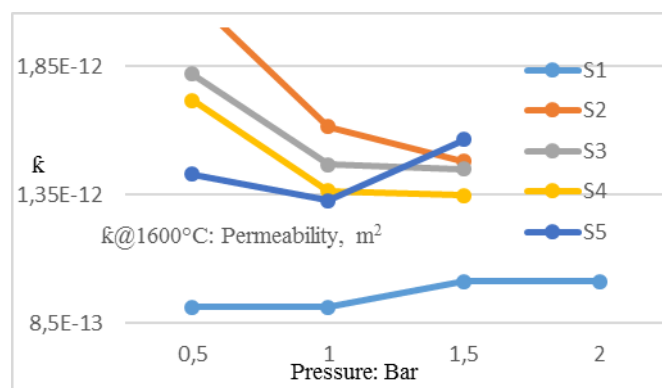


Figure 5: Permeability vs. Pressure gradient: corundum cylinder specimen fired at 1600°C after silica sol impregnation

Generally, silica sols impregnation cannot significantly reduce specimen's permeability.

This is caused by the absorption of silica colloidal particles with negative charge on the surface of alumina base.

With negative charges, silica colloidal particles are absorbed on the surface of alumina base. Figure 6 illustrates a schematic view of an alumina–water–silica sol system before and after freezing [9,10].

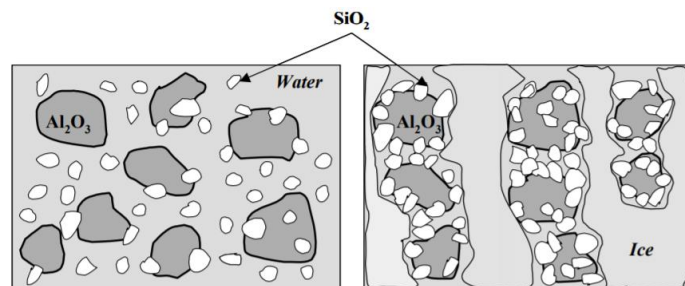


Figure 6: A schematic view of freeze-gelation process of alumina ceramics by silica sol [9,10]

Silica particles cannot scatter around the pores or channels. Pores or channels are apparently separate from alumina base, which shown in right of Figure 6.

To sum up, colloidal silica sols do not have an appreciable effect on permeability reduction of the corundum bricks.

2. Effect of Al₂O₃ sol impregnation on permeability of corundum bricks

There are three alumina sols are used as impregnating solutions in this study, S6, S7 and S8 respectively. S6 is a dilution of S7. Colloidal sol S8(Al₂O₃-1), which show in Table 4. Nevertheless, colloid S8(Al₂O₃-2) is a micro-modified sol which expect the properties are improved.

Table 4 Alumina Sol Characteristic Physico-chemical Data

Al ₂ O ₃ %, wt.	50±1
pH	7--10
Viscosity, cP	< 1000
D50, nm	80
Density, g/cm ³	1,52

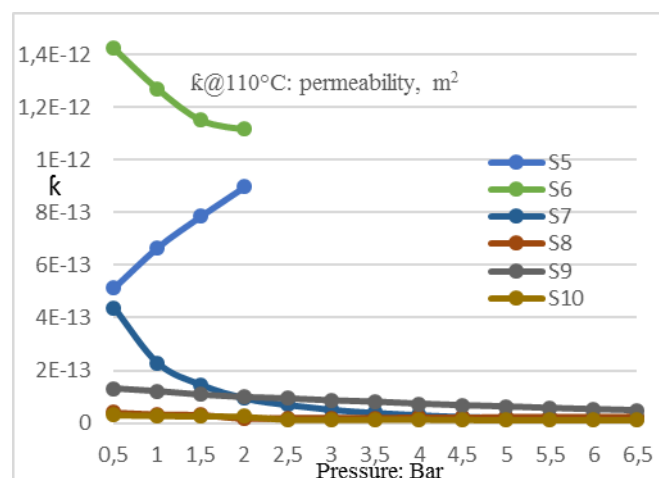


Figure 7: Permeability vs. Pressure gradient: corundum cylinder specimen dried at 110°C after alumina sol impregnation

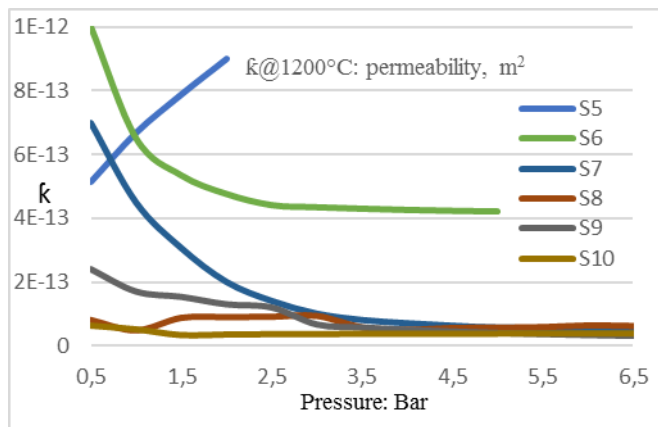


Figure 8: Permeability vs. Pressure gradient: corundum cylinder specimen fired at 1200°C after alumina sol impregnation

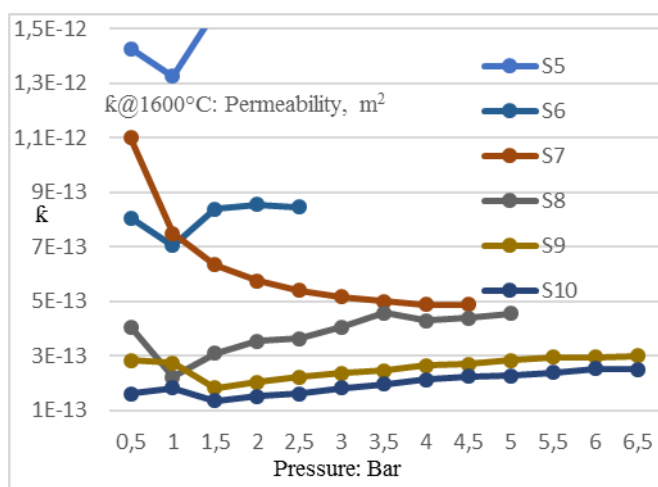


Figure 9: Permeability vs. Pressure gradient: corundum cylinder specimen fired at 1600°C after alumina sol impregnation

Evidently, Alumina sols impregnation can easily reduce permeability of corundum bricks through Figure 7-9. Chemical compatibility can be explain this phenomenon. Chemical compatibility is a measure of how stable a substance is when mixed with another substance. If two substances can mix together and undergo a chemical reaction, they are considered incompatible. Alumina sols are good compatible to its own base. In other words, alumina sols have a better wettability to alumina porous ceramic. Accordingly, alumina sols have a positive effect on permeability reduction of the corundum bricks.

S6, water diluted solution shows a higher permeability than other alumina sols. In this case, alumina sols indicate a better chemical compatibility to alumina base than a dilution.

Micro-modified sol S8 shows better permeability reduction, compared to S7. It is probably related to its proprietary dispersing agent. S8 can even more eliminate the phenomenon of capillary hysteresis.

3. Effect of treated temperature on permeability of corundum bricks

Through Figure 3-5 and 7-9, it is clear that permeability of corundum bricks increase with higher treated temperatures. High temperatures clean pores or channels, that have air passing through the brick easily.

4. Vacuum procedure improves permeability reduction

Obviously, vacuum procedure can reduce permeability through Figure 3-5 and 10-12 regardless of silica or alumina sols.

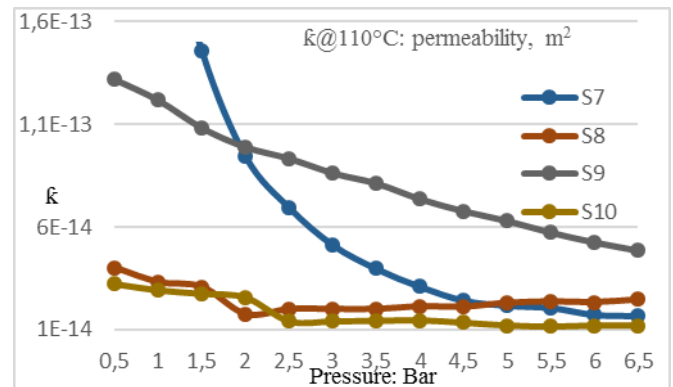


Figure 10: Enlargement brings out the detail in Figure 7

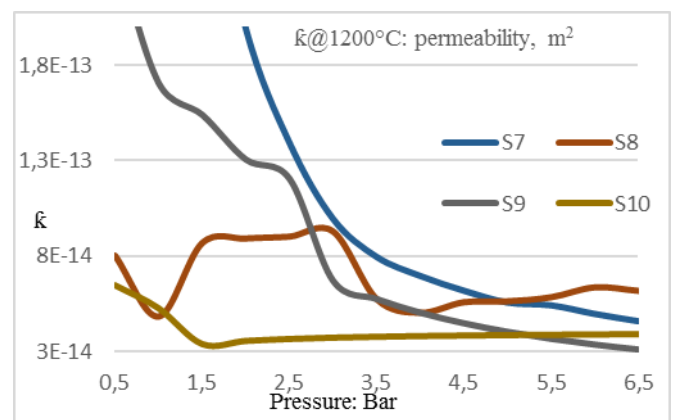


Figure 11: Enlargement brings out the detail in Figure 8

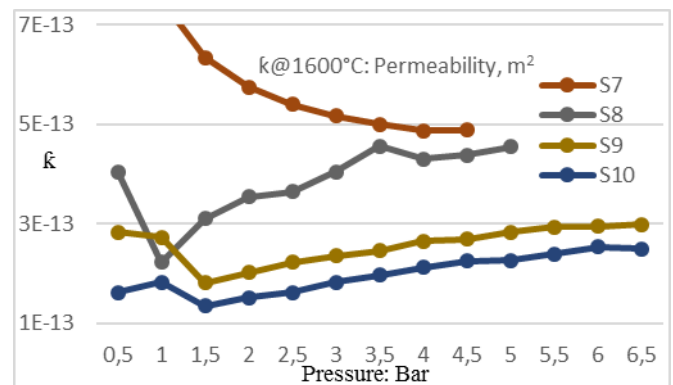


Figure 12: Enlargement brings out the detail in Figure 9

5. Comparison with corresponding castables

Cast LC90, 90% Al_2O_3 low cement castable, has a similar properties to the tested corundum brick, which shown in Table 5.

Figure 13 indicates that the specimen impregnated in S8 (micro-modified alumina sol) has a same level permeability as 90% Al_2O_3 low cement castable Cast LC90 when treated temperature is 1600°C, which is the maximum service temperature of corundum bricks.

When applied a vacuum procedure, the corundum bricks will have the lowest permeabilities (S9 and S10 show that cases in Figure 13).

Table 5 Cast LC 90 Castable Technical Information

High alumina castable based on sinter alumina		
Bulk density [g/cm ³]	800°C	3,05
Apparent porosity [%]	after 1430°C	16
Cold crushing strength [MPa]	1600°C	280
Permanent linear change [%] at 1600°C		-0,5
Reversible thermal expansion [%] 20--1000°C		0,8
Aluminium Oxide, Al ₂ O ₃ [%]		92
Silicon Dioxide, SiO ₂ [%]		6

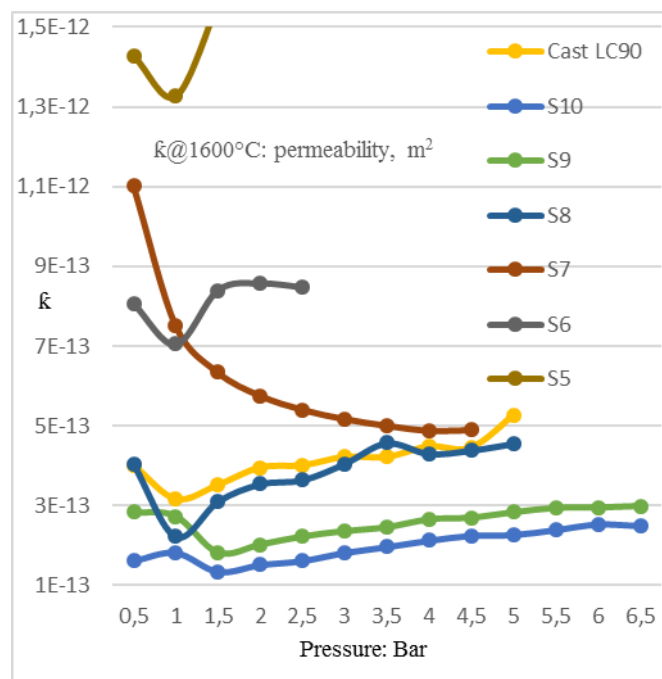


Figure 13: 1600°C Permeability comparison: 90%Al₂O₃ low cement castable (Cast LC90) vs. corundum brick & it impregnated in alumina sol

CONCLUSIONS

The colloidal alumina sols have an appreciable effect on permeability reduction of the corundum bricks. However, colloidal silica sols do not. Vacuum procedure applies a positive improvement on permeability reduction. Alumina sol impregnation could have the corundum bricks the same or even better level permeability as corresponding castable.

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