Investigation of Testing Parameters Influencing the Permanent Linear Change Testing Results of Dense Refractory Bricks

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Current standards for the determination of physical and chemical properties of refractory materials disclose little or no data on precision values. But, such data are basic information for communication between refractory producer and final user. Sometimes differences between the values of properties reported by specialized testing laboratories and those provided by the refractory suppliers lead to discussions, or even claims. This paper reports some results on permanent linear change (PLC) testing of refractory materials, carried out according to EN 993-10. These data were obtained within the frame of the European funded research project known as ReStaR which included a multinational consortium of 13 partners including six independent laboratories. A factorial design of the experiments, followed by an ANOVA analysis, was carried out in order to identify the influencing factors. In the case of Permanent Linear Change (PLC) up to five influencing factors were investigated.

1 Introduction

Most standards for the determination of physical properties of refractories disclose little or no data on the method's precision values. However, such data are the basis for communication between refractory producer and final user. Sometimes differences between the values of properties reported by specialized testing laboratories and those provided by the refractory supplier lead to discussions, or even claims. A greater knowledge on any method's precision will improve current standards and, one would hope, smooth relationships between producers and final users.

For refractory products a key physical property is its permanent linear change (PLC). PLC is defined as the expansion or shrinkage that a refractory material undergoes when it is heated up to a given temperature for a given period of time and then cooled down to room temperature. The ratio between the change in length (final – initial) and the initial one is expressed as a percentage. For dense shaped bricks the testing procedure is described in standard EN 993-10:1997.

Although for PLC there are many possible influencing testing parameters (factors), the objective of this study was to investigate the effect of some of them, considered by our common experience to be most relevant. Those were: length of the test specimens, direction of extraction, method of measurement, and heating schedule (heating rate and soaking time). The study was carried out in two phases. Initially only one laboratory checked the influencing factors, this was followed by a second phase where up to four different laboratories were involved.

2 Experimental procedure

2.1 Materials

Two high alumina dense shaped refractories, specifically provided for the ReStaR project by RHI AG, were used: a 81 mass-% Al_2O_3, of 2,75 g/cm^{-3} bulk density, 20,5 % apparent porosity and 70 MPa cold crushing strength, and a 76 mass-% Al_2O_3 of 2,65 g/cm^{-3} bulk density, 15,5 % apparent porosity and 110 MPa cold crushing strength. Test temperature for both materials was 1400 °C.

Thermal treatment was carried out according to the requirements set in the standard. During soaking the temperature difference in the volume occupied by the samples did not exceed 10 °C.

2.2 Statistical analysis – design of experiments I (DOE I)

A full factorial experimental design, with two levels for each factor, was planned and carried out. The 76 mass-% Al_2O_3 dense refractory was initially tested. Cylindrical test specimens, 50 mm in diameter, were obtained from the geometrical centre of the bricks. To ensure parallelism the upper and lower faces of each cylinder were ground. Tab. 1 shows the factors and levels selected in the experimental design for study.

Measurement method: the method referenced in EN 993-10 describes the use of a dial gauge, with an accuracy of 0,01 mm, mounted in a carrier according to the scheme shown in Fig. 1. This measurement

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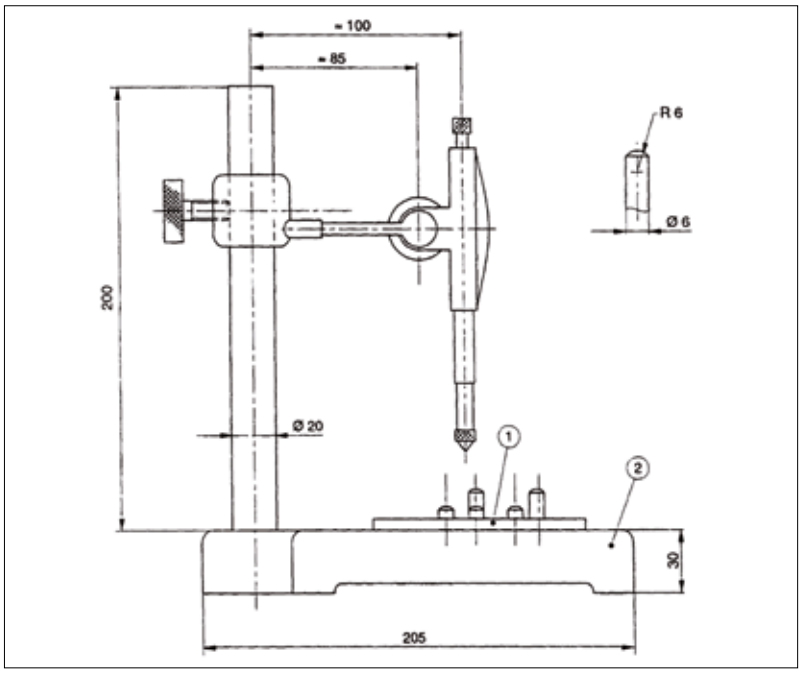


Fig. 1 Scheme of the dial gauge mounted on a carrier according to EN 993-10

Tab. 1 Factors and levels considered in design of experiments I

Factor	Levels	
A – Measurement method	Dial gauge (reference method)	Height gauge resting on a surface plate
B – Heating rate	300 °C h^{-1} up to 1200 °C 120 °C h^{-1} up to 1350 °C 60 °C h^{-1} up to 1400 °C	600 °C h^{-1} up to 1200 °C 300 °C h^{-1} up to 1400 °C
C – Soaking time	5 h	12 h
D – Length of test specimens	60 mm	70 mm
E – Direction of extraction	Parallel to press direction	Perpendicular to press direction

Tab. 2 Factors and levels studied in design of experiments II

Factor	Levels	
	(–)	(+)
A – Measurement method	Dial gauge (reference method)	Calliper
B – Heating rate	300 °C h^{-1} up to 1200 °C 120 °C h^{-1} up to 1350 °C 60 °C h^{-1} up to 1400 °C	600 °C h^{-1} up to 1200 °C 300 °C h^{-1} up to 1400 °C
C – Length of test specimens	60 mm	50 mm
D – Direction of extraction	Parallel to press direction	Perpendicular to press direction
E – Laboratory	Four laboratories	

Tab. 3 Combination of factors tested by each laboratory

A – Measurement Method	B – Heating Rate	C – Length of Test Specimens	D – Direction of Extraction
–	+	–	+
–	+	+	–
–	–	–	–
–	–	+	+
+	–	–	–
+	–	+	+
+	+	–	+
+	+	+	–

method was compared with the use of a height gauge with an accuracy of 0,01 mm supported on a surface plate.

Heating rate: in the standard a wide range of heating rates is allowed. For example for temperatures below 1250 °C heating rates between 300 and 600 °C · h^{-1} are permitted. In the study two levels of this factor were considered: the minimum and maximum heating rate specified by the standard.

Soaking time: two dwelling times were selected: 5 h (the preferred one in the standard) and 12 h, which is specified as optional.

Length of test specimens: the standard length of 60 mm and 70 mm were chosen for study.

Direction of extraction: two extraction directions: perpendicular and parallel to press direction, were studied.

A statistical linear model was followed, including terms up to second order [1, 2]. Since this is a complete factorial design, there is no confusion between the terms of the model.

Tab. 2 shows the influencing factors selected for this study: measurement method, heating rate, length of the test specimens and direction of extraction of the samples. In this occasion the use of a calliper, with an accuracy of 0,02 mm, as measuring device was compared with the reference method. Tab. 3 shows the combination of treatments selected according to a Plackett Burman design [3].

The statistical model used was also linear including only interactions up to second order [1], due to the resolution of the design only the main effects were considered.

2.3 Statistical analysis – design of experiments II (DOE II)

Taking into account the results obtained in the previous DOE, a new factorial design involving four different laboratories was plan. Cylindrical test specimens were extracted from the 81 % Al_2O_3 refractory. Specimens were taken from the geometrical centre and the side of each brick, and prepared as in DOE I. Two test specimens from the side and one from the centre were tested for each combination of factors.

3 Results and discussion

The null hypothesis would be that the factor or combination of factors studied does not

have significant influence in the response. The alternative hypothesis would be the opposite one. If for each contrast the p-value obtained is lower than the significance level then the null hypothesis should not be rejected, and the effect of the factor considered is significantly higher than the random error.

3.1 Design of experiments I (DOE I)

Tab. 4 shows the ANOVA table resulting for DOE I. According to the p-values obtained, and for a significance level of 0,05, three main factors and four second order interactions are to be considered statistically significant. As main factors were found: measurement method, heating rate and direction of extraction of the samples, and as second order interactions: measurement method and heating rate, heating rate and soaking time, heating rate and length of test specimens and heating rate and direction of extraction. Despite the number of factors found to be statistically significant their effect on PLC differs, as it is shown in Fig. 2.

The highest effect on PLC testing results is due to the heating rate (B in Fig. 2). The main effects' plot, Fig. 3, shows that when heating rate is increased from the slowest to the fastest specified in the standard the determined shrinkage increases approximately 0,03 %. Two tests done with this refractory product performed according to the EN 993-10 standard, which would only differ in the heating rate, might lead to a PLC variation of more than 55 %.

The second highest effect was due to the measurement method (A in Fig. 2). Ac-

Tab. 4 ANOVA table and effect of every term of the statistical model for DOE I (DF freedom degrees, SC sum of squares, CM medium squares)

Factor	DF	SC	CM	F-Value	p-Value	Effect
A	1	0,009474	0,009474	8,80	0,004	–0,01721
B	1	0,031529	0,031529	29,28	0,000	–0,03139
C	1	0,000504	0,000504	0,47	0,495	0,00397
D	1	0,001139	0,001139	1,06	0,306	0,00597
E	1	0,006303	0,006303	5,85	0,017	–0,01403
AB	1	0,002899	0,002899	2,69	0,104	–0,00952
AC	1	0,005829	0,005829	5,41	0,022	–0,01350
AD	1	0,001825	0,001825	1,70	0,196	–0,00755
AE	1	0,003642	0,003642	3,38	0,069	–0,01067
BC	1	0,007067	0,007067	6,56	0,012	0,01486
BD	1	0,004407	0,004407	4,09	0,045	–0,01174
BE	1	0,008420	0,008420	7,82	0,006	0,01622
CD	1	0,001916	0,001916	1,78	0,185	0,00774
CE	1	0,000544	0,000544	0,51	0,479	0,00412
DE	1	0,001726	0,001726	1,60	0,208	0,00734
Error	112	0,120582	0,001077			
Total	127	0,207806				

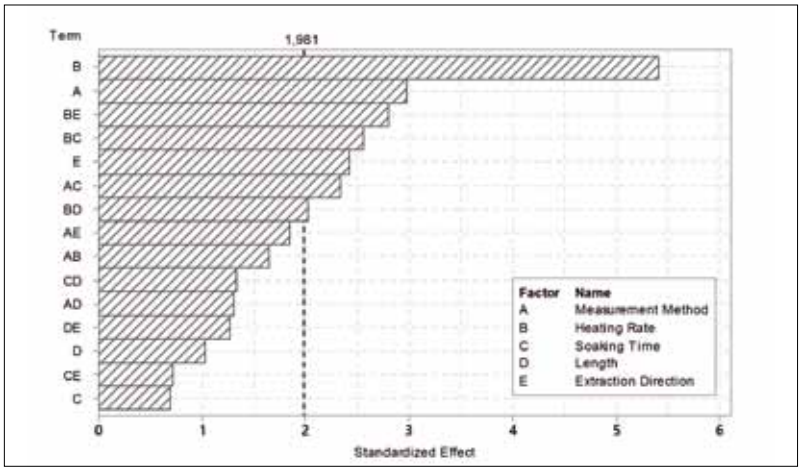


Fig. 2 Pareto's chart for DOE I (α = 0,05)

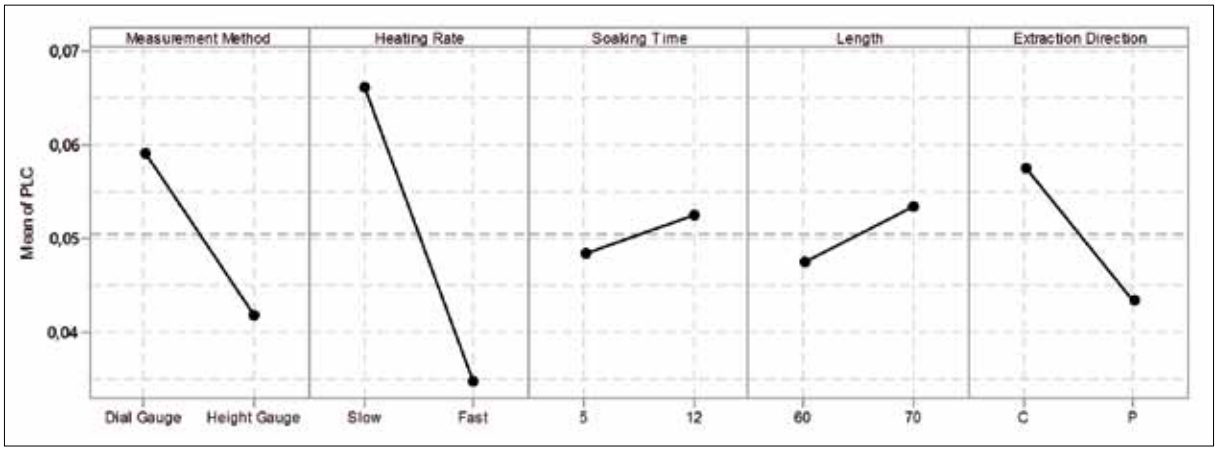


Fig. 3 Main effects' plot for DOE I

Tab. 5 ANOVA table for DOE II

Factor	DF	SC	MS	F-Value	p-Value
A	1	0,0020	0,00196	0,05	0,832
B	1	0,1919	0,19193	4,43	0,036
C	1	0,0213	0,02132	0,49	0,483
D	1	0,0138	0,01382	0,32	0,572
E	3	3,0533	1,01777	23,50	0,000
Error	375	16,2405	0,04331		
Total	382	19,5233			

cording to the results obtained the use of a height gauge resting on a surface plate instead of the device described in the standard results also in an increase of about 0,017 % in the shrinkage measured.

The effect of the interaction between the heating rate and the direction of extraction was also found statistically significant (BE in Fig. 2). Nevertheless its effect intermediate between the heating rate (B) and the direction of extraction (E) suggests that this effect is may be due to the aforementioned high impact that heating rate has on the PLC.

Moreover the effect of E, although statistically significant, is not very important from a practical point of view. A similar consideration can be done with the effect of the interaction between the heating rate and the soaking time (BC in Fig. 2).

3.2 Design of experiments II (DOE II)

The second phase of the investigation on PLC influencing factors involved four laboratories. In order to increase the response signal an 81 mass-% Al_2O_3 content dense refractory was tested at 1400 °C. Considering the results obtained in the previous experimental step the factor "soaking time" was removed from the design, and different levels of the measurement method and length of test specimens were chosen (see Tab. 2). The low level of the factor length was chosen as 50 mm. The advantage would be that, if a 50 mm length was to be adopted in a new standard, the same test specimen could be used for bulk density, cold crushing strength and permanent linear change measurement.

Tab. 5 shows the ANOVA table results. The p-values obtained for each factor show, for a significance level of 0,05, that the effect of the heating rate and the laboratory performing test are statistical significant. The main effect's plot (Fig. 4) shows the relative effect of each factor in PLC testing results. The most important differences are observed among laboratories: laboratories number 1 and 3 show mean values afar from the general mean value. As for the rest of factors considered, heating rate is the second in importance. Increasing heating rate from the minimum to the maximum value resulted in a measured expansion increment of about 0,045 %.

Taking into account these results, an ANOVA analysis was carried out for each laboratory independently. Tab. 6 and Fig. 5 show respectively the resulting ANOVA's tables and the Pareto's charts. According to the latter the measurement method was found to be a significant factor for three laboratories, heating rate was also significant in three of them and the length of test specimens only in one.

For each laboratory the main effect's plot (Fig. 6) reveals that, for those laboratories where the heating rate was found to be significant, an increased heating rate leads to an increase in the determined value for permanent linear change, too. Nevertheless this fact is not observed for the factor "measurement method". For two laboratories the use of a calliper, instead of the standard carrier, leads to an increase in measured PLC, while for the third laboratory the opposite is observed. The direction of extraction of the samples did not have any significant impact on the permanent linear change measured by the laboratories.

4 Conclusions

The results obtained in DOE, involving only one laboratory, found heating rate and measurement method to be the most relevant influencing factors on PLC measurement results. The change from the low level to the high level of the heating rate resulted in an increased value for shrinkage of approximately 0,03 %. On the other hand, when a height gauge with an accuracy of 0,01 mm was used instead of the device described in the standard, the measured shrinkage increased by 0,017 %.

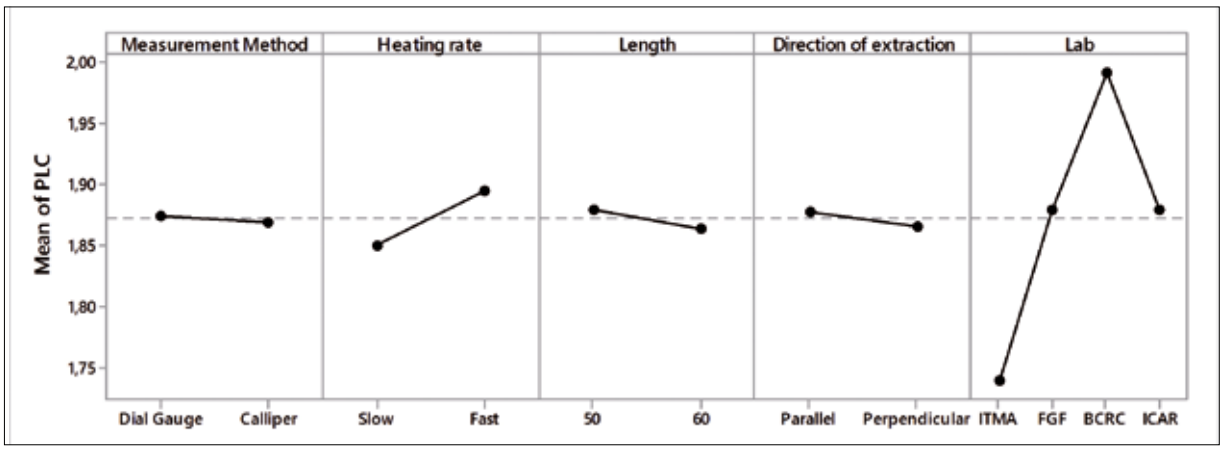
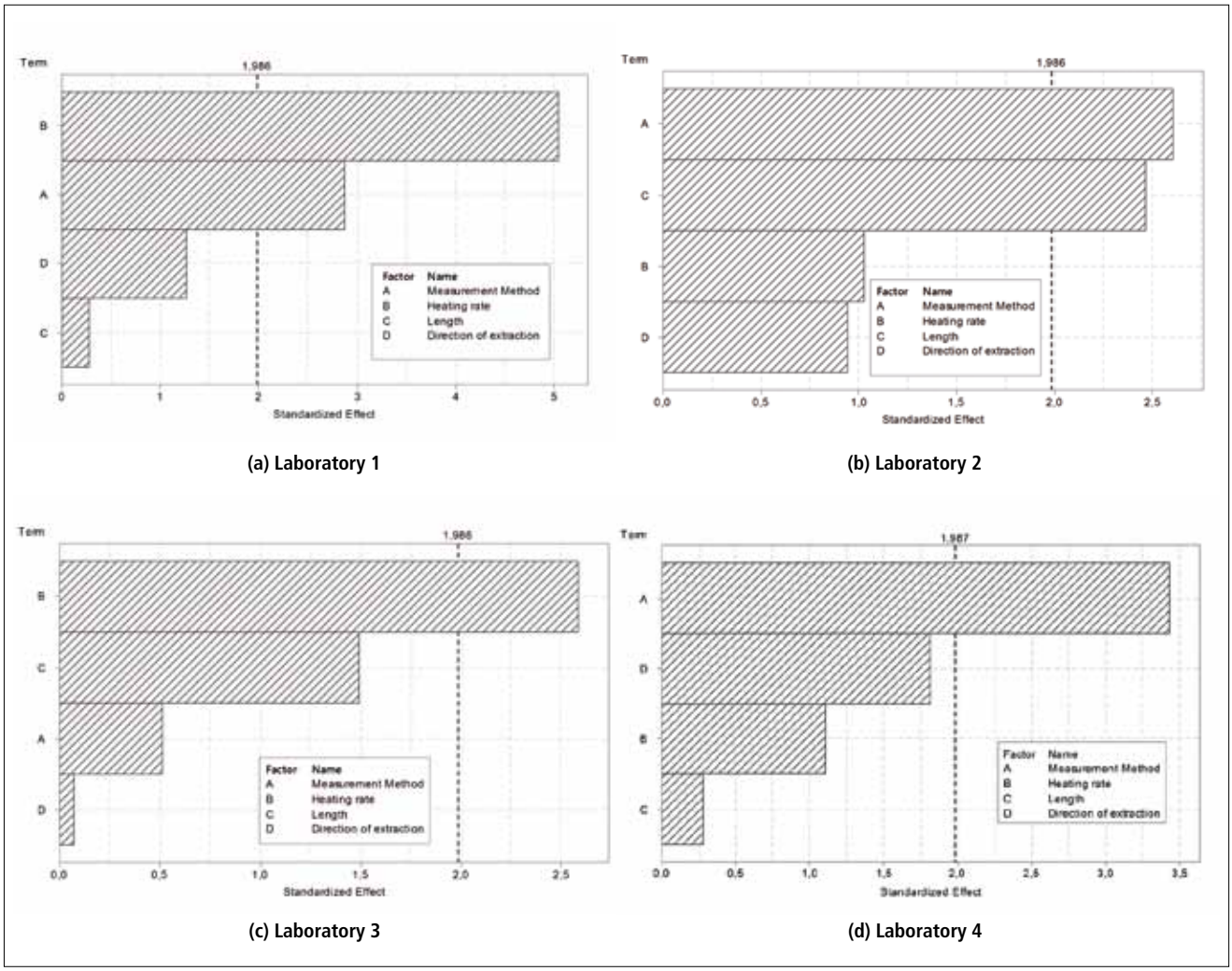


Fig. 4 Main effect's plot for PLC in DOE II

Tab. 6 ANOVA table for each laboratory

LABORATORY 1						LABORATORY 2				
Factor	**DF**	**SC**	**MS**	**F-Value**	**p-Value**	**DF**	**SC**	**MS**	**F-Value**	**p-Value**
A	1	0,13088	0,130884	8,23	0,005	1	0,47264	0,47264	6,77	0,011
B	1	0,40372	0,403717	25,39	0,000	1	0,07348	0,07348	1,05	0,307
C	1	0,00123	0,001231	0,08	0,781	1	0,42427	0,42427	6,08	0,016
D	1	0,02524	0,025238	1,59	0,211	1	0,06171	0,06171	0,88	0,349
Error	91	1,44714	0,015903			91	6,34870	0,06977		
Total	95	2,00821				95	7,38080			
LABORATORY 3						**LABORATORY 4**				
Factor	**DF**	**SC**	**MS**	**F-Value**	**p-Value**	**DF**	**SC**	**MS**	**F-Value**	**p-Value**
A	1	0,01537	0,015366	0,26	0,611	1	0,12817	0,128171	11,80	0,001
B	1	0,39494	0,394940	6,68	0,011	1	0,01341	0,013414	1,23	0,269
C	1	0,13213	0,132132	2,24	0,138	1	0,03599	0,035992	3,31	0,072
D	1	0,00032	0,000320	0,01	0,942	1	0,00086	0,000860	0,08	0,779
Error	91	5,37981	0,059119			90	0,97787	0,010865		
Total	95	5,92257				94	1,15820			



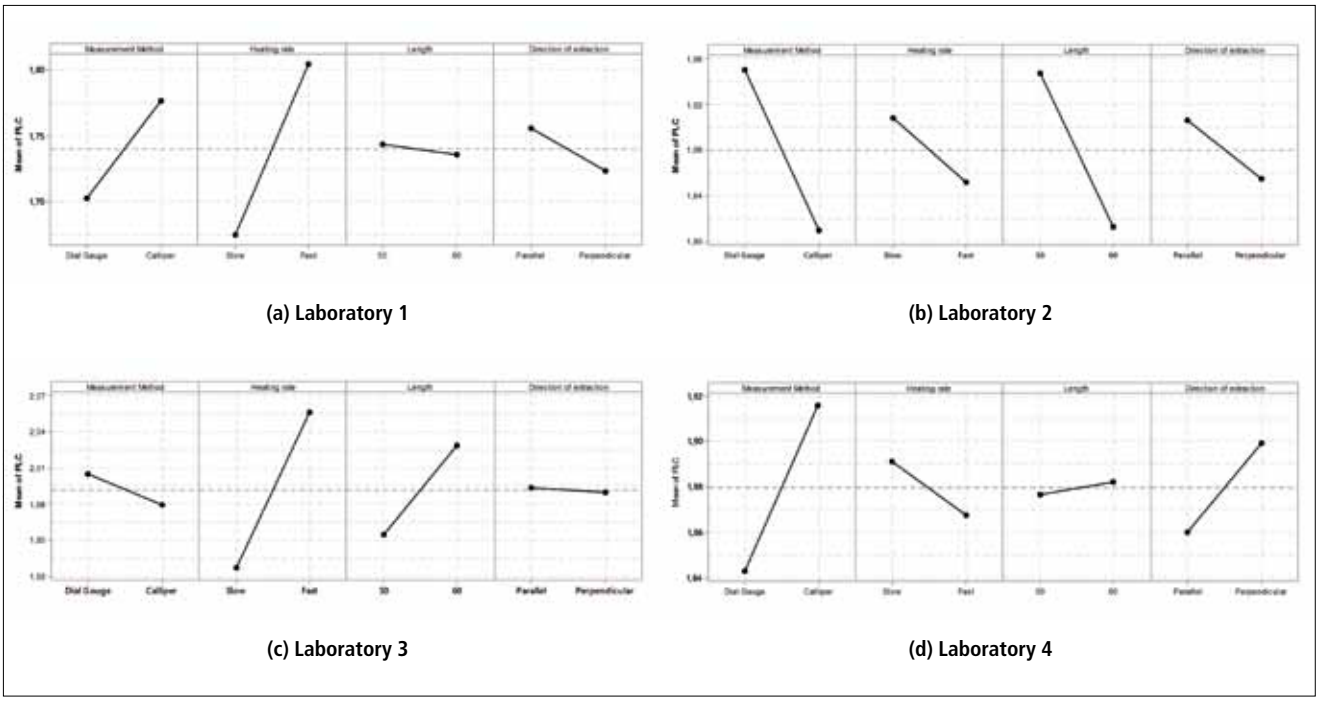
(a) Laboratory 1

(b) Laboratory 2

(c) Laboratory 3

(d) Laboratory 4

Fig. 5 Pareto's chart for each laboratory (α = 0,05)



(a) Laboratory 1

(b) Laboratory 2

(c) Laboratory 3

(d) Laboratory 4

Fig. 6 Main effect's plot for each laboratory involved in DOE II

The results obtained in DOE II, involving four different laboratories, revealed that the use of a calliper with an accuracy of 0,02 mm instead of the dial gauge described in standard, led to statistically different PLC measurement results for three laboratories. Heating rate was found to be a significant factor for two laboratories. Use of cylindrical test specimens of length of 50 mm instead of 60 mm had a significant impact on PLC measurements only for one of the participant laboratories.

According to these results a more restrictive heating rate should be established in the standard. Any modification of the reference method of measurement should be also carefully considered due to its impact on PLC measurements.

On the other hand the direction of extraction, at least for shaped dense refractory products as the ones tested in this work, has a very limited impact on PLC measurement results.

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References

[1] Montgomery, D.C.; Runger, G.C.: Probabilidad y estadística aplicadas a la ingeniería. 1st ed., Mexico 2005

[2] Montgomery, D.C.: Design and analysis of experiments. 6th ed., New York 2004

[3] Plackett, R.L.; Burman, J.P.: The design of optimum multifactorial experiments. Biometrika **34** (1946) 255–272