Study of Linear Shrinkage for Siliceous Materials to Meet Insulating Requirements

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Abstract

Iraqi siliceous rocks were chosen to be used as raw materials in this study which is concern with the linear shrinkage and their related parameters. They are porcelinite from Safra area (western desert) and Kaolin Duekla, their powders were mixed in certain percentage, to shape compacts and sintered. The study followed with thermal and chemical treatments, which are calcination and acid washing. The effects on final compact properties such as linear shrinkage were studied. Linear shrinkage was calculated for sintered compacts to study the effects of calcination processes, chemical washing, weight percentage, sintering processes, loading moment were studied on this property where the compacts for groups is insulating materials.

Linear firing shrinkage test is very important in ceramic industries, when in some uses most be calculated the ceramic body volume before product.

The results indicate that some of these groups satisfy ability the required linear firing shrinkage property for electrical insulator, so they are an ability to be used in industry.

Key words

Linear shrinkage

Porcelinite, Kaolin

X-Ray diffraction

Article info

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دراسة التقلص الخطي للمواد السيليسية المحققة للمواصفة العازلية

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الخلاصة

يتضمن موضوع البحث اختيار نوع من الصخور السيليسية العراقية كمواد أولية وهي صخور البورسيلينايت من موقع الصفرة في الصحراء الغربية والكاؤولين العراقي (سيليكات) / دويخلة وخلط مساحيقهما بنسب وزنية معينة لتشكيل مكبوسات وتلبيدها ودراسة خواص التقلص الخطي وتأثير بعض العوامل المرتبطة بالعينات المشكلة من هذه المواد.

تم إجراء معاملات حرارية (كلسنة) ومعاملات كيميائية (غسل بالحامض) للمواد الأولية ودراسة تأثيرها على الخواص النهائية للمكبوسات (التقلص الخطي). تم حساب التقلص الخطي للمكبوسات الملبدة والمحضرة من هذه الخلطات بهدف معاينة تأثير؛ عمليات الكلسنة والغسل الكيميائي وإضافة الكاؤولين وعمليات التلبيد ومقدار التحميل على تلك الخواص. علما أن هذه المكبوسات هي مادة عازلة أي أنها ناجحة في الفحص الحراري والكهربائي ويعد فحص التقلص الخطي وقياسه ذا أهمية بالغة في الصناعات السيراميكية حيث يتوجب في بعض الاستخدامات المعتمدة في الصناعات

السيرامكية تحديد حجم الجسم السيراميكي المنتج وأبعاده قبل عملية تصنيعه، وبذلك إن النتائج تشير إلى إمكانية تشكيل نماذج صناعية من بعض الخلطات المختارة بالمواصفة المطلوبة في صناعة العوازل.

Introduction

Linear shrinkage, due to firing, is defined as the percentage of decrease in ceramic sample length and it is of great importance in ceramic industry. In some applications, length is required to determine volume of produced ceramic sample and its dimensions before formating process.

The linear shrinkage is due to variations in the size and shape of the sample particles. Linear shrinkage is approximately proportional to the inverse of the particle radius but is not greatly affected by sintering time [1]. Size distribution is important in practical systems where shrinkage is to be minimized. Mostly large particles and sufficiently small particles to fill in the interstices will give a particle compact of highest green density. The linear firing shrinkage also varies with composition and tends to increase as the fluxing components increase with the production of relatively phases during the firing [2-3].

During the firing process this porosity is reduced; the volume shrinkage due to firing is equal to the pore volume eliminated. This firing shrinkage can be substantially decreased by addition of non-shrinkage material to the mix [4].

The principal causes of the permanent changes in volume are: The nature and composition of the material, it's previous treatment, the sizes and grading of the grains, the pressure applied in shaping the articles, the proportion of water used in mixing the materials, the porosity in the material and the temperature at which the material has been fired or reheated.

Nature of materials is important to determine shrinkage, for example, using clay leads to high shrinkage while in case of silica, shrinkage is lower. As well thermal treatment previous to firing decreases shrinkage. Whilst increasing grain size affects shrinkage since pores density is increased. Presence of gradient of grains means filling of pores and presenting a high-density low- shrinkage sample.

Water amount in mixing process determines porosity and hence shrinkage. Also, applied pressure acts an important role to obtain higher density, lower porosity and then lower shrinkage. Sintering temperature controls shrinkage since it's increasing results more molten, hence high shrinkage in material.

Length of produced samples were measured before and after sintering and linear shrinkage was computed as:

Linear shrinkage (%) = ((Lo – L)/ Lo) ×100 (1)

Experimental Work

The rock samples were crushed by mortar then grinded by ball mill for seven hours to obtain particle size ≤50 micron. After that the powder was calcined at different temperatures (shown in table 1) to form homogeneous powder and have maximum density is obtained by calcination process.

The powder was washed by HCL acid (10 normality) to removal free iron oxide from clays, which is regarded as impurities with respect to the goal of present work.

At mixing process, we used wet mixing method and addition 'Poly Viny al-Cohol' as a binder material at 60 ^{o}C. The samples was formed by was used "semi dry pressing" method at moisture content 10%, then the dried by oven at 60 ^{o}C overnight drying, then drying overnight at 100 ^{o}C, then the samples was left in kiln to sinter at different temperature and measure the linear shrinkage for all samples using vernier caliper and application the equation (1).The results are presented in table (1) and (2).

Table(1): Preparation conditions for Powders

Powder no.	Material	Calcination conditions		Chemical treatment		Acid Normality	Particle size
		Temp. °C	Time (hr)	Medium	Time (hr)		
No.1	Porcelinite	200	2	HCL	24	10	≤ 50
No.2	Porcelinite	200	2	HCL	48	10	≤ 50
No.3	Porcelinite	900	2	HCL	12	10	≤ 50
No.4	Kaolin	400	7	HCL	1	10	≤ 50
M1	Porcelinite	-	-	-	-	-	≤ 50
M2	Porcelinite	200	2	-	-	-	≤ 50

Table (2): Pressing and Sintering conditions for groups

Group No.	Composition		Sintering condition			Loading		Binder		Moisture Percentage
	Wt %	Batch No.	Sintering Temp. °K	Sintering time (hr.)	Sintering rate °K/hr	Load (MPa)	Time (sec)	Type	Wt %	
M1	100	Porcelinite 100%	1473 1573 1673	2 2 2	100 100 100	24.97 79.94 7491 = = = = = =	60 = =	**PVA**	1	10
M2	100	Porcelinite 100%	1473 1573 1673	2 2 2	100 100 100	= = = = = = = = =	= = =	**PVA**	1	10
M3	70 30	No1. No.4	1473 1573 1673	2 2 2	100 100 100	= = = = = = = = =	= = =	**PVA**	1	10
M4	70 30	No.2 No.4	1473 1573 1673	2 2 2	100 100 100	= = = = = = = = =	= = =	**PVA**	1	10
M5	70 30	No.3 No.4	1473 1573 1673	2 2 2	100 100 100	= = = = = = = = =	= = =	**PVA**	1	10

Results

Figures (1–5) explain the relation of linear shrinkage to sintering temperature for different loading. It's clear from figures that linear shrinkage increases as sintering temperature increase, while it is lower in samples sintered at 1573˚K for all groups. Shrinkage decreases with the increasing of loading.

In engineering application, known range of linear shrinkage is about 12-15 % and this range was satisfied for all groups except group M1 containing samples sintered at 1673˚K. Experimentally, it was found that linear shrinkage of samples of groups M1 and M5 (sintered at 1673˚K) increases extremely relative to those sintered at 1473˚K and 1573˚K.

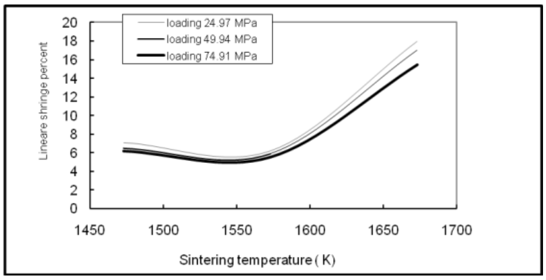

Fig. (1): Linear shrinkage for group M1

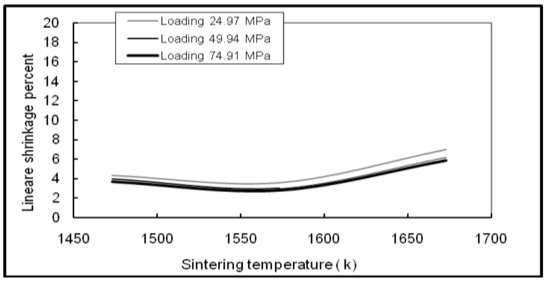

Fig. (2): Linear shrinkage for group M2

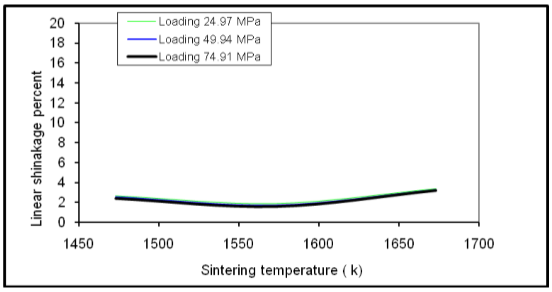

Fig. (3): Linear shrinkage for group M3

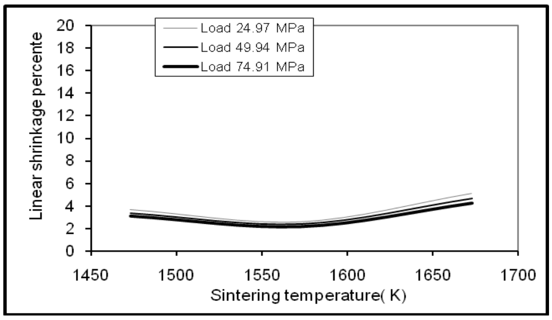

Fig. (4): Linear shrinkage for group M4

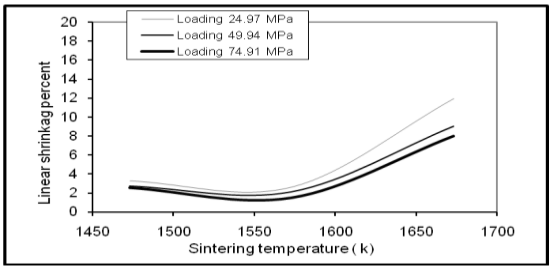

Fig. (5): Linear shrinkage for group M5

Discussion

It could be seen as the vacancies and pores will be filled by molten (glassy phase) [5]. During sintering process, porosity ceramic body is reduced. So, shrinkage size is equal to size of pore removed or lost. Hence, shrinkage could be reduced by increasing loading applied to shape sample [4]. Sintering temperature has an extreme effect on the value of linear shrinkage as increasing sintering temperature leads to increases linear shrinkage due to increasing amount of molten filling pores and product high shrinkage in the sample [5]. But it is seen that linear shrinkage of samples sintered at 1573˚K is lower than that sintered at 1473˚K.

This is attributed to formation of cristobalite phase and little amount of Mullite phase at 1473˚K as shown in figures (6-8). While at 1473˚K as shown in figures (6-8), both phases begin to stabilize and occupy larger size leading to extend grains of these phases and hence larger size sample. Continuing to increase sintering temperature to 1673˚K, sintering process proceeds more and more resulting lower porosity and hence higher shrinkage (90).

Figures (6–8) explain the presence of cristobalite and Mullite phases in samples A, B and C sintered at 1473˚K, 1573˚K and 1673˚K respectively. All samples are of group M2. Figures (1) and (2) explain that shrinkage of groups M2 ranges at between (2.7-7) %, while that of group M1 ranges at (5-17) %.

The reason is the thermal treatment (calcination) of group M2 which lead to reduce porosity and increase powder density and hence reduce shrinkage of powder grains. Shrinkage of groups M3 and M4 is ranging between (2-4.5)%, i.e., lower than that of group M2 which is attributed to addition of kaolin having melting temperature higher than that of Porcelinite, then the molten portion is little. In group M5, shrinkage is ranging between (1.5-12) % except samples sintered at 1573˚K due to increasing of molten.

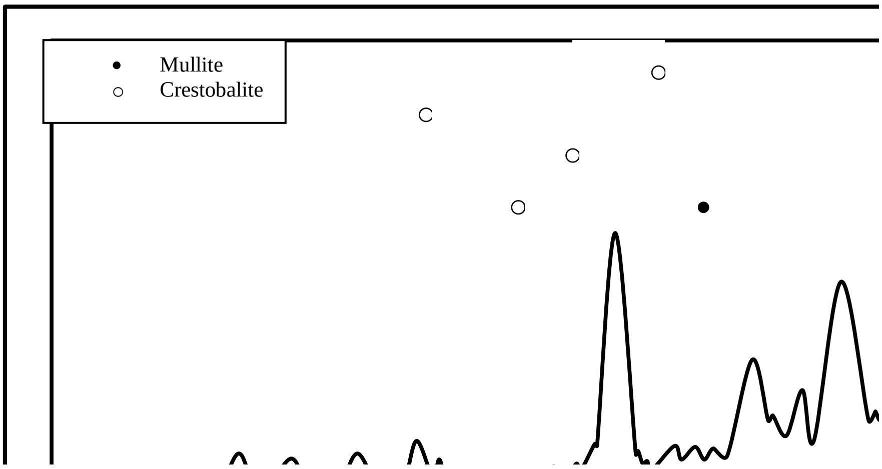

Fig. (6): X-ray diffraction for sample A.

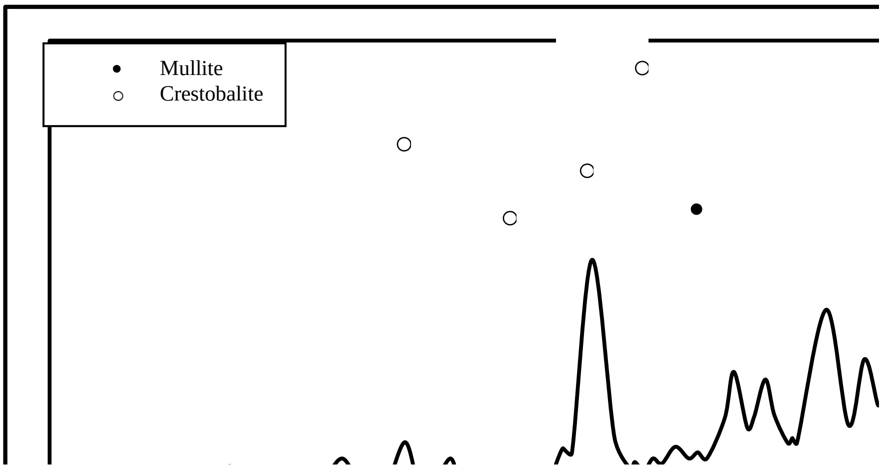

Fig. (6): X-ray diffraction for sample B.

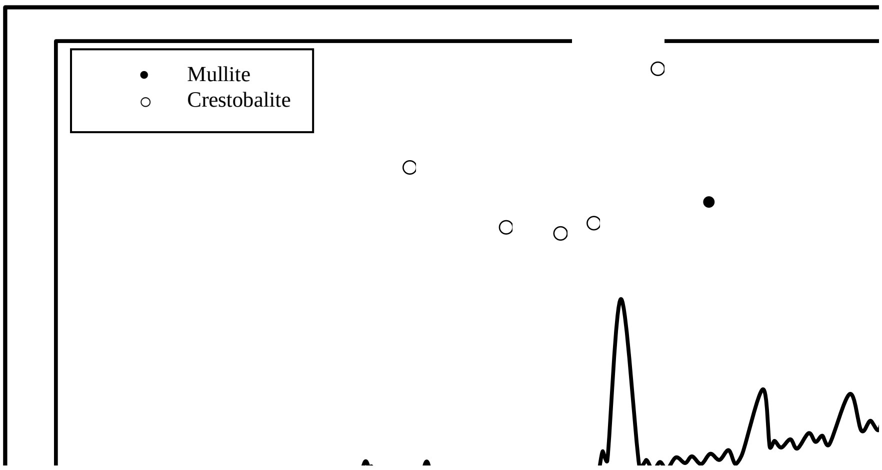

Fig. (6): X-ray diffraction for sample C.

Conclusions

From the result we could conclude the following: -

1. The increased of applied loading lead to reduction of linear shrinkage since increasing of loading would reduce distances among powder particles and

then form a compacted body having high density.

2. Sintering temperature has an extreme effect on the value of linear shrinkage as increasing sintering temperature lead to increase of linear shrinkage due to increasing amount of melt filling pores.

3. The formation of cristobalite phase and little amount of mullite phase in porcelinite material at 1573˚K, which occupy larger size and extend grains, hence larger size for samples.

4. Calcination process effect on linear shrinkage as increasing calcination temperature lead to increasing linear shrinkage due to increasing density of powder after removal of gases and organic materials.

5. Linear shrinkage decreases due to chemical treatment by hydrochloric acid.

6. The addition of kaolin material to porcelinite leads to decrease of linear shrinkage.

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