Institut National Interuniversitaire des Silicates, Sols et Matériaux
Laboratoire de Recherches et d'Essais
Association sans but lucratif

bcrcinisma

Avenue Gouverneur Cornez, 4
B-7000 MONS (Belgique)
Tél. (065)40.34.34
Fax (065)34.80.05
T.V.A. : BE 413.106.271
RC MONS : 130.828 - Enregistrement : 08/02/01

Laboratoire agréé : Voir circulaire 514-B/33 du 09/02/87 du M.T.P.
et communiqués 514-B/33/1 et 2 du M.C.I.

Mons, le 3 Septembre 2004

RAPPORT D'ESSAI N° 94.220

Comprenant pages

Feuille n° 1

DEMANDE PAR : Dr G.N. Georgiadès
Industrial & Commercial Enterprises
Thessaloniki, Greece

REFERENCE DE LA DEMANDE :

DESCRIPTION DE L'ECHANTILLON : Clayey diatomite

NOMBRE ET IDENTIFICATION DES ECHANTILLONS :

OBJET DE LA DEMANDE :

Feasibility study on clayey diatomite « moler type » for a classical ceramic manufacturing route

ECHANTILLON RECU LE : April 2004

DATE DES ESSAIS : April to July

OBSERVATIONS SPECIALES : Néant.

Aim : Feasibility study on the utilization of clayey diatomite « moler type » originated from central Greece for the production of lightweight fired construction blocks.

---

1. Materials

The aim is to produce lightweight fired construction blocks with similar mechanical and thermal properties as the Ytong blocks (see table hereafter)

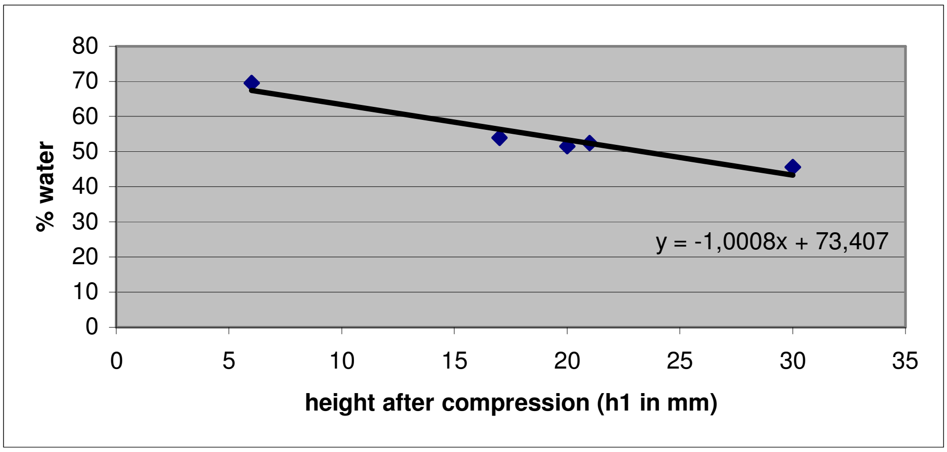
Classification	C2/04	C3/05	C4/06	C5/07
Apparent density (kg/m^3)	$300 \leq \rho < 400$	$400 \leq \rho < 500$	$500 \leq \rho < 600$	$600 \leq \rho < 700$
Compression strength (MPa)	$f_{bk} \geq 2$	$f_{bk} \geq 3$	$f_{bk} \geq 4$	$f_{bk} \geq 5$

The raw material is a clayey diatomite « moler type » from Central Greece . The clayey diatomite is used without any additives and a classical manufacturing route is followed : blending – extrusion – drying – firing.
The working program was as follows :

- ✓ Plasticity test according to Pfefferkorn
- ✓ Extrusion of 70 parallelepipedons with rectangular section (25 x 30 mm)
- ✓ Controlled drying of 5 parallelepipedons in climatic chamber
- ✓ Uncontrolled drying of the other parallelepipedons in ambient conditions with recording of weight loss and shrinkage
- ✓ Firing of 3 series of 20 samples each at 3 temperatures : 600°C, 700°C and 800°C in electrical kiln (air atmosphere) with recording of weight loss and shrinkage
- ✓ Hydrostatic measurement of apparent density (3 x 5 samples)
- ✓ Measurement of water sorption by immersion (3 x 5 samples)
- ✓ Measurement of compression strength according to 2 directions (5 samples // and 5 samples ⊥ to extrusion direction) including rectification of 3 x 10 samples.

2. Plasticity test according to Pfefferkorn

According to Pfefferkorn, the plasticity of a clay is deduced from the compression ratio “a” ($a=h_0/h_1$, with h_1 being the height after compression, and h_0 the initial height 40 mm) of a cylindrical sample under well defined conditions. The samples are prepared according to various water contents. When the height loss of the sample is 70% of the initial height, the water content is considered as the right one for a good plasticity.



The correct plasticity index is calculated for h1 equal to 12 (a= 3.3). On figure 1, we can see that H_1 = 12 corresponds to a % water of 61.4%. The workability index is calculated for a h_1 = 16 (a= 2.5) as a good workability must be 2.5 < a < 4) which corresponds to a % water of 57.4%. In our experimental conditions, a % water of 74.8% was used for the extrusion of the parallelepipedons.

3. Controlled drying in climatic chamber

✓ From the 70 extruded parallelepipedons, 5 have been dried in a climatic chamber, with recording of weight loss and shrinkage, to characterize the resistance to cracking.

Samples	**Weight on wet samples (g)**	**Weight of dry samples (g)**	**weight loss (%)**	**water content (%)**
1	110.71	66.03	40.36	67.6
2	107.00	63.72	40.45	67.9
3	103.21	61.67	40.15	67.3
4	113.47	67.58	40.44	67.9
5	109.50	65.46	40.22	67.3

Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	81	75	7.4
2	80	74	7.5
3	75	70	6.66
4	80	74	7.5
5	80	74	7.5

We can see from these results that the weight loss and the shrinkage are homogeneous among the 5 samples. The water content, lost during drying, is near to the water added to the samples for extrusion. The shrinkage seems quite normal for a clayey sample.

✓ Eleven other samples have been dried in ambient air, with recording of weight loss and shrinkage, also to characterize the resistance to cracking.

Samples	Weight of wet samples (g)	Weight of dry samples (g)	Weight loss (%)	Water content (%)
1	107.00	63.90	40.28	67.4
2	111.67	66.66	40.31	67.5
3	110.05	65.98	40.05	66.8
4	110.99	66.52	40.07	66.8
5	105.10	62.99	40.07	66.8
6	109.38	65.31	40.29	67.5
7	100.05	59.97	40.06	66.8
8	107.41	63.65	40.74	67.4
9	105.69	63.32	40.59	66.9
10	102.75	62.35	39.32	64.8
11	106.48	67.57	36.54	57.6

Samples	Initial length (mm)	Final length (mm)	shrinkage(%)
1	81	73.5	9.25
2	80	74	7.5
3	79	72	8.86
4	81.5	74	8.64
5	77	70	9.09
6	79	72	8.86
7	72	66	8.33
8	80	72.5	9.3
9	78	70	10.25
10	75	70	6.66
11	78	72	7.69

We can see that when letting the samples dry in ambient air, the water loss corresponds to the water content of the samples. The shrinkage seems to vary between 7 and 10 linear %.

4. Firing

6 series of samples have been fired each at 6 temperatures (600, 700, 800, 850, 900 and 950℃) in electrical kiln (air atmosphere) with recording of weight loss and shrinkage

The firing cycle is as follows :

Temperature steps	Speed
20 to 600°C	150°C / h
600°C to T°C	200°C / h
stage at T°C	3 h
T°C to 600°C	200°C / h
600 to 500°C	50°C / h
500 to 20°C	free

Recording of the weight loss

			Firing at 600°C
Samples	**initial weight (g)**	**Weight after firing (g)**	**Weight loss (%)**
1	65.08	58.40	11.44
2	66.93	60.06	11.44
3	65.44	58.67	11.54
4	69.71	62.44	11.64
5	69.01	61.83	11.61
6	65.39	58.58	11.62
7	65.95	58.98	11.81
8	69.10	61.83	11.75
9	66.42	59.22	12.15
10	65.59	58.74	11.66

Firing at 700°C			
1	61.95	55.11	12.41
2	66.59	59.22	12.44
3	67.84	60.43	12.26
4	62.70	55.83	12.30
5	69.16	61.49	12.47
6	65.36	58.10	12.49
7	67.33	59.83	12.53
8	70.39	62.76	12.16
9	63.18	56.17	12.47
10	63.08	56.23	12.18

Firing at 800°C			
1	66.40	59.31	11.95
2	64.86	57.93	11.96
3	67.64	60.37	12.04
4	62.47	55.77	12.01
5	67.53	60.30	11.99
6	67.23	60.03	11.99
7	67.99	60.67	12.06
8	68.95	61.55	12.02
9	68.09	60.79	12.01
10	68.49	61.14	12.02

A mean weight loss of 11.67, 12.37 and 12.01% is observed after firing at 600, 700 and 800°C, respectively.

Recording of the shrinkage

Firing at 600°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**Shrinkage (%)**
1	71	71	0
2	70	70	0
3	71	71	0
4	74	74	0
5	75	75	0
6	70	70	0
7	68	68	0
8	74	74	0
9	71	71	0
10	69	69	0

Firing at 700°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	67	67	0
2	71	71	0
3	73	73	0
4	67	67	0
5	74	74	0
6	70	70	0
7	73	73	0
8	73.5	73.5	0
9	67.5	67.5	0
10	68.5	68.5	0

Firing at 800°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	71	71	0
2	71	70	1.4
3	74	73	1.4
4	67	67	0
5	70	69	1.4
6	70	70	0
7	72	71	1.4
8	72	71	1.4
9	74	73	1.4
10	72	72	0

Firing at 850°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	73	72	1.4
2	75	72	4.0
3	70	69	1.4
4	72	70	2.7
5	70	69	1.4
6	72	70	2.7

Firing at 900°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	69	65	5.8
2	72	68	5.5
3	70	67	4.3
4	71	69	2.8
5	75	71	5.3
6	75	71	5.3

Firing at 950°C			
Samples	**Initial length (mm)**	**Final length (mm)**	**shrinkage (%)**
1	74	68	8.1
2	74	68	8.1
3	66	61	7.6
4	72	67	6.9
5	73	68	6.8
6	70	65	7.1

No shrinkage is observed when firing at 600 and 700°C, and a small shrinkage up to 1.4 % is observed when firing at 800°C.

From 850°C, the shrinkage increases and is within 1.4 and 8 % of the initial length. At 900°C, the shrinkage is quite acceptable.

5. Hydrostatic measurement of densities and porosity

Hydrostatic measurements of bulk and apparent densities, and open porosity have been carried out after full vacuum on 3 series of samples fired at 600, 700 and 800°C.

Firing at 600°C			
Samples	**bulk density (g/cc)**	**open porosity (%vol)**	**apparent density (g/cc)**
1	1.195	51.23	2.450
2	1.205	51.33	2.476
3	1.187	51.96	2.471
4	1.193	50.96	2.432
5	1.208	51.28	2.479
Mean values	***1.198***	***51.35***	***2.461***

Firing at 700°C			
1	1.177	51.67	2.435
2	1.190	51.27	2.441
3	1.183	51.60	2.445
4	1.188	51.68	2.458
5	1.197	51.62	2.475
Mean values	***1.187***	***51.57***	***2.451***

Firing at 800°C			
1	1.189	51.25	2.438
2	1.185	51.25	2.430
3	1.183	51.20	2.423
4	1.202	51.36	2.470
5	1.183	51.66	2.447
Mean values	***1.188***	***51.34***	***2.442***

Firing at 850°C			
1	1.21	51.41	2.50
2	1.21	51.46	2.50
3	1.22	51.36	2.50
4	1.21	51.46	2.49
5	1.21	51.38	2.49
6	1.21	51.65	2.49
Mean values	***1.21***	***51.45***	***2.50***

Firing at 900°C			
1	1.30	48.22	2.50
2	1.31	47.71	2.51
3	1.30	48.29	2.51
4	1.31	47.68	2.51
5	1.26	49.78	2.50
6	1.26	49.89	2.50
Mean values	***1.29***	***48.60***	***2.51***

Firing at 950°C			
1	1.44	43.17	2.53
2	1.45	42.71	2.52
3	1.45	42.71	2.53
4	1.45	42.62	2.52
5	1.40	44.59	2.52
6	1.40	44.60	2.52
Mean values	***1.43***	***43.4***	***2.52***

After firing at 600, 700 and 800°C, no important difference is observed nor in the density values nor in porosity. The porosity has been measured as being about 51%.

When firing above 800°C, density and porosity values start to increase and especially at 900 and 950°C. Porosity at 950 °C becomes to be less interesting from a insulating point of view.

6. Measurement of water sorption by immersion

Samples	water sorption (%)		
	after firing at 600 °C	after firing at 700 °C	after firing at 800 °C
1	41.98	41.21	42.59
2	41.03	41.65	42.13
3	40.24	43.95	43.11
4	41.92	42.50	41.50
5	40.91	41.95	42.23
Mean values	***41.22***	***42.25***	***42.31***

Samples	water sorption (%)		
	after firing at 850 °C	after firing at 900 °C	after firing at 950 °C
1	40.90	34.89	27.54
2	40.48	34.16	27.19
3	40.04	35.57	27.12
4	41.22	34.28	27.22
5	41.01	29.26	28.78
6	41.57	37.50	29.17
Mean values	***40.87***	***34.28***	***27.84***

The mean water sorption at 600, 700, 800 and 850 °C is 41.22, 42.25, 42.31 and 40.87% after 48 hr immersion. It starts to decrease significantly when firing at 900 and 950 °C.

7. Compression strength

The compression strength has been measured on 19.3mm diameter cylinders in the extrusion direction (E) and perpendicular to it (T).

Samples	Compression strength (N/mm^2) at 600°C	
	E direction	**T direction**
1	12.6	-
2	12.2	-
3	12.4	-
4	14.7	-
5	14.7	14.5
6	11.9	-
Mean values	***13.1***	

Remark : many ruptures have been observed during boring and machining in this series.

Samples	Compression strength (N/mm^2) at 700°C	
	E direction	**T direction**
1	18.3	18.6
2	12.6	19.3
3	14.0	19.2
4	17.3	18.1
5	17.0	16.1
6	12.3	16.8
7	13.8	-
8	19.8	16.1
Mean values	***15.6***	***17.7***

Samples	Compression strength (N/mm²) at 800°C	
	E direction	**T direction**
1	12.8	15.0
2	13.6	14.2
3	16.3	11.8
4	14.2	-
5	14.4	-
6	13.9	-
7	21.6	15.5
8	17.9	17.6
9	19.7	14.6
10	18.9	19.7
Mean values	***16.3***	***15.5***

Samples	Compression strength (N/mm²) at 850°C	
	E direction	**T direction**
1	14.2	11.8
2	16.7	13.5
3	20.1	13.6
4	15.9	16.9
Mean values	***16.7***	***14.0***

Samples	Compression strength (N/mm²) at 900°C	
	E direction	**T direction**
1	35.0	27.4
2	42.2	34.9
3	17.6	15.2
4	24.2	12.5
Mean values	***29.8***	***22.5***

Samples	Compression strength (N/mm²) at 950°C	
	E direction	**T direction**
1	21.5	20.9
2	26.5	35.7
3	25.0	13.8
4	20.6	16.0
Mean values	***23.4***	***21.6***

Until 850°C, the compression strength in both directions varies between 13 and 17 N/mm^2. From 900 °C , the compression strength increases twice in the extrusion direction which is very interesting

8. Discussion of the results

Material	Ytong Cellular Block	Clayey diatomite Material	Porotherm Hollow Block
Bulk density (g/cc)	0.4 - 0.5	1.3	1.8
Water sorption (%)		34	13-16
Compression strength (N/mm2)	3	22-30	25

The properties of the clayey diatomite material fired at 900° C are to be ranked between the Ytong cellular concrete blocks and the fired (at 950° C) clay hollow blocks (extruded). Nevertheless, it looks like the clayey diatomite material has a higher compression strength than the other materials with very good lightweight and insulating properties. This last property could be quantified in a later stage by doing thermal conductivity measurement on the three types of materials.

Remark : bulk density and compression strength of Porotherm blocks in this table are figures calculated from the data sheet, taking into account that the area occupied by the holes is about 50% of the total area perpendicular to the extrusion direction .

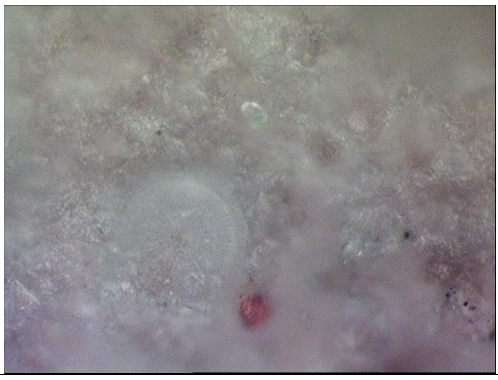
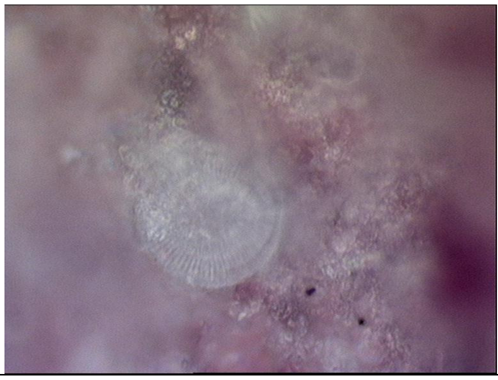
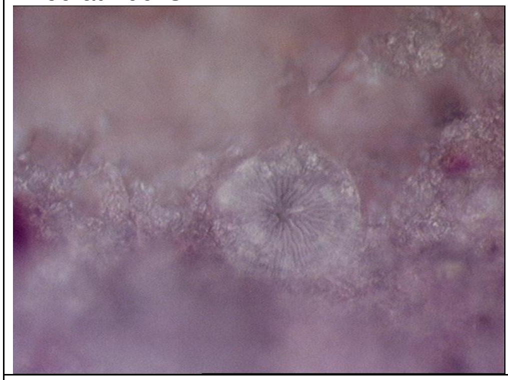
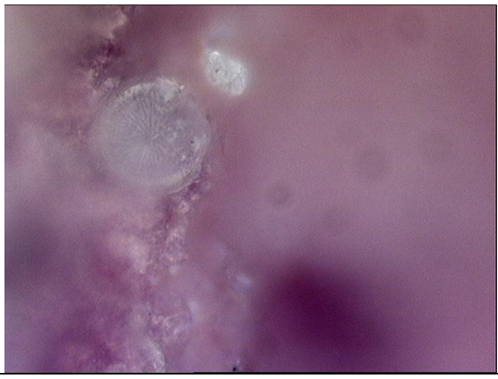
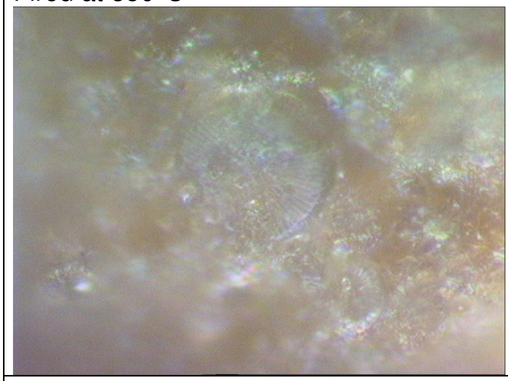
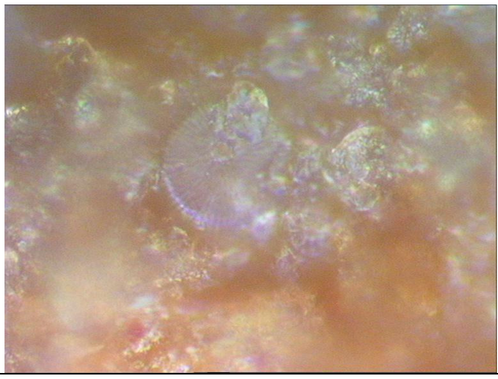
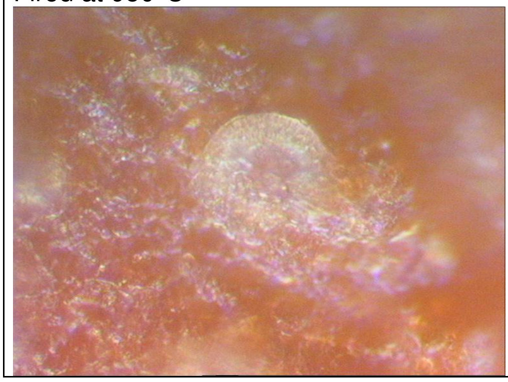
9. Optical microscopy pictures

We took pictures of the samples with a CCD coupled to an optical microscope to check the behavior of the diatomite texture at each firing temperature. The detailed texture features of the fossils starts to grow blurred from 900°C but the structure stays intact (see page 15).

Mons , the 3rd of September 2004 ;

Dr N. Malengreau

Dr J. Tirlocq – Head of research department

Raw material 	Fired at 600℃ 
Fired at 700℃ 	Fired at 800℃ 
Fired at 850℃ 	Fired at 900℃ 
Fired at 950℃ 	Pictures taken by optical microscopy (x1000)