INVESTIGATION OF INORGANIC MATERIALS DERIVED FROM WATER PURIFICATION PROCESSES FOR CERAMIC APPLICATIONS

Report to

THE WATER RESEARCH COMMISION

by

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EXECUTIVE SUMMARY

INVESTIGATION OF INORGANIC MATERIALS DERIVED FROM WATER PURIFICATION PROCESSES FOR CERAMIC APPLICATIONS

MOTIVATION

Sludges and silts produced by waterworks and dredging operations often create disposal problems. Finding a use for such materials could eliminate or reduce these disposal problems, eliminate the spoiling of land or fouling of waterways, reduce disposal costs and create possibilities of financial return from the sale of products produced.

The supply of low-cost housing is a national priority and the manufacture of cheap bricks, blocks, and tiles in the rural and urban areas would help to alleviate this problem. The production of such building elements can also be labour intensive, thus supplying employment opportunities.

Work at MATTEK has identified waterworks sludge as a source of raw material for the production of bricks and tiles. The building elements developed either meet or are well below usual production costs. Extension of this work to other waterworks sludges shows possibilities. A feasibility study was carried out, based on an independent market analysis, for the manufacture of bricks, blocks and tiles. This study proved such manufacture to be a viable proposition.

OBJECTIVE

The main objective was to study the technical and economical feasibility of using waterworks sludges from Umgeni Water for the production of bricks, blocks, tiles or possibly other ceramic applications.

RESULTS

The suitability of the Wiggins Waterworks sludge for brickmaking and tilemaking was investigated.

Severe difficulties were experienced in all critical areas of ceramic processing, i.e. forming, drying and firing. Fortunately most of these difficulties have been overcome to a greater or lesser extent and valuable experience has been gained in processing waterworks sludge in general.

Whilst the pressing of tiles worked well, the pressing of bricks proved to be the greatest area of difficulty of the whole investigation. The major effort of the investigation was focused on overcoming this difficulty. The bricks pressed well in the die, but after being removed from the die for several hours they were prone to cracking. This difficulty has been largely overcome by optimisation studies.

The drying of the pressed tiles and bricks was originally thought to present no difficulties, but eventually it was found that this was the cause of the cracking of the unfired bricks. This was unexpected and very unusual because the drying shrinkage of the bricks was very low and in the case of extruded bricks this would normally never cause such a problem. This difficulty was overcome by perforating the bricks.

In the case of firing it was found that if the bricks or tiles were fired in the normal manner up to maximum temperature, usually in the range of 900° to 1000°C, they would crack or warp badly. This difficulty was overcome by introducing a calcination step at 600°C, to burn off deflocculants and organic matter. The firing cycle finally adopted was: one day to reach 600°C from room temperature, left for one day at 600°C, followed by an 80°C rise per hour to reach the required maximum temperature. This solved the problem of the cracking and warping for the tiles and the rings, but not for the solid bricks. It was found that noxious gases were evolved in the temperature range of 250°C to 550°C. This could be overcome by venting or scrubbing the gases.

CONCLUSIONS

It was concluded that the Wiggins Waterworks sludge would be suitable for producing rustic tiles and for manufacturing stock or face bricks.

ACHIEVEMENT OF OBJECTIVES

The technical feasibility of using the Wiggins Waterworks' sludge (Umgeni Water) for the production of bricks and tiles was demonstrated. Most of the difficulties experienced in the critical areas of ceramic processing, i.e. forming, drying and firing, have been overcome to a greater or lesser extent. This has provided valuable experience in processing waterworks sludge from other areas in South Africa.

RECOMMENDATIONS

It is recommended that a techno-economic feasibility study of glazed and unglazed tiles, including pilot plant trials, be undertaken on the Wiggins Waterworks sludge. It is further recommended that full-size perforated bricks be investigated, before pilot plant trials are undertaken on this sludge.

TECHNOLOGY TRANSFER

A summary of all the results obtained in this study will be made available to all water boards which have problems with the disposal of waterworks sludges.

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1. INTRODUCTION

1.1 SCOPE

To establish the suitability of waterworks sludge for brickmaking and tilemaking.

1.2 MOTIVATION

Sludges and silts produced by waterworks and dredging operations often create disposal problems and finding a use for such materials could possibly eliminate or reduce these disposal problems, eliminate the spoiling of land or fouling of waterways, reduce disposal costs, and create possibilities of financial return from the sale of products produced.

The supply of low-cost-housing is a national priority and the possible development of cheap bricks, blocks, and tiles in the rural and urban areas would help alleviate this problem. The production of such building elements can also be labour intensive thus supplying employment opportunities.

Work at MATTEK has identified a water work sludge as a source of raw material for the production of bricks and tiles (1). The building elements developed either meet or are well below usual production costs. Extension of this work to other waterworks sludges shows possibilities. Funding of further development by waterworks is usually hampered by their size, by the fact that it is not considered part of their normal business portfolio, and by their opinion that the levy on water use should pay for it.

A feasibility study (1) was carried out based on an independent market analysis for the manufacture of bricks, blocks, tiles etc. This project proved to be viable.

1.3 OBJECTIVES

a) Preliminary technical and economic feasibility study of waterworks sludges from Umgeni Water for the production of bricks, blocks, tiles, or perhaps other alternative traditional ceramic applications.

b) Preliminary technical feasibility studies of waterworks sludges produced at Cape Town as well as the Orange Free State.

c) Technical and economic feasibility study of the commercially viable identified materials for the production of bricks, blocks, tiles, or other alternative ceramic applications.

1.4 RESTRICTION (Objective b)

b) As instructed by the Water Research Commission no preliminary technical feasibility study of waterworks sludge, produced at Cape Town as well as the Orange Free State, was done and all efforts were focused on Objective a).

1.5 WIGGINS BACKGROUND (1)

The amount of waterworks sludge produced by Umgeni Water at Wiggins in Durban was estimated at 5 m^3/day, at a solids content of 30-40%, in 1992. This represents a small sludge producer in an urban area. This amount of sludge is minimal but the Durban City Corporation does not allow the sludge to be dumped at the waterworks concerned and it must be transported in 7 m^3 bins at a 1992 transport cost of R167/bin. The annual transport cost was R43 587 at that time.

This annual transport cost assumes an average solids content of 35% and that 1 m^3 wet = 1 ton wet which gives 1,75 tons of dry sludge daily. The estimated annual Wiggins potential brick production from sludge if technically feasible is 213 000 assuming 3 tons/1000 bricks. The amount of bricks produced is negligible and poses no threat to any brickmaker but may suit Umgeni Water's own internal needs or that of a small entrepreneur. The yearly value of the sludge excludes capital equipment and production costs. A medium size brick plant can produce at a rate of 10 000 to 20 000 bricks/hour. Assuming R200/1000 bricks this will generate annually R42 600. Umgeni Water could nearly recoup their transport costs by producing bricks.

Another possibility is using the sludge for a higher value product such as glazed wall tiles, rustic tiles or pottery if technically feasible. If one assumes that 500 grams of dry sludge will be used in a 150 x 150 mm tile and that 42 tiles are necessary for 1 m^2 then 21 kgs will produce 1 m^2 of tiling. The estimated Wiggins potential tile production from sludge if technically feasible is 30 400 m^2. Assuming R30/m^2 this will generate annually R912 500. This yearly value of the sludge excludes capital equipment and production costs.

Tile production is a much more lucrative option in the view of the low amount of sludge produced. We envisage that the recipient of the developed technology would be a local tilemaker or small entrepreneur. This technology could be labour intensive as well as capital intensive. The amount of tiles produced, though small, poses some threat to the Transvaal tilemakers. One advantage of Durban sludge tiles is their proximity to the Natal market as opposed to the Transvaal tile manufacturers' distances to the Natal tile market. However, cheap imports through Durban could be a threat.

1.6 SAMPLE INFORMATION

A sample of five tons of centrifuged waterworks sludge was submitted for investigation on 11 November 1992. The sample originated from Umgeni Water, Wiggins Waterworks in Durban. The sample was produced on 9 and 10 November 1992.

1.7 REPORT

Sections 1 and 2 provide basic information for the rest of the report. Sections 3 to 11 are more or less independent of each other and the work progressed in that order except for Section 11 which took place after Section 3. Sections 12 to 14 provide general information on the whole report.

2. CHARACTERIZATION AND TEST METHODS

2.1 SCOPE

To characterize the sludge submitted by different analytical techniques and to describe the test methods.

2.2 EXPERIMENTAL WORK

2.2.1 Sample preparation

Samples from the sludge material were dried at 110°C, pulverized and sieved through a 0,125 mm mesh. This prepared powder was used for mineralogical and chemical composition analysis, including carbon and hydrogen determinations.

2.2.2 Mineralogical composition

The mineralogical composition of the sludge was determined by means of X-Ray diffraction analysis.

2.2.3 Chemical composition

The chemical composition of the sludge was determined by means of X-Ray fluorescence analysis.

2.2.4 Particle size distribution

A sample was taken from the supplied sludge and analyzed for particle size distribution. The particle size distribution was determined using a Leeds and Northrup Microtrac small particle analyzer with a 0,005M sodium pyrophosphate solution as dispersant. The suspension using 1 mm sieved material was dispersed by means of an ultrasonic probe before analysis.

2.2.5 Differential thermal analysis

The differential thermal analysis trace was determined with a DTA with a 2 holed Ni sample holder, Pt/Pt10 Rh t/c at 5 x magnification.

2.2.6 Thermogravimetric analysis

The thermogravimetric analysis trace was determined with a Dupont TG with a rate of 15°C/min in air and sensitivity of 2 mg/inch.

2.2.7 Water content

The water content was determined by drying the sample at 110°C overnight.
The water content is expressed as a percentage of dry sludge or as a water/solids percentage.

2.2.8 Drying shrinkage

The drying shrinkages were determined across marks made 100 mm apart on the samples or by direct measurement of the samples.

2.2.9 Firing

The dried samples were fired in a laboratory electric furnace. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were soaked at this temperature for 5 hours and then they were allowed to cool to room temperature.

2.2.10 Firing shrinkage

The firing shrinkages were determined across marks made 100 mm apart on the dried samples or by direct measurement of the samples.

2.2.11 Water absorption

The water absorption was determined by drying the samples at 110°C overnight, cooling them down to room temperature and soaking them in water for 24 hours (C) and boiling them in water for 5 hours (B) and were determined according to SABS 227-1986 (2). The saturation coefficient is the ratio of C/B.

2.2.12 Pressing

A 100 ton tile press was used to press the different samples. The samples were pressed at different dial readings which correspond to the different loads given in Table 2.1.

Table 2.1 : Tile press calibration

Dial readings (kp^*/cm^2)	Load (kN)
50	80
100	167
150	253
200	340
250	427
300	513
350	600
400	686
450	773
500	859

*German kilo pond

2.3 RESULTS

2.3.1 Mineralogical composition

The crystalline components of the sludge consisted mainly of quartz, and a minor amount of kaolinite; a very small amount of feldspar is present. No swelling clay minerals were detected. The free quartz content was approximately 15% (to the nearest 5%).

2.3.2 Chemical composition

The chemical composition of the sludge is given in Table 2.2.

The rational analysis for the sludge, calculated from the chemical analysis, is given in Table 2.3. The assumptions of the rational analysis (3) are assumed and the feldspar convention was used.

The C and H analysis of the sludge sample using the combustion method presented as percent by mass, is given in Table 2.4.

2.3.3 Particle size distribution

The results of the particle size distribution of the sample as cumulative percent finer than equivalent spherical diameter are given in Table 2.5 and Figure 2.1.

2.3.4 Differential thermal analysis

The trace of the differential thermal analysis curve of the sample is given in Figure 2.2. A large endotherm occurs at approximately 100°C, then a small one at approximately 300°C, then a medium one at approximately 540°C. A broad exotherm or the start of an endotherm occurs at approximately 900°C.

2.3.5 Thermogravimetric analysis

The trace of the thermogravimetric analysis curve of the sample is given in Figure 2.3. Up to 200°C a 6,1% loss occurs, between 200 and 420°C a further loss of 7,3% occurs, and from 420 to 680°C a final loss of 6,2% occurs. A total loss of 19,6% is recorded for the sample. It was also found that noxious gases were evolved in the temperature range of 250 to 550°C. This can be overcome by venting or scrubbing the gases.

2.3.6 Water content

The water content of the sludge is 91,5% on a dry basis or 47,8% water and 52,2% sludge as a water/solids mixture.

Table 2.2 : Chemical composition of sludge F in mass percentage

Expressed as oxides	F
SiO_2	45,6
Al_2O_3	20,0
Fe_2O_3	8,41
MgO	0,77
CaO	0,84
Na_2O	0,48
K_2O	1,32
S	0,10
TiO_2	0,74
MnO	0,64
LOI*	19,18
Total	98,08

*Loss-on-ignition

Table 2.3 : Rational analysis of the sludge in mass percentage

Phase	F
Clay substance	45,64
Feldspar	10,66
Quartz	17,47
Fe_2O_3	8,41
TiO_2	0,74
MgO	0,77
CaO	0,84
S	0,10
MnO	0,64
Organics + CO_2	12,81
Total	98,08

Table 2.4 : Carbon and hydrogen determinations

Sample	Carbon	Hydrogen
F	4,66	1,81

Table 2.5 : Particle size distribution of waterworks sludge

Equivalent spherical diameter(microns)	Cumulative percent finer
	F
14,92	100,0
10,55	87,8
7,46	62,9
5,27	42,7
3,73	26,8
2,63	14,6
1,01	7,7
0,66	3,3
0,43	0,5
0,24	0,0
0,17	0,0
% 10	2,0
% 50	6,1
% 90	11,4

2.4 DISCUSSION OF RESULTS

2.4.1 Mineralogical composition

The major crystalline phase in the sludge is approximately 15% quartz which means that the minor phase of kaolinite cannot account for most of the material and a high content of one or more amorphous phases are present. From the rational analysis it is seen that 46% clay substance is present indicating that a major portion of it is amorphous.

2.4.2 Chemical composition

The silica, alumina and alkali contents of the sludge are normal from the brickmaking point of view. The iron and loss-on-ignition contents, however, are both high.

Figure 2.1 : Sludge particle size distribution

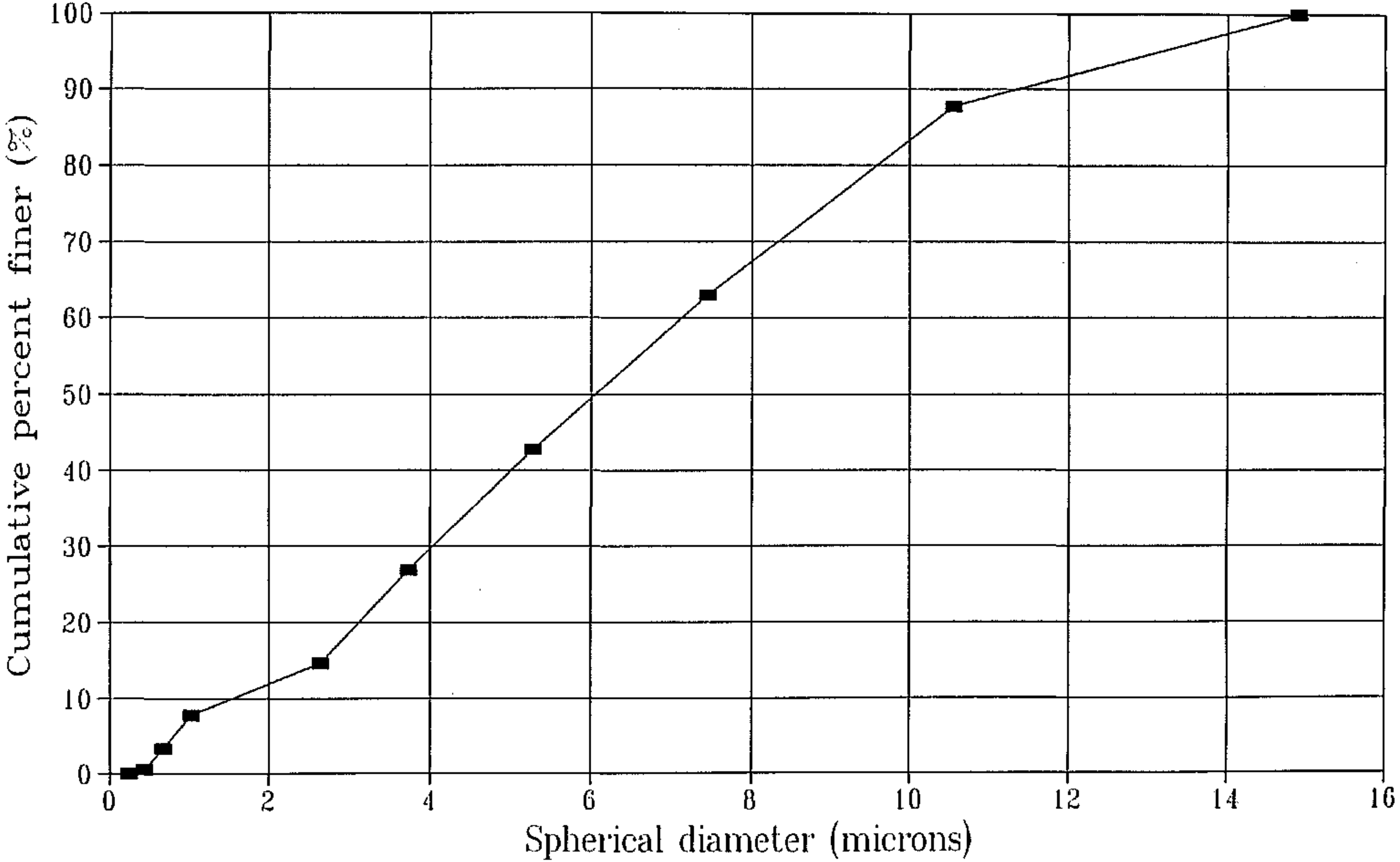
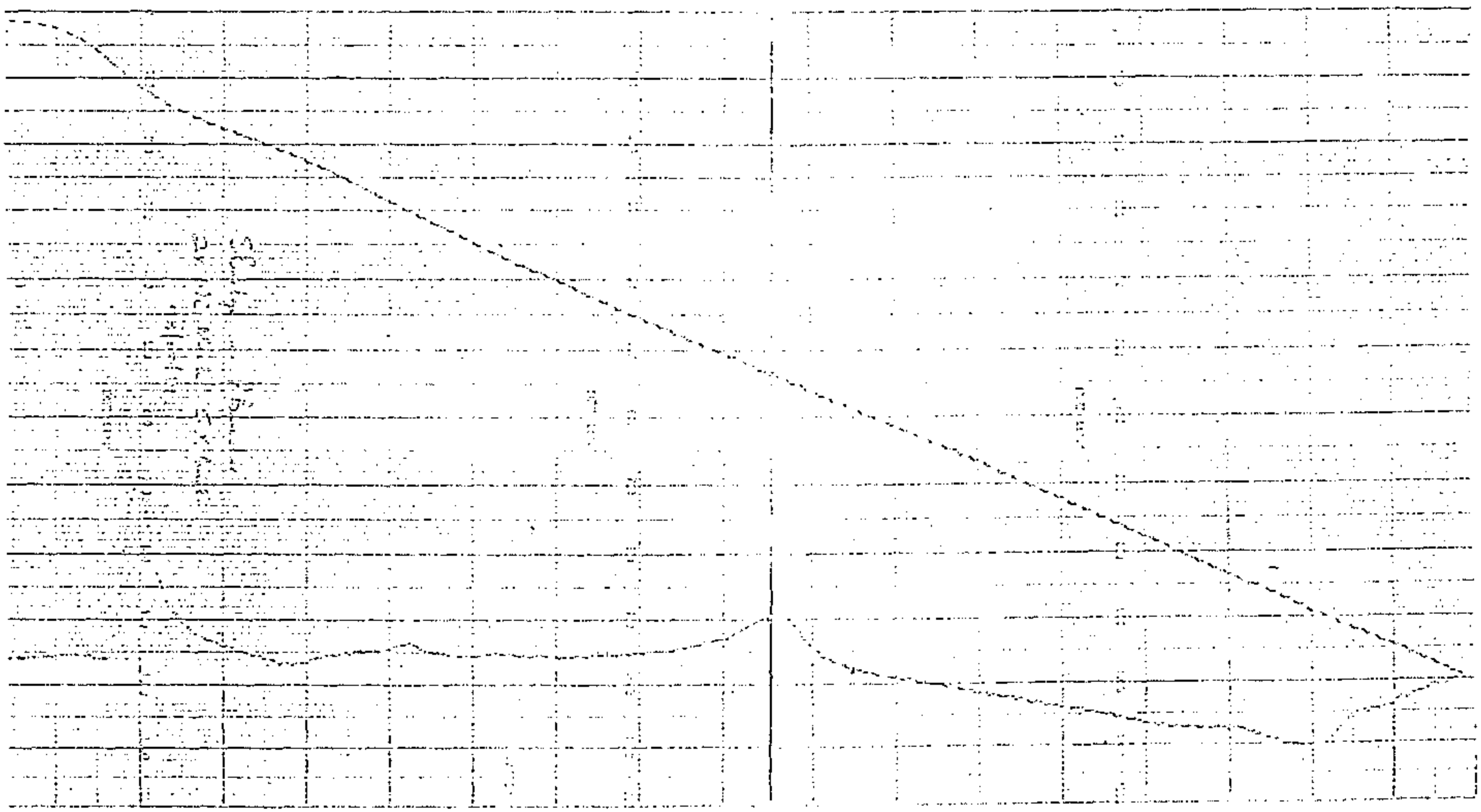


Figure 2.2 : Sludge differential thermal analysis



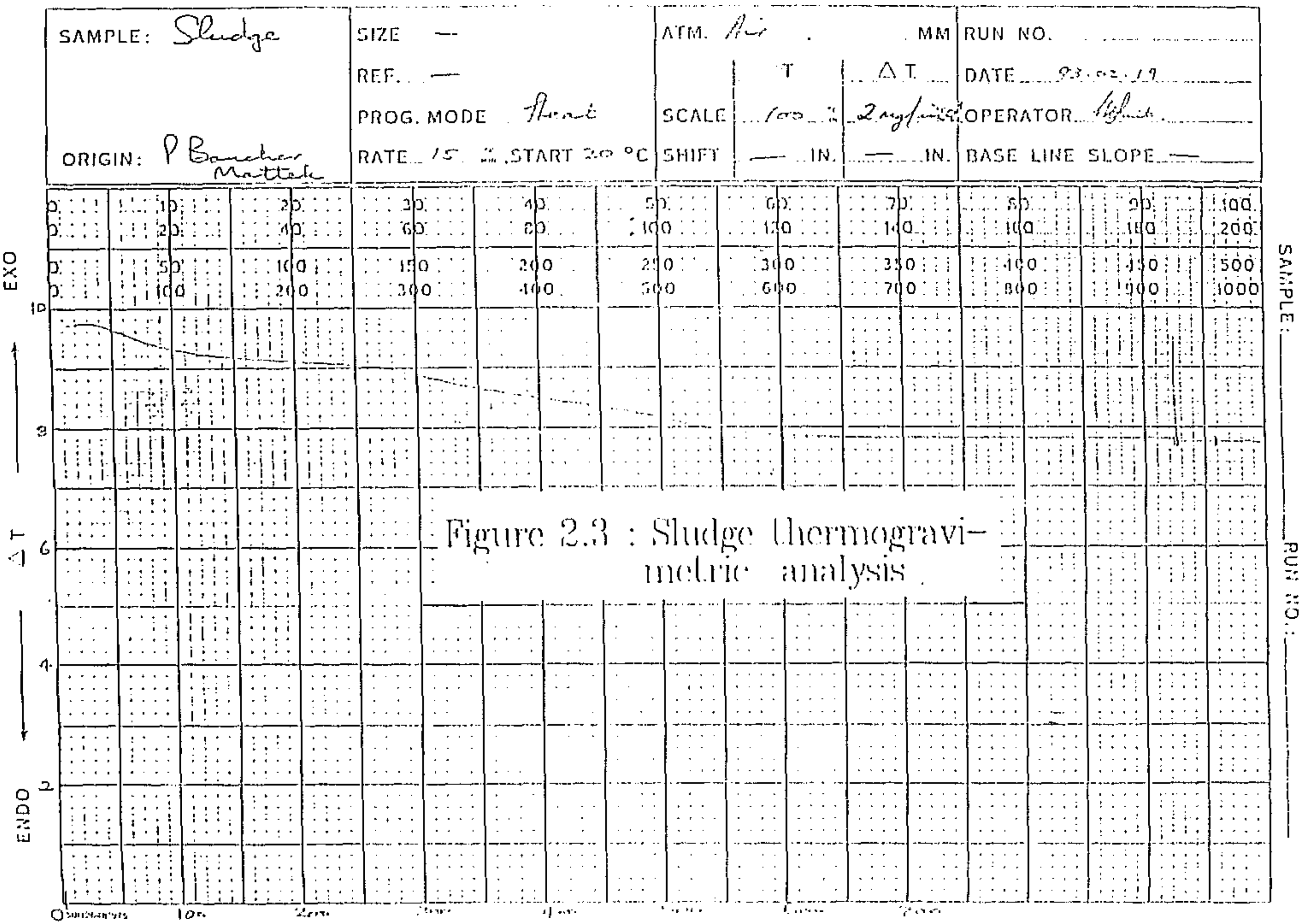


Figure 2.3 : Sludge thermogravimetric analysis

2.4.3 Particle size distribution

The particle size distributions of Table 2.5 are grouped into brickmaking fractions in Table 2.6.

Table 2.6 : Particle size fractions of sludge

Fraction	F (%)
< 2 μm	14
> 2 & < 20 μm	86
> 20 μm	0

When compared with brickmaking clays the sludge is too fine. The fraction greater than 20 μm is too low, the fraction between 2 μm and 20 μm is too high, however, the fraction below 2 μm is comparable to brickmaking clays. The implications for brickmaking are that the sludge may probably not extrude well due to drying shrinkage problems but may press reasonably.

2.4.4 Differential thermal analysis

From Figure 2.2 the first endotherm is due to the drying of the sample, the 300°C one is probably due to breakdown of the flocculant and the 540°C one due to dehydroxylation and burning off of carbonaceous matter. The event at 900°C is the due to formation of ceramic compounds. No evidence of quartz is found.

2.4.5 Thermogravimetric analysis

The loss in mass of the sample is approximately uniform up to 680°C where it ceases. However, three areas are distinguished which correspond to the first three DTA events of Section 2.4.4. A drying event at 80°C, flocculant burnt out at 320°C and carbonaceous burn off and dehydroxylation at 570°C.

The loss in mass of the sample agrees with the loss-on-ignition value from Table 2.2 if it is normalized.

2.4.6 Water content

At a 48% water content the drying of the sludge will be a major processing step in practice. In the rest of this report the drying step is assumed. It is recommended that drying techniques be investigated.

2.5 CONCLUSIONS

a) The major crystalline phase in the sludge is approximately 15% quartz and a minor amount of kaolinite. The rational analysis indicates that a major portion of the clay substance is amorphous.

b) The silica, alumina and alkali contents of the sludge are normal from the brickmaking point of view. The iron and loss-on-ignition contents are both high.

c) When compared with brickmaking clays the sludge is too fine. The fraction greater than 20 μm is too low, the fraction between 2 μm and 20 μm is too high, however, the fraction below 2 μm is comparable to brickmaking clays.

d) The brickmaking implications of the particle size distribution are that the sludge may probably not extrude well but may press reasonably.

e) At a 48% water content the drying of the sludge will be a major processing step in practice.

f) It was found that noxious gases were evolved from the sludge in the temperature range of 250 to 550°C.

2.6 RECOMMENDATIONS

a) It is recommended that the present investigation concentrate on the pressing of the sludge.

b) It is recommended that drying techniques be investigated.

c) It is recommended that noxious gases evolved in the temperature range of 250 to 550°C be eliminated by venting or scrubbing the gases.

3. PRESSING OF MOSAIC TILES

3.1 SCOPE

To determine the firing range of pressed mosaic tiles and the effect of the water used for pressing on the fired properties of these tiles.

3.2 EXPERIMENTAL WORK

3.2.1 Sample preparation

For the preparation of mosaic tiles the dried material was first hammer milled until all material passed through a 1 mm sieve. It was then mixed by hand with 0, 1, 3, 5, 10, and 18% water. The tiles were then pressed with the tile press. The dimension of the mosaic tile die was 50 mm square. The tiles were then dried in air at room temperature for two days followed by drying overnight at 110°C.

3.2.2 Firing

The dried tiles were fired in a laboratory electric furnace. A tile of each water content was fired at each of the following temperatures: 700, 800, 900, 950 and 1000°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

3.3 RESULTS

3.3.1 Forming properties

The results obtained on the pressed tiles are given in Table 3.1. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F3 means 3% water was added to sludge F on a dry basis.

Table 3.1 : Summary of forming properties

Forming property	F0	F1	F3	F5	F10	F18
Pressing	good	good	good	good	good	good
Dial reading (kp/cm^2)	250	250	250	250	250	200
Mixing water (%)	1,3	1,2	2,6	4,7	10,1	17,8
Green strength	good	good	good	good	good	good
Drying shrinkage (%)	-1,4	-1,4	-0,8	-0,4	0,5	2,1
Drying	good	good	good	good	good	good
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

The dial readings of 200 and 250 kp/cm^2 correspond to pressures of 136 MPa (340 000 N/2 500 mm^2) and 171 MPa (427 000 N/2 500 mm^2), respectively. See Table 2.1 for tile press calibration.

3.3.2 Fired properties

The results obtained on the fired tiles are given in Tables 3.2 to 3.7, and are graphically presented in Figures 3.1 and 3.4. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F3 means 3% water was added to unfired sludge F on a dry basis. Because of the small size of the mosaics total shrinkage is based on a nominal 50 mm size.

Table 3.2 : Fired properties of the mosaic tiles for sample F0

PROPERTY	TEMPERATURE (°C)						
	500	700	800	900	950	1000	1050
Total shrinkage (%)		0,3	1,0	3,5	6,7	12,2	
Mass loss (%)		18,5	19,4	21,1	21,2	8,6	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		33,2 33,9 0,98	27,7 29,4 0,94	22,5 23,2 0,97	16,4 16,8 0,98	8,6 9,3 0,93	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good, <craze	good, <craze	good, <craze	

Table 3.3 : Fired properties of the mosaic tiles for sample F1

PROPERTY	TEMPERATURE (°C)						
	500	700	800	900	950	1000	1050
Total shrinkage (%)		0,7	1,2	3,9	6,7	12,4	
Mass loss (%)		18,8	18,6	21,7	20,8	19,1	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		30,5 31,2 0,98	27,4 28,0 0,98	24,1 24,5 0,99	17,6 18,3 0,96	8,2 8,5 0,96	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good, <craze	good, <craze	good, <craze	

Table 3.4 : Fired properties of the mosaic tiles for sample F3

PROPERTY	TEMPERATURE (°C)						
	500	700	800	900	950	1000	1050
Total shrinkage (%)		1,1	1,6	4,2	6,3	11,3	
Mass loss (%)		19,0	21,6	21,4	22,9	24,2	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		26,9 27,6 0,97	25,7 23,6 1,09	21,0 21,4 0,98	15,1 15,5 0,98	6,9 7,6 0,90	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good, <craz e	poor, cracked	poor, cracked	

Table 3.5 : Fired properties of the mosaic tiles for sample F5

PROPERTY	TEMPERATURE (°C)						
	500	700	800	900	950	1000	1050
Total shrinkage (%)		1,4	1,9	4,3	6,1	9,5	
Mass loss (%)		18,5	22,2	21,7	21,8	19,2	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		23,5 24,2 0,97	19,4 21,2 0,92	17,3 18,0 0,96	12,1 12,5 0,97	5,6 6,3 0,89	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good, <craze	bad, cracked	bad, cracked	

Table 3.6 : Fired properties of the mosaic tiles for sample F10

PROPERTY	TEMPERATURE (°C)						
		700	800	900	950	1000	1050
Total shrinkage (%)		2,0	2,7	5,1	6,8	9,5	
Mass loss (%)		18,8	19,4	21,9	22,2	23,6	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		23,8 24,1 0,99	21,5 19,0 1,13	14,9 15,6 0,96	11,5 12,2 0,94	5,1 6,2 0,82	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good	bad, cracked	bad, cracked	

Table 3.7 : Fired properties of the mosaic tiles for sample F18

PROPERTY	TEMPERATURE (°C)						
		700	800	900	950	1000	1050
Total shrinkage (%)		3,5	4,0	6,4	8,9	15,0	
Mass loss (%)		18,2	19,3	21,7	21,4	21,0	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		29,1 29,8 0,98	26,6 27,3 0,97	23,8 23,8 1,00	16,3 17,0 0,96	6,9 8,0 0,86	
Colour		brown	brown	brown	brown	brown	
Remarks after firing		good	good	good	good, crazed	good, <craze	

Figure 3.1 : Drying shrinkage vs pressing water (mosaics)

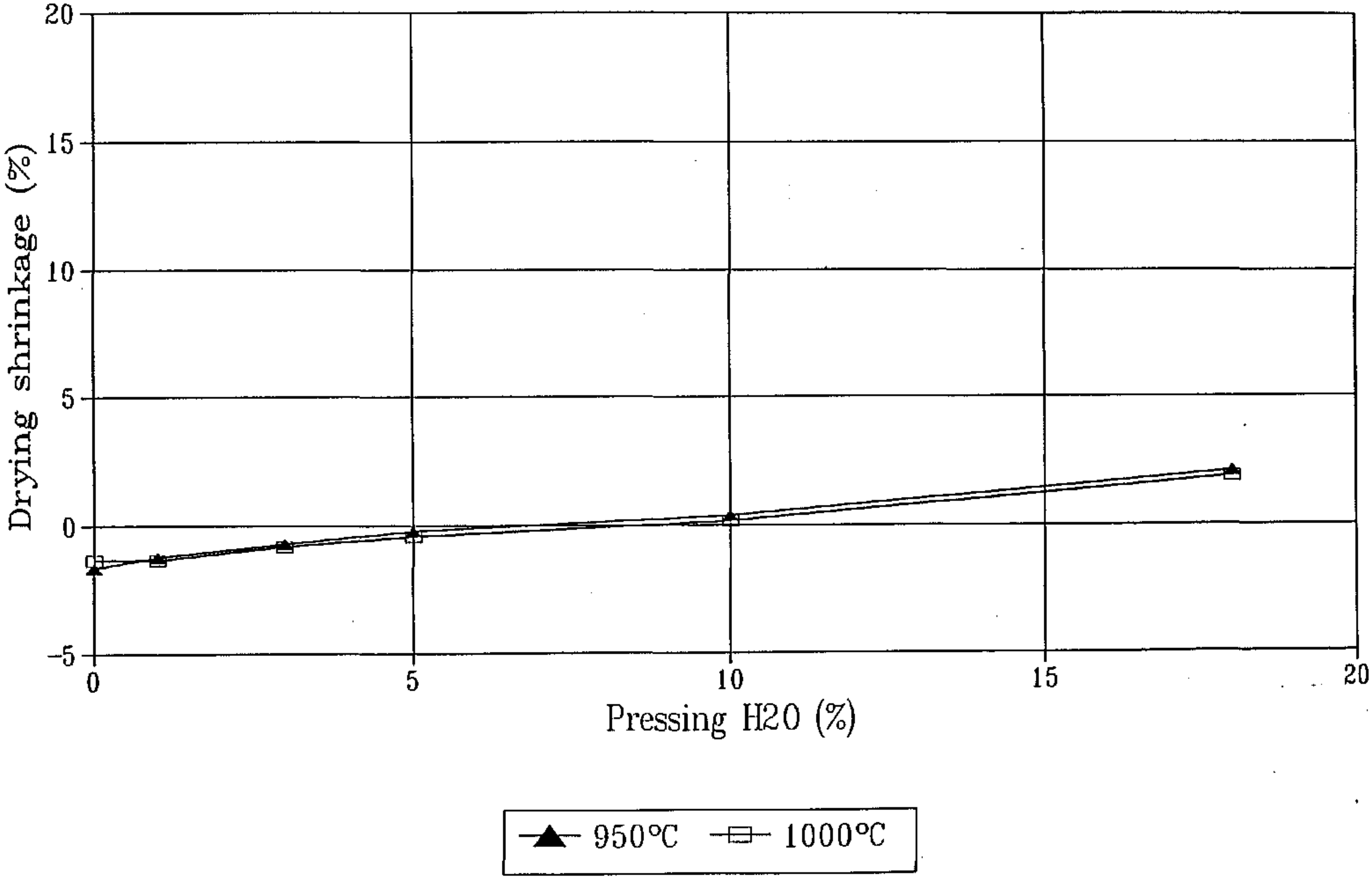


Figure 3.2 : Water absorption vs firing temperature (mosaics)

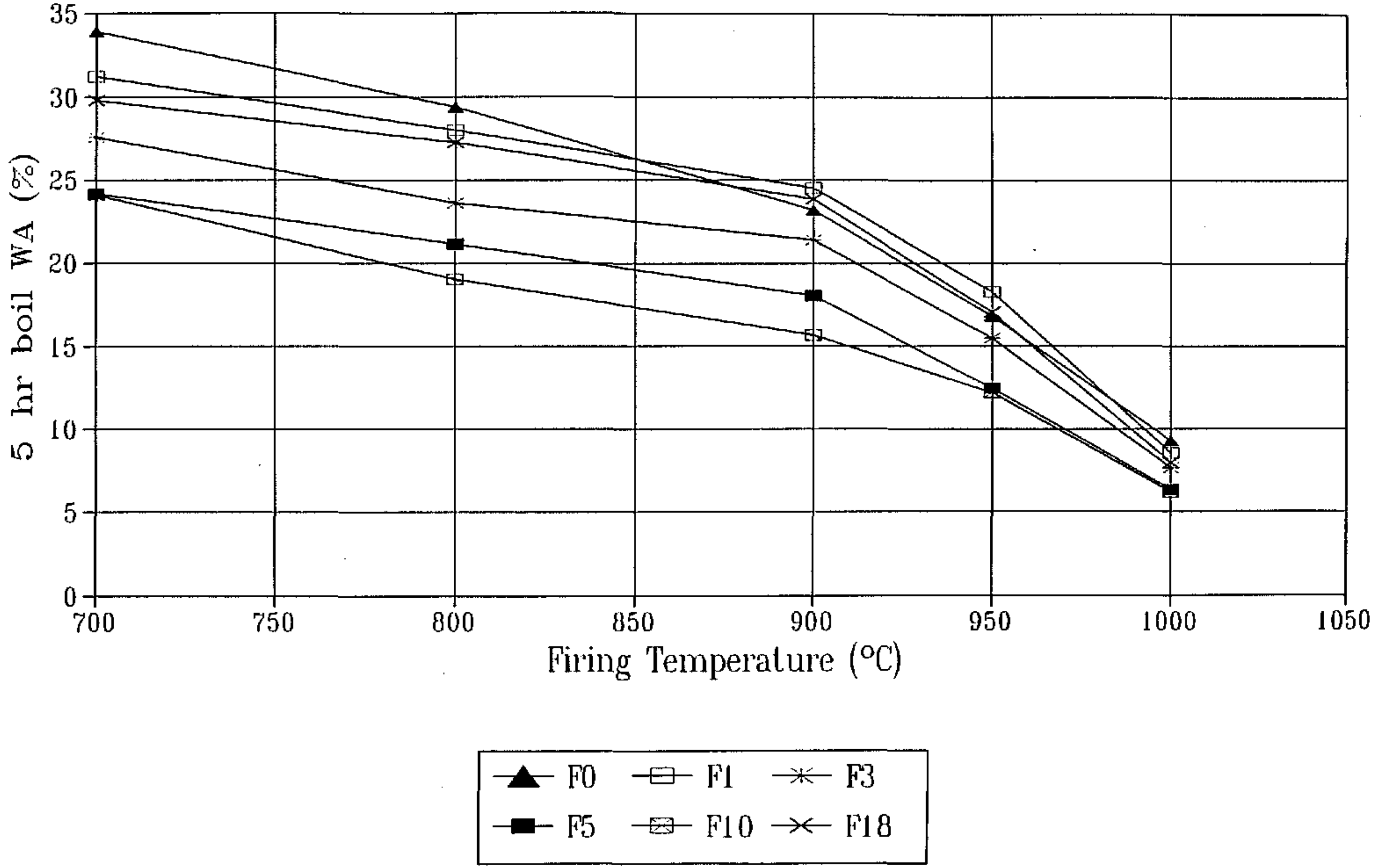


Figure 3.3 : Water absorption vs pressing water (mosaics)

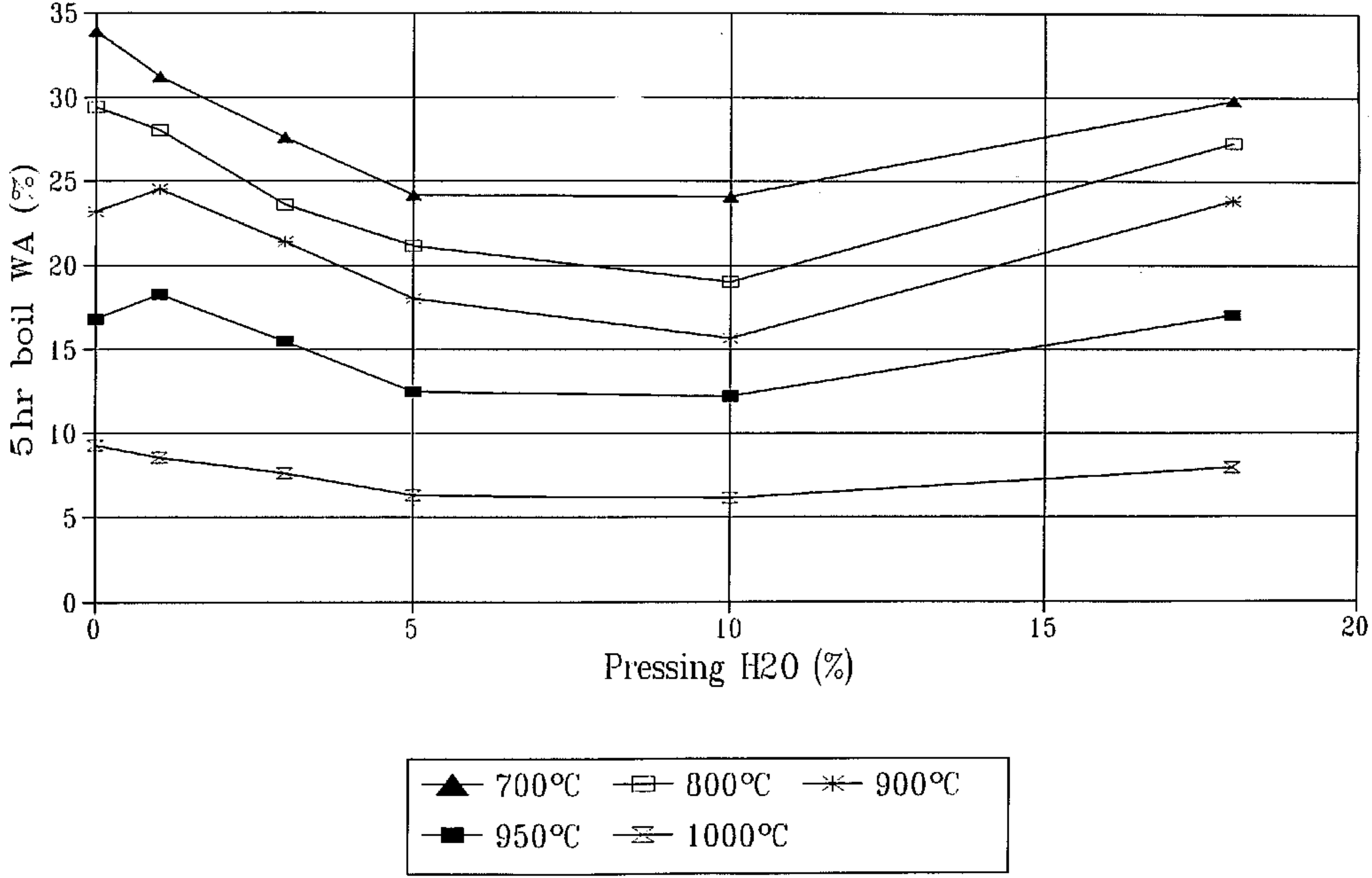
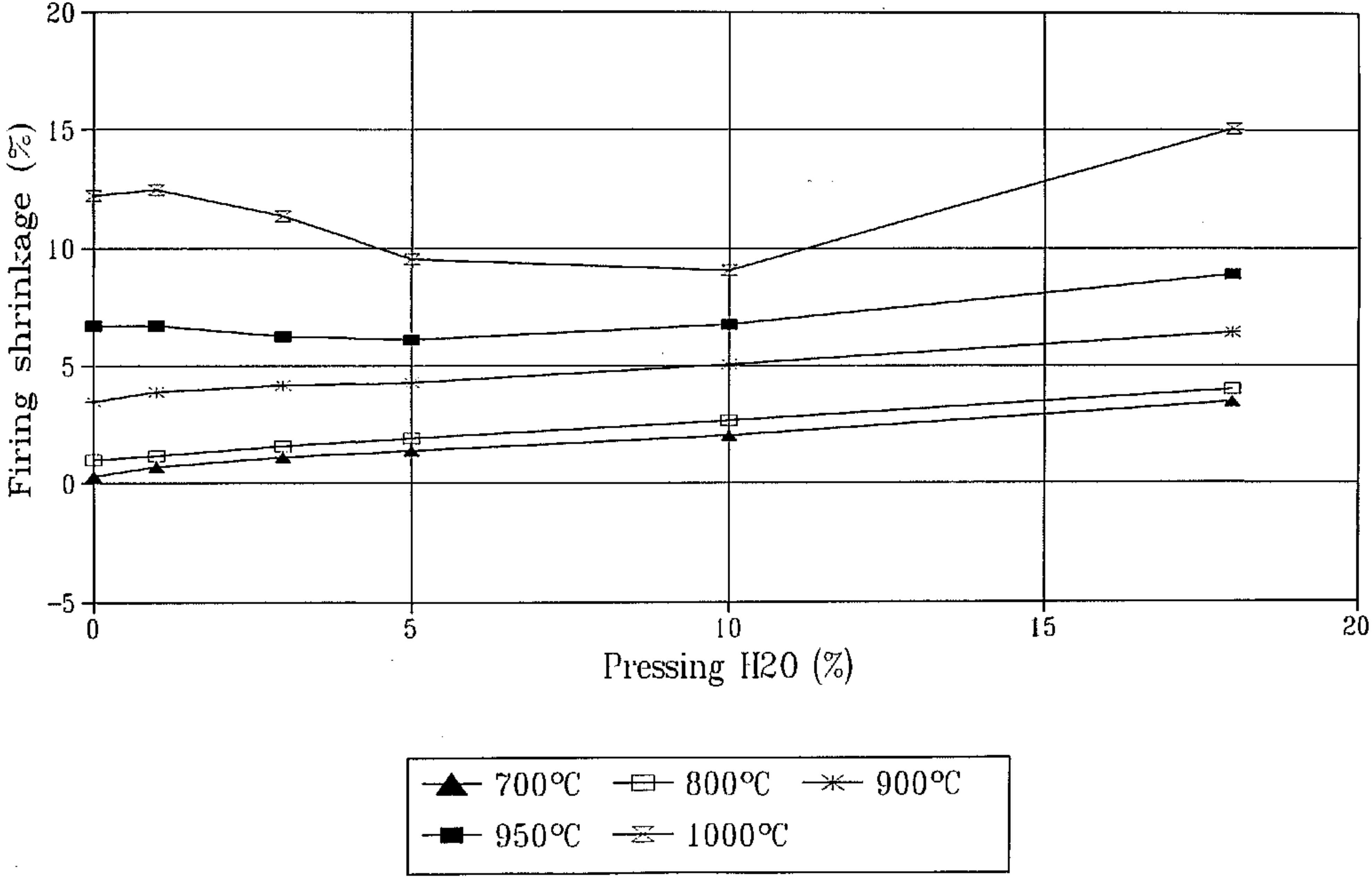


Figure 3.4 : Total shrinkage vs pressing water (mosaics)



3.4 DISCUSSION OF RESULTS

3.4.1 Forming properties

Table 3.8 gives comments on the brickmaking forming of mosaics. This Table is based upon the data given in Table 3.1.

Table 3.8 : Comments on Brickmaking forming of mosaics

Sample	Pressing behaviour	Drying behaviour	Green strength	Brickmaking comments
F0	good	good	good	good
F1	good	good	good	good
F3	good	good	good	good
F5	good	good	good	good
F10	good	good	good	good
F18	good	good	good	good

The comments on brickmaking forming of mosaics are all good.

Drying shrinkage or expansion values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. Figure 3.1, based on samples to be fired at 950 and 1000°C, shows that the mosaic drying shrinkages vary between -2 and 2%, with water content up to 18%, which is good.

All the mosaics which were dried at room temperature showed no signs of cracking. The samples are thus not sensitive to drying and precautions do not have to be taken in practice to ensure that tiles made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

3.4.2 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks. From Figures 3.2 and 3.3 all samples fired at 950°C have good water absorptions.

Table 3.9 gives the firing range of the mosaics based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 3.2 to 3.7 and Figures 3.2 and 3.3.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable. Ceramic kiln firing ranges of 20, 40 and 50°C are regarded as short, medium and long, respectively.

Table 3.9 : Water absorption firing range of mosaics

Sample	5hr WA range ($\sim 20 \geq WA \geq \sim 8$) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
F0	17 - 9	950 - 1000	very short	poor
F1	18 - 8	950 - 1000	very short	poor
F3	16 - 8	950 - 1000	very short	poor
F5	18 - 12	900 - 950	very short	poor
F10	19 - 12	800 - 950 +	medium	good
F18	17 - 8	950 - 1000	very short	poor

All the samples, except F10, would make poor clamp brickmaking materials from the point of view of water absorption firing range. The water absorption range of sample F10 is good. All the samples, except F10 which is good, would make acceptable ceramic kiln brickmaking materials from the water absorption firing range point of view.

3.4.3 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. Ceramic kiln firing ranges of 20, 40 and 50°C are regarded as short, medium and long, respectively.

Table 3.10 gives the firing range of the different mosaics based upon an acceptable firing shrinkage range as described above. This Table is derived from the data given in Tables 3.2 to 3.7 and Figure 3.4. This data, though based on total shrinkage, instead of firing shrinkage only, will make very little difference in this case.

Table 3.10 : Firing shrinkage firing range of mosaics

Sample	Firing shrinkage ($0 \leq FS \leq 7$) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
F0	0 - 7	700 - 950	long	good
F1	1 - 7	700 - 950	long	good
F3	1 - 6	700 - 950	long	good
F5	1 - 6	700 - 950	long	good
F10	2 - 7	700 - 950	long	good
F18	4 - 6	700 - 900	long	good

All the mosaics would make good clamp and ceramic kiln brickmaking materials from the firing shrinkage point of view. Above 950°C high shrinkage begins, corresponding to the start of vitrification.

3.4.4 Brickmaking

Table 3.11 gives the brickmaking firing range of the different mosaics based upon the combination of the remarks after firing, water absorption and firing shrinkage firing ranges given in Tables 3.2 to 3.7 and Tables 3.9 to 3.10. Clamp kiln firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable. Ceramic kiln firing ranges of 20, 40 and 50°C are regarded as short, medium and long, respectively.

Table 3.11 : Brickmaking firing range of mosaics

Sample	Firing range (°C)	Firing range comments	Remarks after firing	Clamp brickmaking comments	Ceramic kiln brickmaking comments
F0	950 - 950	too short	good, <craze	unacceptable	acceptable
F1	950 - 950	too short	good, <craze	unacceptable	acceptable
F3	950 - 950	too short	poor, cracked	unacceptable	unacceptable
F5	900 - 950	very short	good to bad	poor	fair
F10	800 - 950	medium	good,bad(950)	fair	good
F18	-	unacceptable	-	unacceptable	unacceptable

The overall brickmaking comments given in Table 3.11 become the final comments because the brickmaking comments on the different mosaics were all good based upon the forming properties given in Table 3.10.

All the samples except F5 and F10 are unacceptable clamp brickmaking materials from the firing range and, the forming and firing properties points of view. Samples F5 and F10 are poor and fair clamp brickmaking materials, respectively, from the firing range and, the forming and firing properties points of view.

All the samples except F3 and F18 are acceptable ceramic kiln brickmaking materials from the firing range and, the forming and firing properties points of view. Sample F10 is good in this respect.

The colours of the fired mosaics should be acceptable for bricks.

The problems with the mosaics are their short firing range and, the crazing and cracking.

It is recommended that attempts be made to increase the firing range and eliminate the crazing and the cracking of the mosaics.

3.4.5 Tilemaking

SABS 1449-1989 (4) classifies floor tiles in accordance with their method of manufacture and their water absorption properties, as shown in Table 3.12. Pressing is the most popular tilemaking forming method in this country. The water absorption test for tiles is a 2 hour boil and not 5 hours as has been done for bricks, however, except for tiles with less than 6% water absorption, we assume this should make very little difference.

Table 3.12 : Classification of ceramic floor tiles

Method of manufacture	Tile group			
	Water absorption E (%)			
	$E \leq 3$	$3 < E \leq 6$	$6 < E \leq 10$	$E > 10$
Extruded (Group A) Dust-pressed (Group B)	AI BI	AII BII	AIII BIII	AIV BIV*

* Glazed tiles only

The maximum deviation of dimensions of BI to BIII floor tiles having an area greater than 9000 mm^2 is $\pm$ 0,5 % and for BIV tiles $\pm$ 0,3 %.

SABS 22-1975 (5) requires that the water absorption of glazed wall tiles shall not exceed 20%. The dimensions also shall not differ by more than 0,5 mm from stated sizes. For 150 mm square tiles this represents 0,3%.

According to the above water absorption specifications all mosaics fired up to and including 950°C are classified as BIV floor tiles and would have to be glazed. All the mosaics fired at 1000°C would be classified as BIII floor tiles. For glazed wall tiles the left hand limit of water absorption of the second column of Table 3.9 would apply.

The above mentioned dimensional tolerances are quite stringent but it is believed that the two compositions F5 and F10, if fired in a ceramic kiln where the temperature can be controlled accurately, could possibly meet these floor tile requirements.

The colours of the fired mosaics should be acceptable for tiles.

Considering all the above and, the crazing and cracking it is concluded that mosaics F5 and F10 would make fair glazed wall tile materials and it is recommended that glazed trials be undertaken.

It is recommended that pressing of larger tiles of successful compositions be undertaken.

3.5 CONCLUSIONS

a) None of the samples are sensitive to drying and no precautions have to be taken in practice to ensure that mosaics made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

b) All the samples, except F5 and F10, are unacceptable clamp brickmaking materials from the firing range and, the forming and firing properties points of view. Samples F5 and F10 are poor and fair, respectively, clamp brickmaking materials from the firing range and, the forming and firing properties points of view.

c) All the samples except F3 and F18 are acceptable ceramic kiln brickmaking materials from the firing range and, the forming and firing properties points of view. Sample F10 is good in this respect.

d) All mosaics fired up to and including 950°C are classified as BIV floor tiles and would have to be glazed. All the mosaics fired at 1000°C would be classified as BIII floor tiles. It is believed that the two compositions F5 and F10 if fired in a ceramic kiln where the temperature can be controlled accurately could possibly meet the quite stringent dimensional tolerances requirements for floor tiles.

e) It is also concluded that mosaics F5 and F10 would make fair glazed wall tile materials.

f) The colours of the fired mosaics should be acceptable for bricks or tiles.

g) The problems with the mosaics are their short firing range and, the crazing and cracking.

3.6 RECOMMENDATIONS

a) It is recommended that pressing of larger tiles of successful compositions be undertaken.

b) It is recommended that attempts be made to increase the firing range of the mosaics.

c) It is recommended that attempts be made to eliminate the crazing and cracking of the mosaics.

d) It is recommended that glaze trials be undertaken on the successful compositions.

4. EFFECT OF CORING ON 150 MM PRESSED TILES

4.1 SCOPE

To determine the effects of water and pressure used for pressing, and coring on the fired properties of floor and wall tiles. (See Section 4.2.2 for explanation of coring). To implement Recommendation 3.6 a) i.e. that the pressing of larger tiles of successful compositions be undertaken.

4.2 EXPERIMENTAL WORK

4.2.1 Sample preparation

For the preparation of tiles the dried material was first hammer milled until all material passed through a 1 mm sieve. It was then mixed by hand with 3, 5, and 7% water. The dimension of the tile die was 150 mm square. The difference in the floor and wall tiles is the amount material pressed, resulting in thick and thin tiles, respectively. The tiles were then dried in air at room temperature for at least one week followed by drying overnight at 110°C.

4.2.2 Firing

The dried tiles were fired in a laboratory electric furnace. In the first series a tile of each water content and pressure was fired at 900 and 950°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature. With this firing regime most of the tiles developed firing faults. This difficulty was overcome by introducing a calcination or coring step at 600°C to burn off deflocculants and organic matter. The firing cycle adopted was one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature of 950 or 1000°C.

If a brick shaped from clays containing carbonaceous matter is fired too rapidly, this carbonaceous matter will not be burned out before vitrification begins and the presence of carbon and the consequent reduced state of iron compounds in the centre of the brick results in a black core.

4.3 RESULTS

4.3.1 Fired properties

The results obtained on the fired tiles are given in Tables 4.1 to 4.15, and are graphically presented in Figures 4.1 to 4.12. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F3 means 3% water was added to unfired sludge F on a dry basis.

The dial readings of 350, 400 and 450 kp/cm² correspond to pressures of 26,7 MPa (600 000 N/22 500 mm²), 30,5 MPa (686 000 N/22 500 mm²) and 34,4 MPa (773 000 N/22 500 mm²), respectively.

Table 4.1 : Properties of floor and wall tiles of sample F3 fired at 900°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	4,9	4,4	4,6	4,3	4,3	4,6
Mass loss (%)	19,5	18,9	19,1	19,3	18,7	19,2
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	35,2 37,6 0,94	25,2 26,7 0,94	23,8 25,2 0,95	33,7 34,6 0,97	30,5 31,4 0,97	29,9 30,6 0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped, cracked	crazed, warped	crazed, warped	good, < warp	good	good

Table 4.2 : Properties of floor and wall tiles of sample F3 fired at 950°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	8,5	8,5	8,3	7,4	8,0	8,2
Mass loss (%)	19,7	19,3	19,6	20,0	19,1	20,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	25,7 27,0 0,95	24,4 25,7 0,95	20,1 21,3 0,94	28,2 28,6 0,99	24,7 25,2 0,98	23,5 24,0 0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped, cored	crazed, warped	crazed, warped	good	good, warped	good, warped

Table 4.3 : **Properties of floor and wall tiles of sample F3 cored at 600°C and fired at 950°C and pressed at different pressures**

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	7,7	7,0	5,7	8,3	8,3	8,3
Mass loss (%)	20,1	22,4	19,5	19,8	20,5	21,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	28,4 29,4 0,97	27,7 28,8 0,96	24,7 25,4 0,97	27,1 27,3 0,99	24,8 25,7 0,97	23,6 23,9 0,99
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	good	bad, cracked	bad, cracked	fair, cracked	fair, cracked, warped	good, warped

Table 4.4 : **Properties of floor and wall tiles of sample F3 cored at 600°C and fired at 1000°C and pressed at different pressures**

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	13,8	12,9	13,7	13,1	13,2	14,3
Mass loss (%)	20,0	19,4	19,7	19,4	19,9	19,7
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	17,5 19,1 0,91	16,6 16,6 1,00	12,8 13,7 0,94	17,4 18,0 0,97	14,6 15,5 0,94	13,0 13,5 0,96
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	fair, cracked	bad, cracked	good	good, edge loss	fair, cracked, warped	fair, broke, warped

Table 4.5 : **Properties of floor and wall tiles of sample F3 cored at 600°C and fired at 1050°C and pressed at different pressures**

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	17,6	16,8	16,7	16,3	16,3	16,9
Mass loss (%)	21,1	20,4	20,1	20,4	21,4	21,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	9,5 11,1 0,85	7,9 8,8 0,89	5,6 6,3 0,89	8,5 9,4 0,91	7,9 8,7 0,91	5,3 5,8 0,90
Colour	dark brown	dark brown	dark brown	dark brown	dark brown	dark brown
Remarks after firing	fair, cracked	good, cracked	good	good, cracked	fair, cracked	fair, broke

Table 4.6 : **Properties of floor and wall tiles of sample F5 fired at 900°C and pressed at different pressures**

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	4,9	4,4	4,3	4,7	-	3,7
Mass loss (%)	19,2	19,2	19,3	19,3	25,7	19,3
24hr cold absorption(%) 5hr boil absorption(%) Saturation coefficient	36,5 38,6 0,95	27,8 29,4 0,95	26,5 28,1 0,94	37,7 38,8 0,97	31,0 31,9 0,97	30,4 31,0 0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped, < coring	crazed, warped	crazed, warped	good, < warp	good, < warp	good, < warp

Table 4.7 : Properties of floor and wall tiles of sample F5 fired at 950°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	8,2	6,8	7,7	7,7	7,6	8,0
Mass loss (%)	19,9	19,6	19,9	20,0	19,6	20,0
24hr cold absorption (%)	23,7	19,2	20,9	31,6	25,8	24,0
5hr boil absorption(%)	24,9	20,7	22,2	32,5	26,4	24,4
Saturation coefficient	0,95	0,93	0,94	0,97	0,98	0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped	crazed, warped	crazed, warped	good, <warp	good, warp	good, warp

Table 4.8 : Properties of floor and wall tiles of sample F5 cored at 600°C and fired at 950°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	7,7	7,4	7,8	8,2	7,7	8,5
Mass loss (%)	20,0	20,1	20,1	20,1	21,4	20,5
24hr cold absorption (%)	27,2	24,7	24,2	31,2	25,4	23,9
5hr boil absorption (%)	28,3	25,6	24,9	31,4	26,0	24,4
Saturation coefficient	0,96	0,97	0,97	0,99	0,98	0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	fair, edge loss	poor, cracked	bad, cracked	fair, edge loss, <warp	fair, warped	fair, cracked, warped

Table 4.9 : Properties of floor and wall tiles of sample F5 cored at 600°C and fired at 1000°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	13,3	13,2	13,3	13,7	13,3	13,4
Mass loss (%)	19,8	19,6	19,6	19,9	19,8	19,9
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	15,5 16,8 0,93	14,1 15,0 0,94	12,3 13,1 0,94	19,7 20,6 0,96	14,8 15,5 0,96	13,7 14,0 0,97
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	fair, edge loss	good, <cracks	good, <cracks, edge loss	good, <warp	good, warped, edge loss	good, warped, edge loss

Table 4.10 : Properties of floor and wall tiles of sample F5 cored at 600°C and fired at 1050°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	17,3	16,9	17,0	17,6	17,0	17,1
Mass loss (%)	20,8	22,3	20,8	21,3	21,2	21,0
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	11,0 12,1 0,91	7,0 8,0 0,87	6,0 6,8 0,89	11,5 12,5 0,92	7,2 7,8 0,93	6,2 6,8 0,91
Colour	dark brown	dark brown	dark brown	dark brown	dark brown	dark brown
Remarks after firing	fair, cracked	bad, cracked	bad, cracked	good, bad edge	fair, <cracks	poor, cracks

Table 4.11 : Properties of floor and wall tiles of sample F7 fired at 900°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	4,3	4,6	4,3	4,3	4,4	4,8
Mass loss (%)	19,3	19,2	19,2	19,3	19,4	19,2
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	28,7 29,9 0,96	27,1 28,5 0,95	24,8 26,4 0,94	30,7 31,3 0,98	31,8 32,4 0,98	30,5 31,0 0,98
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped	<coring , warped, crazed	warped, crazed	good, <warp	good, <warp	good, <warp

Table 4.12 : Properties of floor and wall tiles of sample F7 fired at 950°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	8,4	7,6	7,8	7,5	7,7	7,9
Mass loss (%)	19,6	19,8	19,7	19,7	19,8	20,2
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	23,3 24,8 0,94	21,4 22,9 0,93	20,4 21,6 0,94	27,4 27,9 0,98	27,0 27,4 0,98	24,1 24,8 0,97
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	crazed, warped	crazed, warped, <coring	warped, crazed, coring	good, warped	good, warped	good, warped

Table 4.13 : Properties of floor and wall tiles of sample F7 cored at 600°C and fired at 950°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	7,6	7,5	7,6	8,1	7,7	8,1
Mass loss (%)	19,3	20,1	19,3	19,5	20,3	20,2
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	26,1 26,9 0,97	25,2 25,9 0,97	24,0 24,6 0,97	26,0 26,2 0,99	26,5 26,8 0,99	23,9 24,2 0,99
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	poor, cracked	bad, cracked	bad, cracked	good, warped	bad, cracked, warped	good, warped

Table 4.14 : Properties of floor and wall tiles of sample F7 cored at 600°C and fired at 1000°C and pressed at different pressures

PROPERTY	PRESSURE (kp/cm^2)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	13,6	13,8	13,6	13,6	13,6	13,5
Mass loss (%)	19,4	19,7	19,4	18,8	20,8	19,6
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	14,2 15,2 0,94	13,6 14,1 0,96	11,9 12,6 0,95	15,3 16,0 0,95	14,9 15,3 0,97	13,7 14,2 0,97
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	good, <warp	bad, cracked	good	good, edge loss, warped	good, edge loss, <crack, warped	good, edge loss, <crack, warped

Table 4.15 : **Properties of floor and wall tiles of sample F7 cored at 600°C and fired at 1050°C and pressed at different pressures**

PROPERTY	PRESSURE (kp/cm²)					
	Floor tiles			Wall tiles		
	350	400	450	350	400	450
Firing shrinkage (%)	17,5	17,6	16,9	17,7	17,5	17,3
Mass loss (%)	20,0	20,1	20,0	20,1	20,9	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	6,5 7,3 0,89	6,5 7,3 0,90	5,7 6,4 0,90	6,6 7,0 0,94	10,4 11,1 0,94	5,1 5,6 0,90
Colour	dark brown	dark brown	dark brown	dark brown	dark brown	dark brown
Remarks after firing	fair, <cracks	bad, cracked	good	good, <edge loss	fair, cracked	bad, cracked

Figure 4.1 : Water absorption vs firing temp. of F3 floor and wall tiles

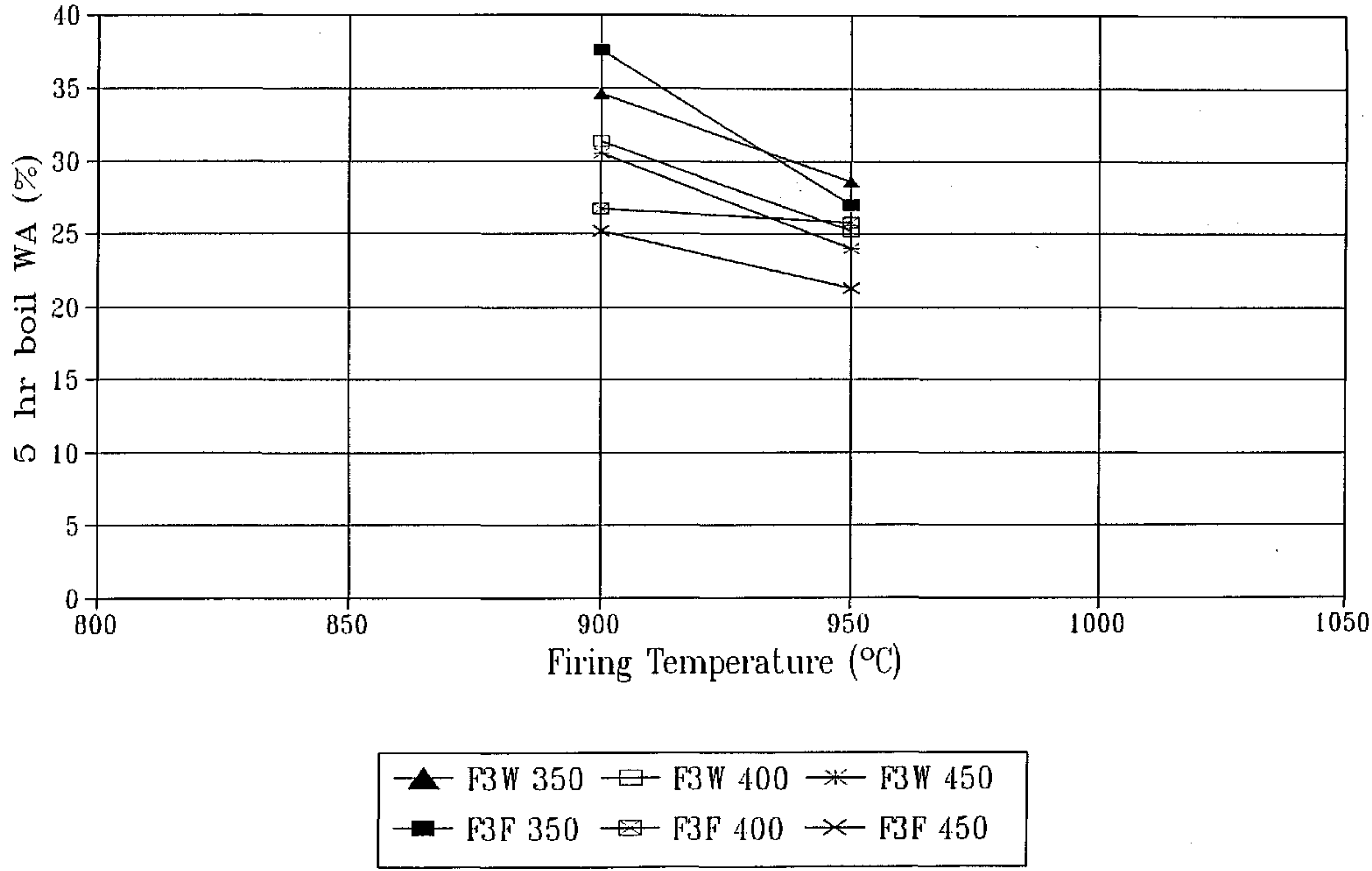


Figure 4.2 : Water absorption vs firing temp. of F3 cored floor and wall tiles

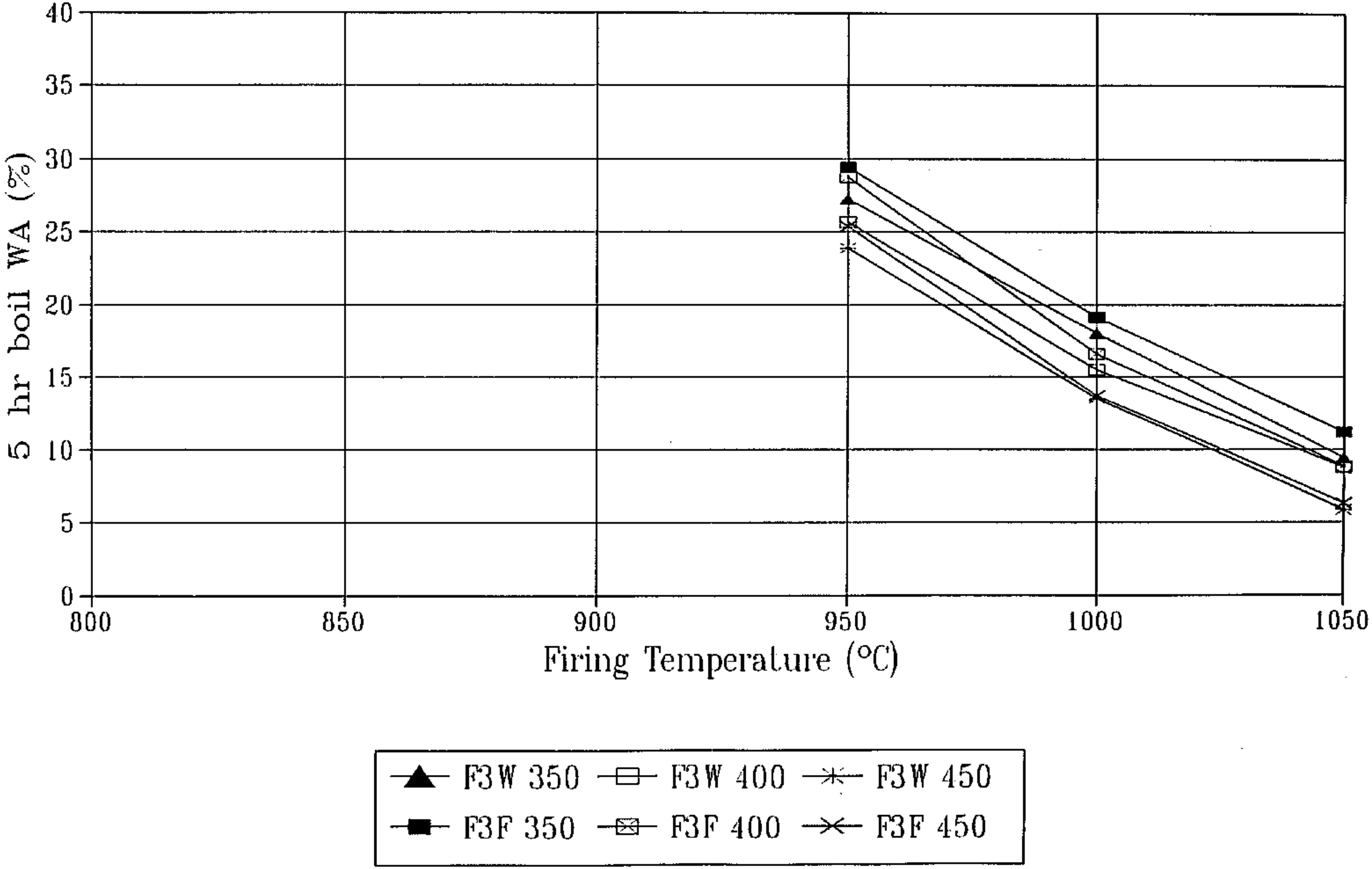


Figure 4.3 : Water absorption vs firing temp. of F5 floor and wall tiles

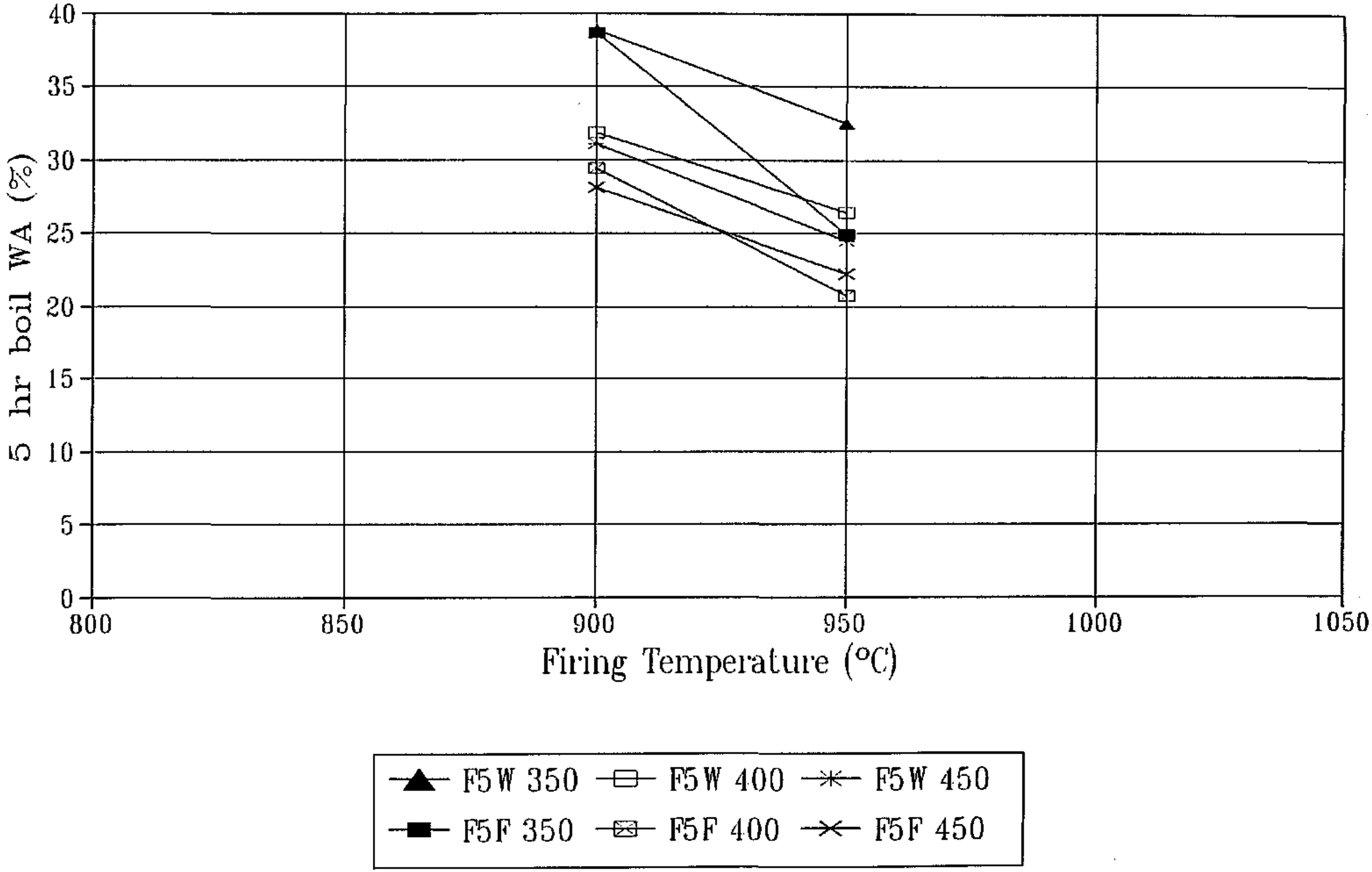


Figure 4.4: Water absorption vs firing temp. of F5 cored floor and wall tiles

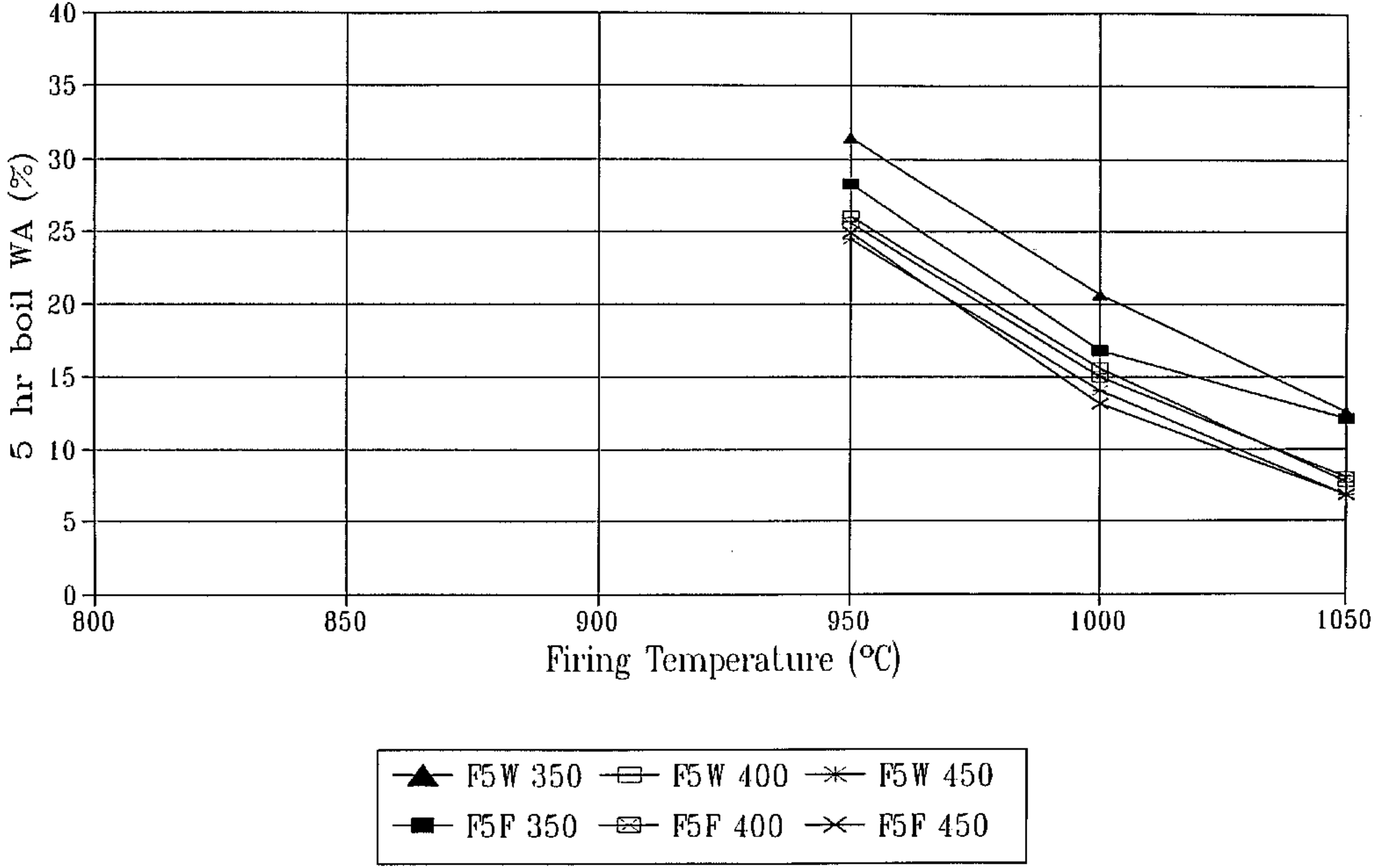


Figure 4.5 : Water absorption vs firing temp. of F7 floor and wall tiles

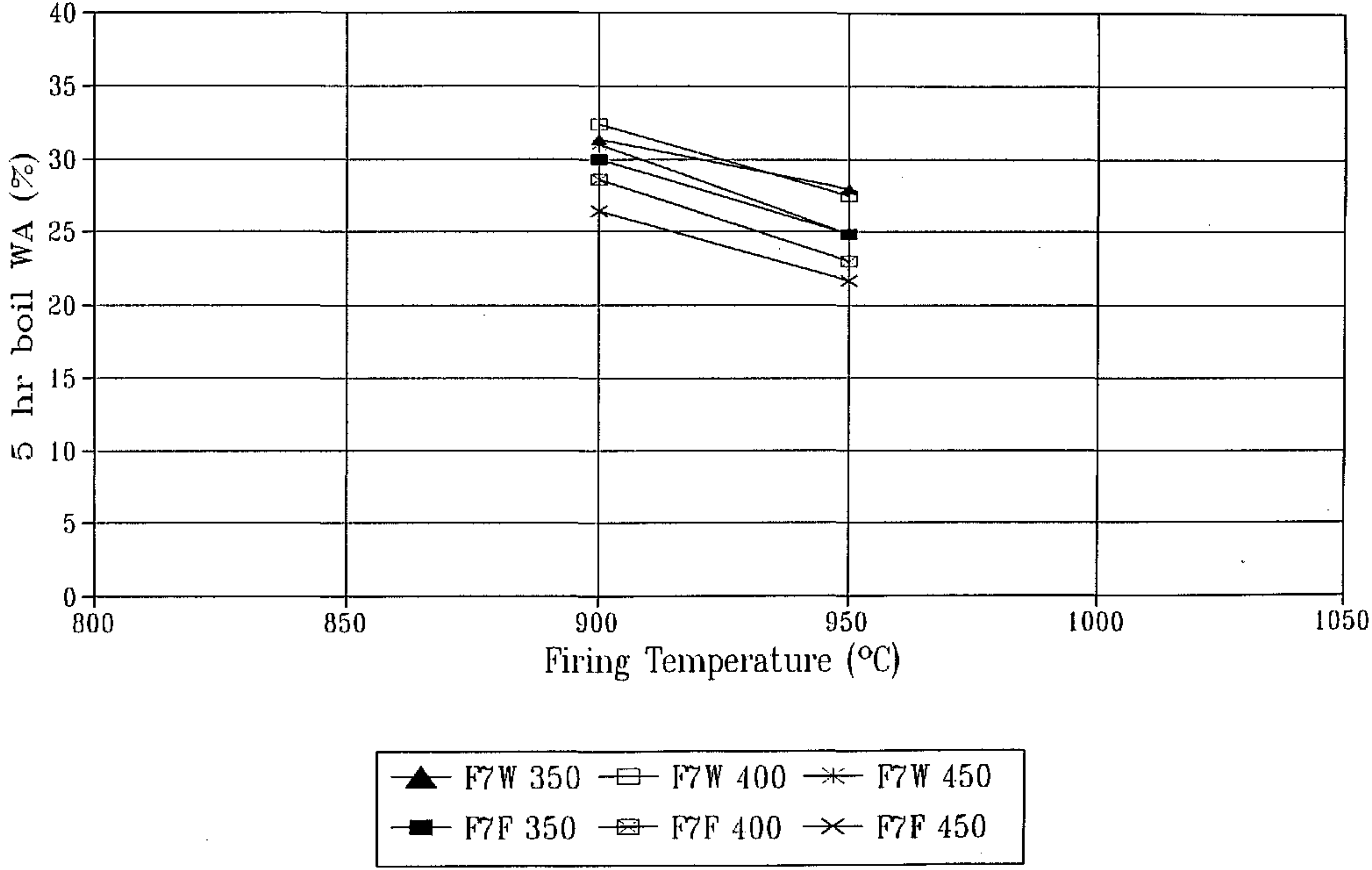


Figure 4.6 : Water absorption vs firing temp. of F7 cored floor and wall tiles

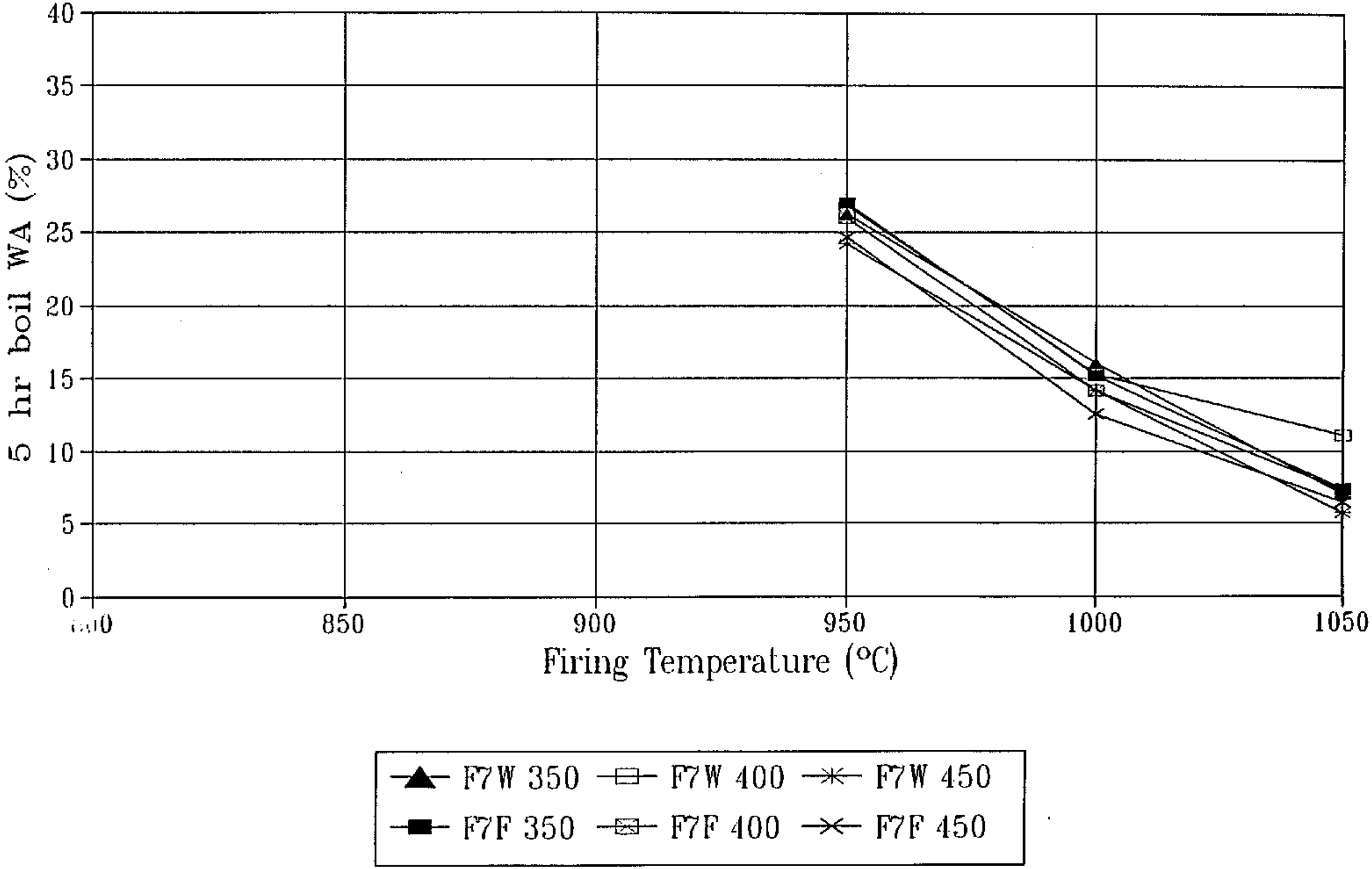


Figure 4.7: Firing shrink. vs firing temp. of E3 floor and wall tiles

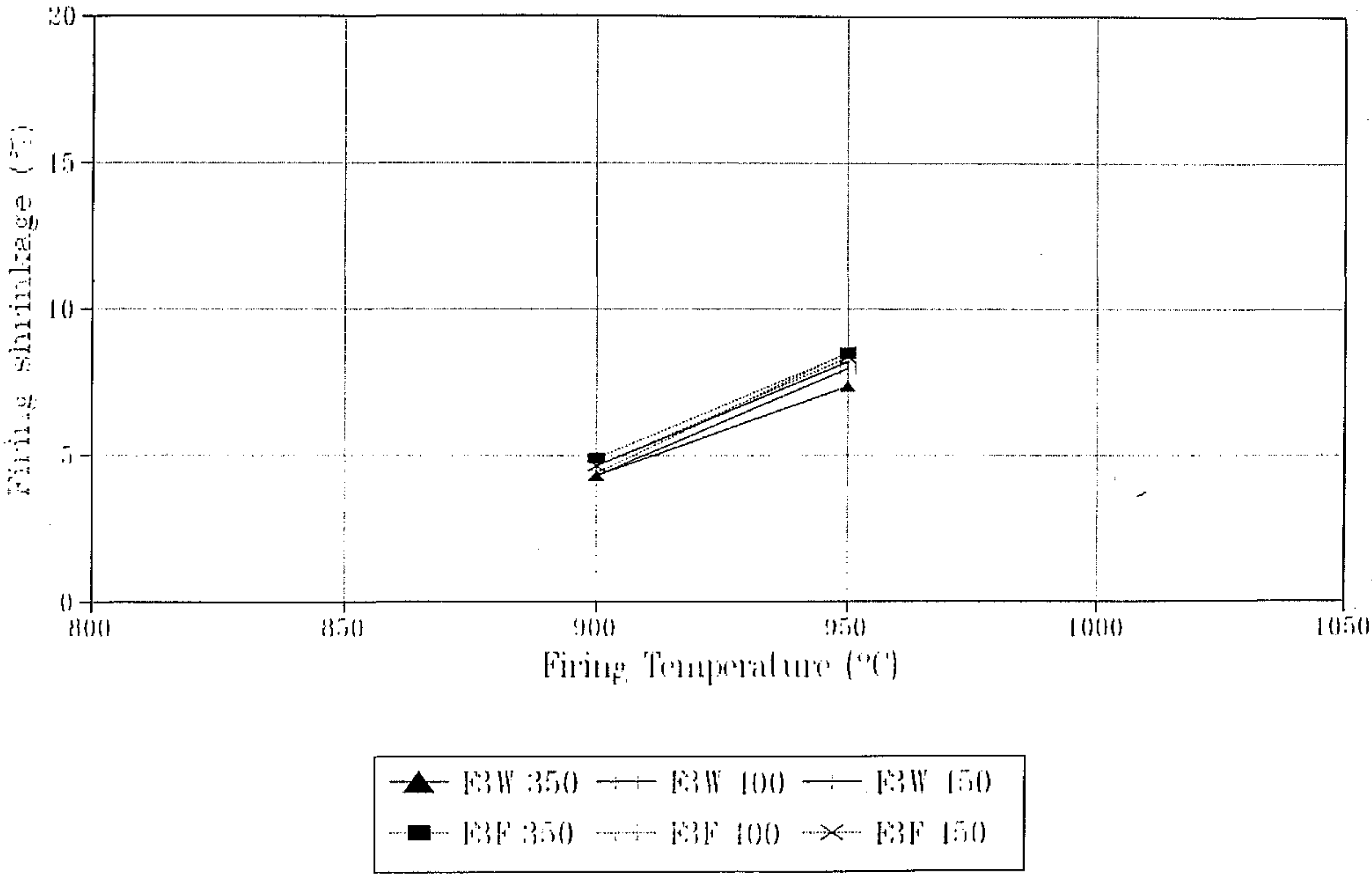


Figure 4.8: Firing shrink. vs firing temp. of F3 cored floor and wall tiles

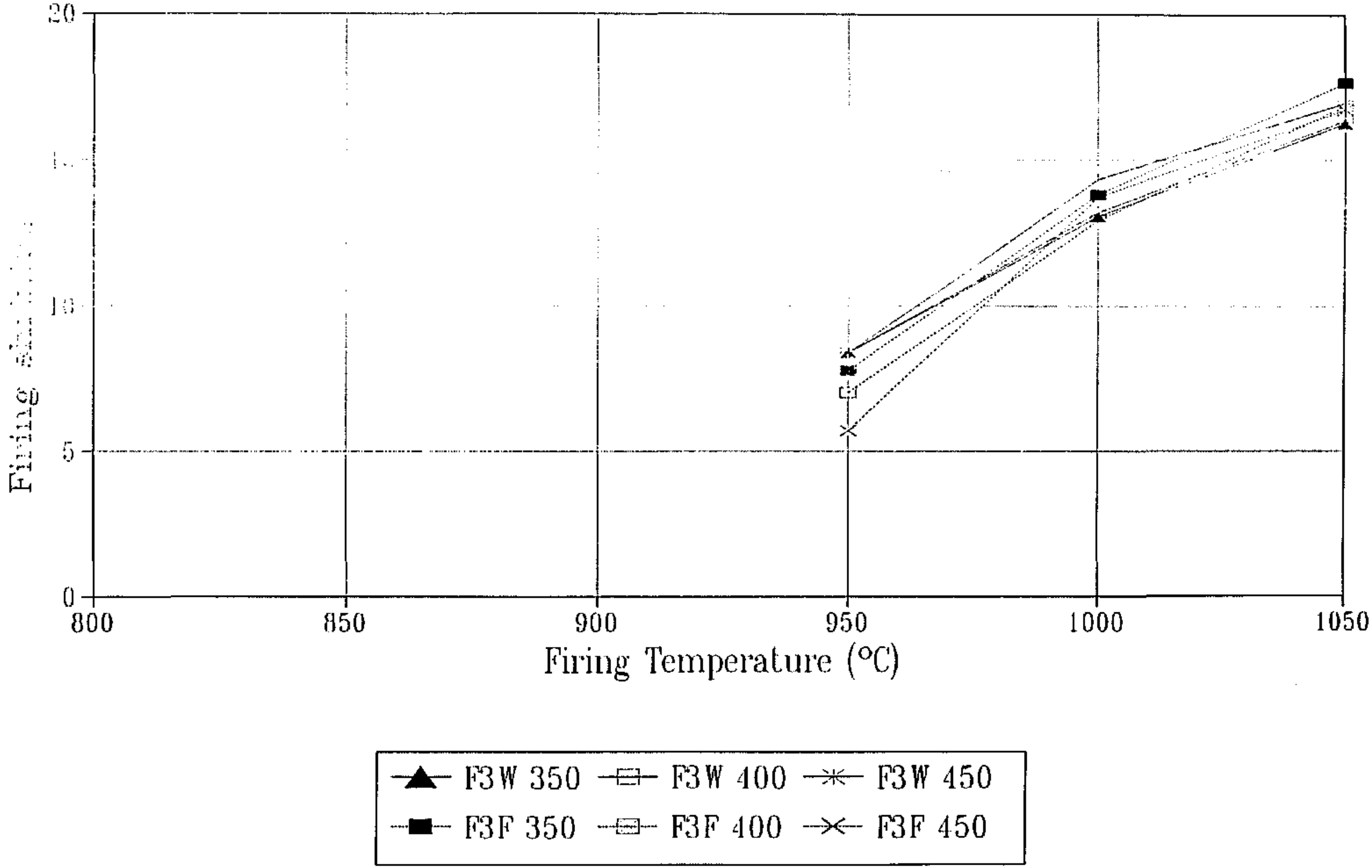


Figure 4.9: Firing shrink. vs firing temp. of F5 floor and wall tiles

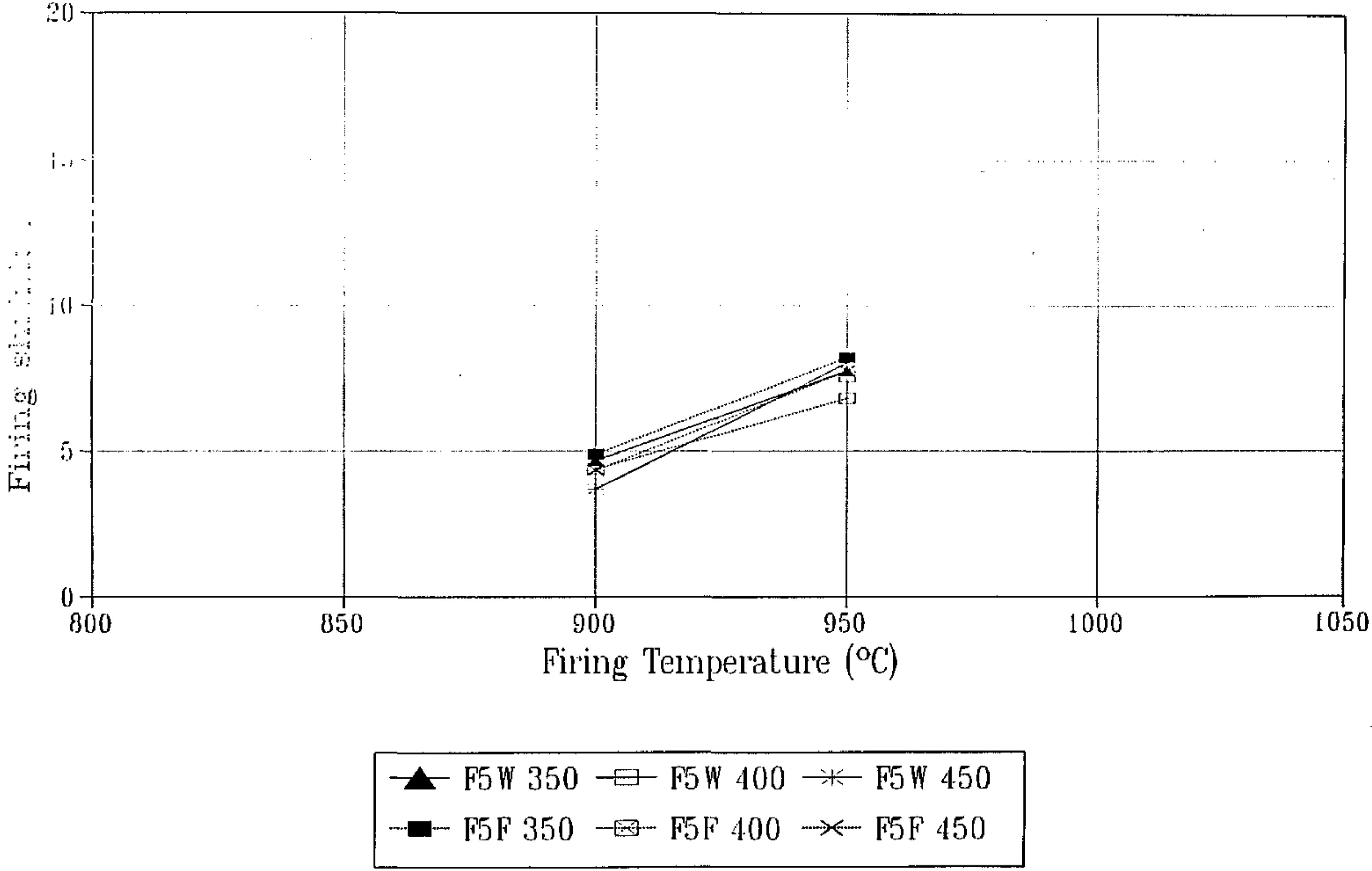


Figure 4.10: Firing shrink. vs firing temp. of F5 cored floor and wall tiles

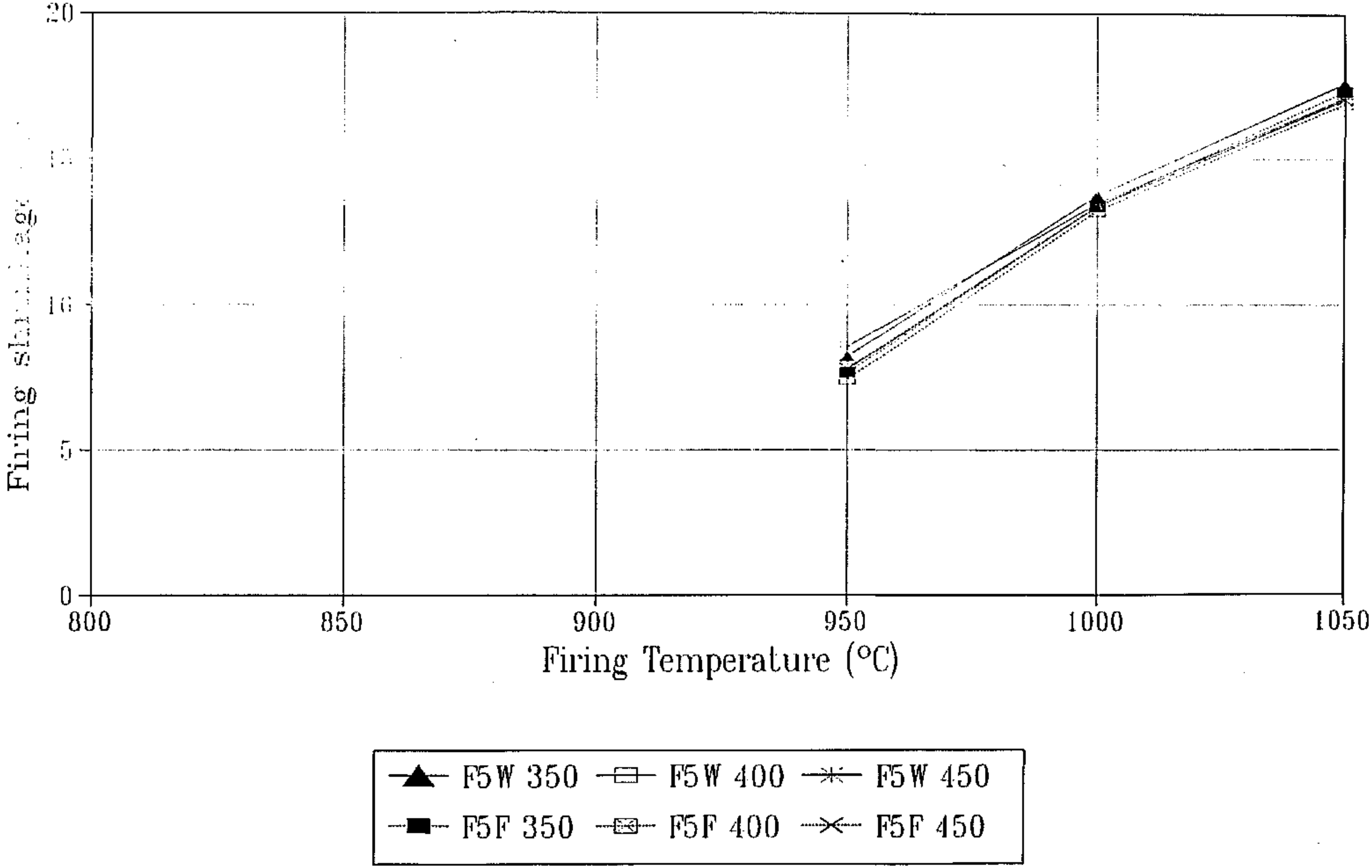


Figure 4.11: Firing shrink. vs firing temp. of F7 floor and wall tiles

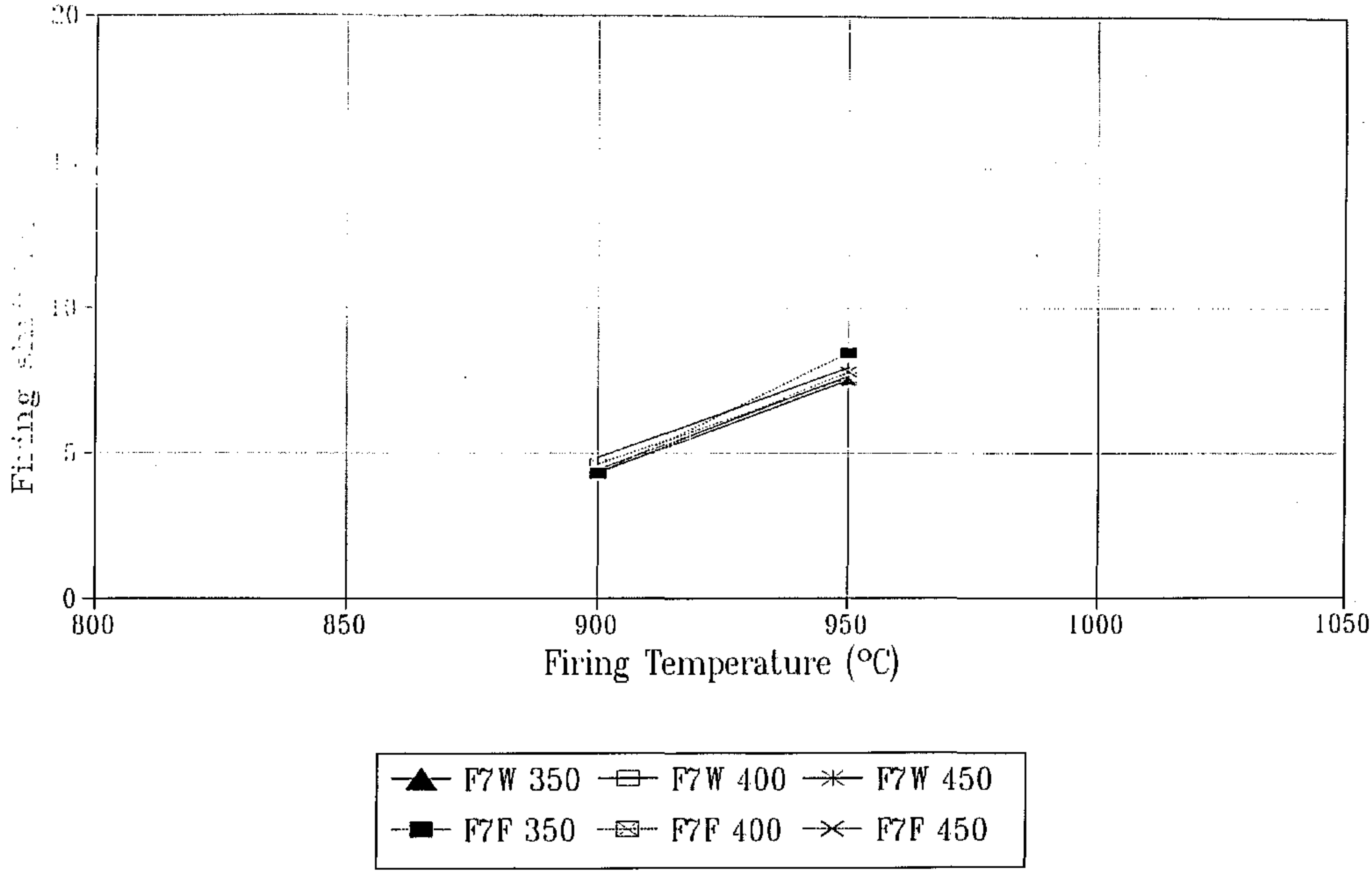
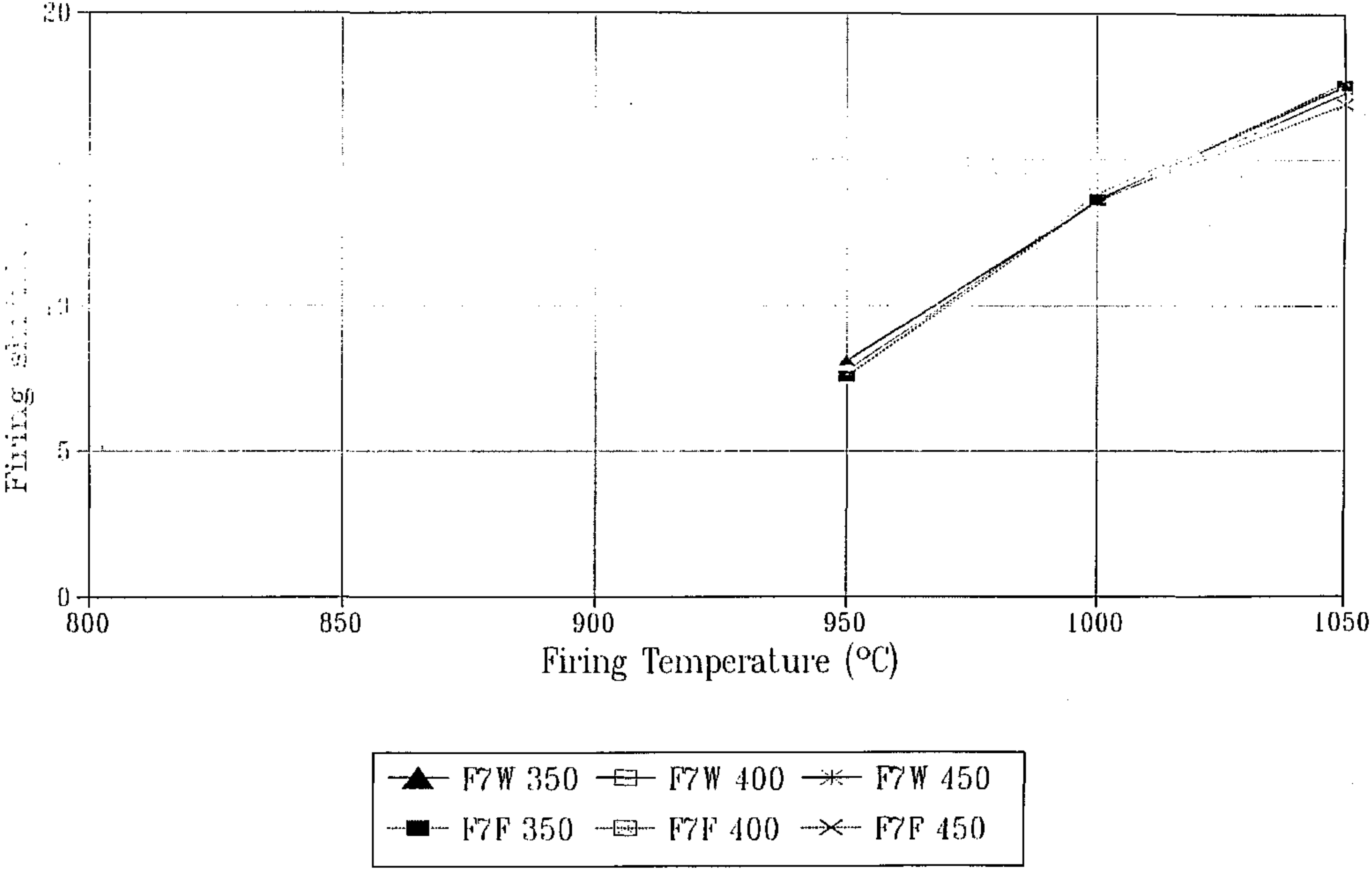


Figure 4.12: Firing shrink. vs firing temp. of F7 cored floor and wall tiles



4.4 DISCUSSION OF RESULTS

4.4.1 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks. From Figures 4.1 to 4.6 all samples fired at 1000°C have good water absorptions except sample F5W 350.

All the uncored tiles which were fired at 900 to 950°C were above 20% water absorption. Table 4.16 gives the firing range of the cored tiles based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 4.1 to 4.15 and Figures 4.1 to 4.6.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable.

Table 4.16 : Water absorption firing range of cored tiles

Sample	5hr WA range (~20≥WA≥~8) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
F3F 350	19 - 11	1000 - 1050	very short	poor
F3F 400	17 - 9	1000 - 1050	very short	poor
F3F 450	14	1000 - 1000+	too short	bad
F3W 350	18 - 9	1000 - 1050	very short	poor
F3W 400	16 - 9	1000 - 1050	very short	poor
F3W 450	14	1000 - 1000+	too short	bad
F5F 350	17 - 12	1000 - 1050	very short	poor
F5F 400	15 - 8	1000 - 1050	very short	poor
F5F 450	13	1000 - 1000+	too short	bad
F5W 350	21 - 12	1000 - 1050	very short	poor
F5W 400	16 - 8	1000 - 1050	very short	poor
F5W 450	14	1000 - 1000+	too short	bad
F7F 350	15	1000 - 1000+	too short	bad
F7F 400	14	1000 - 1000+	too short	bad
F7F 450	13	1000 - 1000+	too short	bad
F7W 350	16	1000 - 1000+	too short	bad
F7W 400	15 - 11	1000 - 1050	very short	poor
F7W 450	14	1000 - 1000+	too short	bad

All the samples would make poor or bad clamp brickmaking materials from the water absorption firing range point of view. All the poor and bad clamp brickmaking samples would make acceptable and poor, respectively, kiln brickmaking materials from the water absorption firing range point of view. All the F3 and F5 samples except the 450 kp/cm^2 pressing, and only the F7W 400 sample would make acceptable kiln brickmaking materials from the point of view of water absorption firing range.

4.4.2 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively.

Table 4.17 gives the firing range of the different series of tiles based upon an acceptable firing shrinkage range as described above. This Table is derived from the data given in Tables 4.1 to 4.15 and Figures 4.7 to 4.12.

Table 4.17 : Firing shrinkage firing range of tiles

Sample	Firing shrinkage (0 ≤ FS ≤ 7) (%)	Firing range (°C)	Firing range comments	Brickmaking comments
F3F 350	5	Up to 900+	medium*	good
F3F 400	4 - 7	Up to 950	medium	good
F3F 450	5	Up to 900+	medium*	good
F3W 350	4	Up to 900+	medium*	good
F3W 400	4	Up to 900+	medium*	good
F3W 450	5	Up to 900+	medium*	good
F3F 400 cored	7	Up to 950	medium	good
F5F 350	5	Up to 900+	medium*	good
F5F 400	4 - 7	Up to 950	medium	good
F5F 450	4	Up to 900+	medium*	good
F5W 350	5	Up to 900+	medium*	good
F5W 400	-	Up to 900+	medium*	good
F5W 450	4	Up to 900+	medium*	good
F5F 400 cored	7	Up to 950	medium	good
F7F 350	4	Up to 900+	medium*	good
F7F 400	5	Up to 900+	medium*	good
F7F 450	4	Up to 900+	medium*	good
F7W 350	4	Up to 900+	medium*	good
F7W 400	4	Up to 900+	medium*	good
F7W 450	5	Up to 900+	medium*	good

* Estimated

All the uncored tiles fired at 900°C would make good brickmaking materials from the firing shrinkage point of view. The F3 400 and F5 400 cored and uncored floor tiles fired at 950°C would also make good brickmaking materials from the firing shrinkage point of view. It is expected that cored tiles fired at 900°C would also be acceptable. Above 950°C high shrinkage corresponding to the start of vitrification begins.

4.4.3 Brickmaking

Based upon the combination of the water absorption and firing shrinkage firing ranges given in Tables 4.16 and 4.17 there is no common brickmaking firing range for the different series of tiles. All the samples are unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

The colours of the fired tiles should be acceptable for bricks.

It is recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

4.4.4 Tilemaking

According to the water absorption floor tile requirements given in Section 3.4.5 all the floor tiles fired below 1050°C would be classified as BIV floor tiles and would have to be glazed.

According to the water absorption glaze wall tile requirements given in Section 3.4.5 none of the fired uncored wall tiles are acceptable but all the cored wall tiles fired above 950°C are acceptable except the F5W 350 fired at 1000°C.

The dimensional tolerances given in Section 3.4.5 are quite stringent and it is believed that only the tiles meeting the brickmaking firing shrinkage range given in Table 4.17, if fired in a ceramic kiln where the temperature can be controlled accurately, could possibly meet these tile requirements.

In Table 4.18 the tiles which had good remarks and only slight defects after firing from Tables 4.1 to 4.15 are given. It is to be noted that of the floor tiles only the cored ones have good remarks. Of the common tiles fired at 950°C, of the uncored 17 are warped whereas 8 only are warped when cored. Overall it is seen that coring does have a beneficial effect in decreasing the warping of the tiles, however, it does not have a great effect on water absorption and firing shrinkage.

Table 4.18 : Tiles which had good remarks after firing

Sample	Firing temperature (°C)
F3F 350	950 C*
F3F 400	-
F3F 450	1000 C, 1050 C
F3W 350	900, 950, 1000 C
F3W 400	900
F3W 450	900
F5F 350	-
F5F 400	1000 C
F5F 450	1000 C
F5W 350	900, 950, 1000 C, 1050 C
F5W 400	900
F5W 450	900
F7F 350	1000 C
F7F 400	-
F7F 450	1000 C, 1050 C
F7W 350	900, 1050 C
F7W 400	900
F7W 450	900

C* = Cored

The colours of the fired tiles should be acceptable for tiles.

Considering all the above it is concluded that the cored tiles may make fair glazed floor and wall tile materials and it is recommended that glazing trials be undertaken on the successful compositions.

It is recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

Though the tiles do not at this stage meet the specified requirements for unglazed tiles it is believed that it may be possible to produce a lower grade, such as quarry or rustic tiles, from these materials.

We conclude that the sludge may be suitable for producing rustic tiles.

4.5 CONCLUSIONS

a) From the point of view of a common firing range all the samples are unacceptable clamp and kiln brickmaking materials.

b) Coring does have a beneficial effect in decreasing the warping of the tiles, however, it does not have a great effect on water absorption and firing shrinkage.

c) The colours of the fired tiles should be acceptable for bricks or tiles.

d) The cored tiles may make fair glazed floor and wall tile materials.

e) We conclude that the sludge may be suitable for producing rustic tiles.

4.6 RECOMMENDATIONS

a) It is recommended that attempts be made to obtain a common water absorption and firing range for the bricks and tiles.

b) It is recommended that glazing trials be undertaken on the successful compositions.

5. EFFECT OF ADDITIONS ON 150 MM PRESSED TILES

5.1 SCOPE

To determine the effects of additions, such as calcines and fluxes on the fired properties of floor tiles. To implement Recommendation 4.6 a) i.e. that attempts be made to obtain a common water absorption and firing range for the bricks and tiles.

5.2 EXPERIMENTAL WORK

5.2.1 Sample preparation

For the preparation of tiles the dried sludge and calcines were first hammer milled until all material passed through a 1 mm sieve. The calcines used were 1000 and 1050°C sludge calcines. The fluxes used were ground brown bottle glass (cullet), soda ash (Na_2CO_3) and water glass. The different samples were mixed dry by hand with the required percentage by mass of additives. They were then mixed by hand with 10% water. The tiles were then pressed at a pressure of 300 kp/cm^2 corresponding to 22,8 MPa (513 000N/22 500 mm^2). The dimension of the tile die was 150 mm square. The tiles were then dried in air at room temperature for at least one week followed by drying overnight at 110°C.

5.2.2 Firing

The dried tiles were fired in a laboratory electric furnace. A coring cycle (see Section 4.2.2) was adopted of one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature or left to cool. A tile of each composition was fired at most of the following temperatures: 600, 900, 920, 930, 950 and 1000°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

5.3 RESULTS

5.3.1 Forming properties

The results obtained on the pressed tiles are given in Tables 5.1 to 5.4. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F10 means 10% water was added to unfired sludge F on a dry basis.

Table 5.1 : Summary of forming properties of tiles F10 1000°C calcines

FORMING PROPERTY	COMPOSITION			
	90S10C*	80S20C	70S30C	50S50C
Pressing	good	good	good	good
Mixing water (%)	11,4	11,9	11,8	11,2
Drying shrinkage (%)	1,5	1,9	1,1	0,2
Colour	grey brown	grey brown	grey brown	grey brown

* See Tables 5.5 to 5.23 for sample composition identification

Table 5.2 : Summary of forming properties of tiles F10 1050°C calcines

FORMING PROPERTY	COMPOSITION			
	90S10C	80S20C	70S30C	50S50C
Pressing	good	good	good	good
Mixing water (%)	14,5	13,9	11,4	11,2
Drying shrinkage (%)	1,8	1,6	1,0	0,7
Colour	grey brown	grey brown	grey brown	grey brown

Table 5.3 : Summary of forming properties of tiles F10 1050°C calcines, glass and soda ash

FORMING PROPERTY	COMPOSITION					
	95S5C	90S10C	85S15C	80S10C10G	70S10C20G	80S10C10A
Pressing	good	good	good	good	good	good
Mixing water (%)	11,3	12,1	12,1	14,1	12,3	12,8
Drying shrinkage (%)	1,3	1,4	1,3	1,4	1,1	1,4
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

Table 5.4 : Summary of forming properties of tiles F10 soda ash and waterglass

FORMING PROPERTY	COMPOSITION				
	80S10C10WA	70S10C20WA	89S10C1WG	87S10C3WG	85S10C5WG
Pressing	good	good	good	good	good
Mixing water (%)	11,4	11,7	12,1	12,6	17,6
Drying shrinkage (%)	0,7	0,7	1,3	1,4	2,0
Colour	grey brown	grey brown	grey brown	grey brown	grey brown

5.3.2 Fired properties

The results obtained on the fired tiles are given in Tables 5.5 to 5.23, and are graphically presented in Figures 5.1 to 5.6. In Figures 5.3 and 5.4 the 500°C firing shrinkage values are the drying shrinkage values.

Table 5.5 : Fired properties of cored tiles for sample F10 90% sludge / 10% calcines (1000°C)

PROPERTY	TEMPERATURE (°C)			
	600	930	950	1000
Firing shrinkage (%)	2,7	4,7	7,8	11,3
Mass loss (%)	15,0	1,8*	2,9*	9,9*
24hr cold absorption (%)	34,4	26,8	25,6	14,4
5hr boil absorption (%)	36,1	28,2	27,1	15,9
Saturation coefficient	0,95	0,95	0,95	0,91
Colour	brown	brown	brown	brown
Remarks after firing	good	good	good	poor, crazed

* mass loss from 600°C

Table 5.6 : **Fired properties of cored tiles for sample F10 80% sludge / 20 % calcines (1000°C)**

PROPERTY	TEMPERATURE (°C)			
	600	**930**	**950**	**1000**
Firing shrinkage (%)	2,9	3,2	5,6	5,8
Mass loss (%)	13,8	1,7*	2,5*	18,0
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	33,3 34,9 0,95	26,7 29,1 0,92	26,2 28,3 0,93	18,4 21,3 0,86
Colour	brown	brown with black spots	brown with black spots	brown
Remarks after firing	good	poor, crazed	poor, crazed	bad, crazed

* mass loss from 600°C

Table 5.7 : **Fired properties of cored tiles for sample F10 70% sludge / 30% calcines (1000°C)**

PROPERTY	TEMPERATURE (°C)			
	600	**930**	**950**	**1000**
Firing shrinkage (%)	1,2	2,7	3,2	5,4
Mass loss (%)	11,4	1,7*	2,1*	16,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	30,0 32,0 0,94	25,2 28,3 0,89	25,4 28,5 0,89	19,8 22,6 0,87
Colour	brown	brown	brown with black spots	brown with black spots
Remarks after firing	good	poor, crazed	poor, crazed	bad, crazed

* mass loss from 600°C

Table 5.8 : Fired properties of cored tiles for sample F10 50% sludge / 50% calcines (1000°C)

PROPERTY	TEMPERATURE (°C)			
	600	930	950	1000
Firing shrinkage (%)	0,1	0,9	0,8	1,8
Mass loss (%)	7,7	2,2*	1,4*	8,7
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	26,5 28,5 0,93	24,1 27,6 0,87	24,3 25,9 0,94	20,8 19,1 1,09
Colour	brown	brown	brown	dark brown
Remarks after firing	poor, friable	bad, friable	bad, friable	bad, friable

* mass loss from 600°C

Table 5.9 : Fired properties of cored tiles for sample F10 90% sludge / 10% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	920	1000
Firing shrinkage (%)	2,8	4,7	7,8	12,6
Mass loss (%)	15,7	1,8*	2,9*	25,9
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	33,5 35,2 0,95	29,9 31,2 0,96	28,7 30,3 0,95	14,3 15,5 0,92
Colour	brown	brown	brown	brown
Remarks after firing	good	-	good, <craze	good, <craze

* mass loss from 600°C

Table 5.10 : Fired properties of cored tiles for sample F10
80% sludge / 20% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	920	1000
Firing shrinkage (%)	1,8	3,2	5,6	8,1
Mass loss (%)	13,4	1,7*	2,5*	12,9
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	31,5 33,0 0,95	29,3 31,0 0,95	27,4 30,2 0,91	17,4 19,7 0,88
Colour	brown	brown	brown	brown
Remarks after firing	good	good, <craze	fair, crazed	poor, crazed

* mass loss from 600°C

Table 5.11 : Fired properties of cored tiles for sample F10
70% sludge / 30% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	920	1000
Firing shrinkage (%)	1,4	2,7	3,2	4,7
Mass loss (%)	11,3	1,7*	2,1*	9,6
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	30,8 32,9 0,94	29,4 31,5 0,93	28,5 31,2 0,91	20,0 22,7 0,88
Colour	brown	brown	brown	brown with black spots
Remarks after firing	good	good, <craze	good, <craze	bad, crazed

* mass loss from 600°C

Table 5.12 : Fired properties of cored tiles for sample F10 50% sludge / 50% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	920	1000
Firing shrinkage (%)	0,7	0,9	0,8	1,9
Mass loss (%)	7,8	2,2*	1,4*	9,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	25,1 26,9 0,94	25,2 26,7 0,94	25,3 27,3 0,93	22,0 23,6 0,93
Colour	brown	brown	brown	dark brown
Remarks after firing	poor, friable	poor, friable	poor, friable	bad, friable

* mass loss from 600°C

Table 5.13 : Fired properties of cored tiles for sample F10 95% sludge / 5% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	1,1	3,9	7,2	11,1
Mass loss (%)	17,4	18,0	18,5	18,5
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	33,2 34,0 0,97	26,3 27,7 0,95	15,4 16,7 0,92
Colour	brown	brown	brown	brown
Remarks after firing	good	good	good	good, <craze

Table 5.14 : Fired properties of cored tiles for sample F10 II 90% sludge / 10% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	1,2	3,2	5,7	10,1
Mass loss (%)	16,4	16,9	17,5	17,5
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	32,4 33,7 0,96	26,7 28,7 0,93	16,4 18,4 0,89
Colour	brown	brown	brown	dark brown
Remarks after firing	good	good	cracked, crazed	fair, crazed

Table 5.15 : Fired properties of cored tiles for sample F10 85% sludge / 15% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,9	2,0	5,0	6,9
Mass loss (%)	15,2	15,7	16,2	16,3
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	31,8 33,3 0,96	26,6 28,5 0,93	18,8 21.4 0,88
Colour	brown	brown	brown	brown
Remarks after firing	good	good	poor, cracked	poor, crazed

**Table 5.16 : Fired properties of cored tiles for sample F10
80% sludge / 10% calcines (1050°C) / 10% glass**

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	1,0	4,0	6,9	9,1
Mass loss (%)	14,1	14,6	14,9	15,4
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	26,0 27,8 0,94	19,8 22,1 0,90	14,4 16,9 0,86
Colour	brown	brown	dark brown	dark brown
Remarks after firing	good	good	fair, crazed	poor, crazed

**Table 5.17 : Fired properties of cored tiles for sample F10
70% sludge / 10% calcines (1050°C) / 20% glass**

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,7	4,7	6,1	6,3
Mass loss (%)	12,3	13,3	13,4	15,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	22,8 24,8 0,92	19,0 21,6 0,88	17,7 20,6 0,86
Colour	brown	brown	dark brown	dark brown
Remarks after firing	-	poor	poor, cracked	poor

Table 5.18 : Fired properties of cored tiles for sample F10 80% sludge / 10% calcines (1050°C) / 10% powder soda ash

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,6	3,1	7,1	8,8
Mass loss (%)	18,1	19,1	19,3	19,4
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	20,4 26,2 0,78	16,6 22,1 0,75	11,8 16,3 0,73
Colour	-	dark brown	dark brown	very dark brown
Remarks after firing	-	good	good	very good

Table 5.19 : Fired properties of cored tiles for sample F10 80% sludge / 10% calcines (1050°C) / 10% wet soda ash

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	1,0	3,0	3,6	7,0
Mass loss (%)	18,0	19,9	20,1	20,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	25,9 32,4 0,80	23,4 29,9 0,78	17,0 22,7 0,75
Colour	-	dark brown	dark brown	dark brown
Remarks after firing	good	good	fair	fair

Table 5.20 : Fired properties of cored tiles for sample F10 70% sludge / 10% calcines (1050°C) / 20% wet soda ash

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,7	2,9	3,6	4,5
Mass loss (%)	18,3	23,8	24,0	24,1
24hr cold absorption (%)	-	24,8	21,1	19,5
5hr boil absorption (%)	-	33,2	30,8	27,8
Saturation coefficient	-	0,75	0,69	0,70
Colour	-	dark brown	dark brown	dark brown
Remarks after firing	-	fair, pinholes	fair, cracked, pinholes	fair, pinholes

Table 5.21 : Fired properties of cored tiles for sample F10 89% sludge / 10% calcines (1050°C) / 1% water glass

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,7	3,4	6,0	9,2
Mass loss (%)	15,8	16,5	17,1	17,1
24hr cold absorption (%)	-	31,8	25,7	17,1
5hr boil absorption (%)	-	33,5	27,9	19,5
Saturation coefficient	-	0,95	0,92	0,88
Colour	brown	brown	brown	brown
Remarks after firing	good	good	fair, crazed	fair, crazed

Table 5.22 : Fired properties of cored tiles for sample F10
87% sludge / 10% calcines (1050°C) / 3% water glass

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,8	3,5	7,5	10,2
Mass loss (%)	15,7	16,3	16,7	17,1
24hr cold absorption (%)	-	29,9	21,6	15,5
5hr boil absorption (%)	-	32,0	24,0	17,8
Saturation coefficient	-	0,94	0,90	0,87
Colour	brown	brown	brown	brown
Remarks after firing	good	good	fair, crazed	poor, crazed

Table 5.23 : Fired properties of cored tiles for sample F10
85% sludge / 10% calcines (1050°C) / 5% water glass

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	0,6	4,0	7,0	10,2
Mass loss (%)	15,4	16,2	16,8	17,2
24hr cold absorption (%)	-	26,5	21,1	14,2
5hr boil absorption (%)	-	28,4	24,0	16,7
Saturation coefficient	-	0,93	0,88	0,85
Colour	brown	brown	brown	dark brown
Remarks after firing	good	good	fair, crazed	poor, crazed

Figure 5.1 : Water abs. vs firing temp.
F10 tiles fired 600/24hrs 1000 calcines

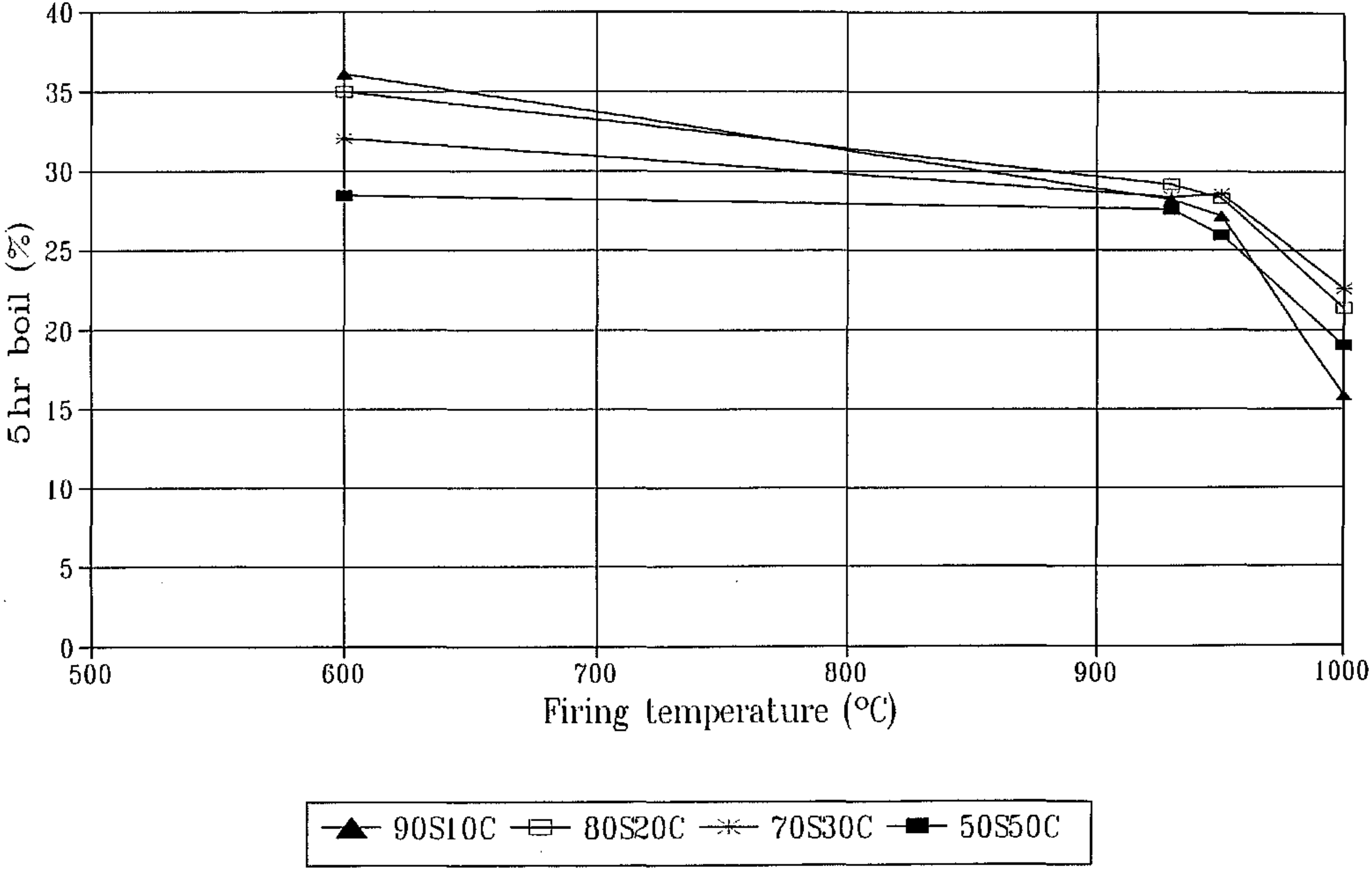


Figure 5.2 : Water abs. vs firing temp.
F10 tiles fired 600/24hrs 1050 calcines

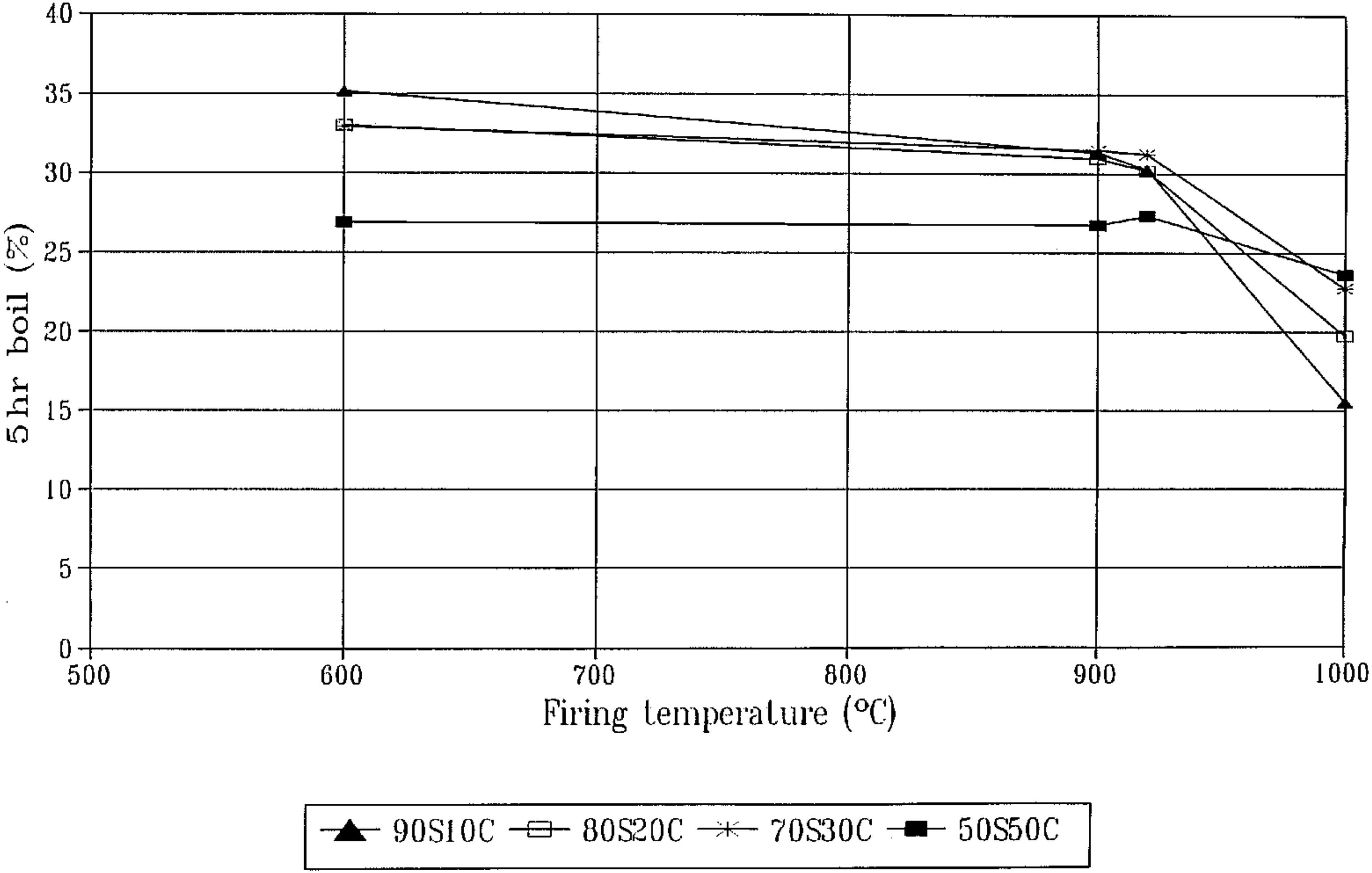


Figure 5.3 : Shrinkage vs firing temp.
F10 tiles fired 600/24hrs 1000 calcines

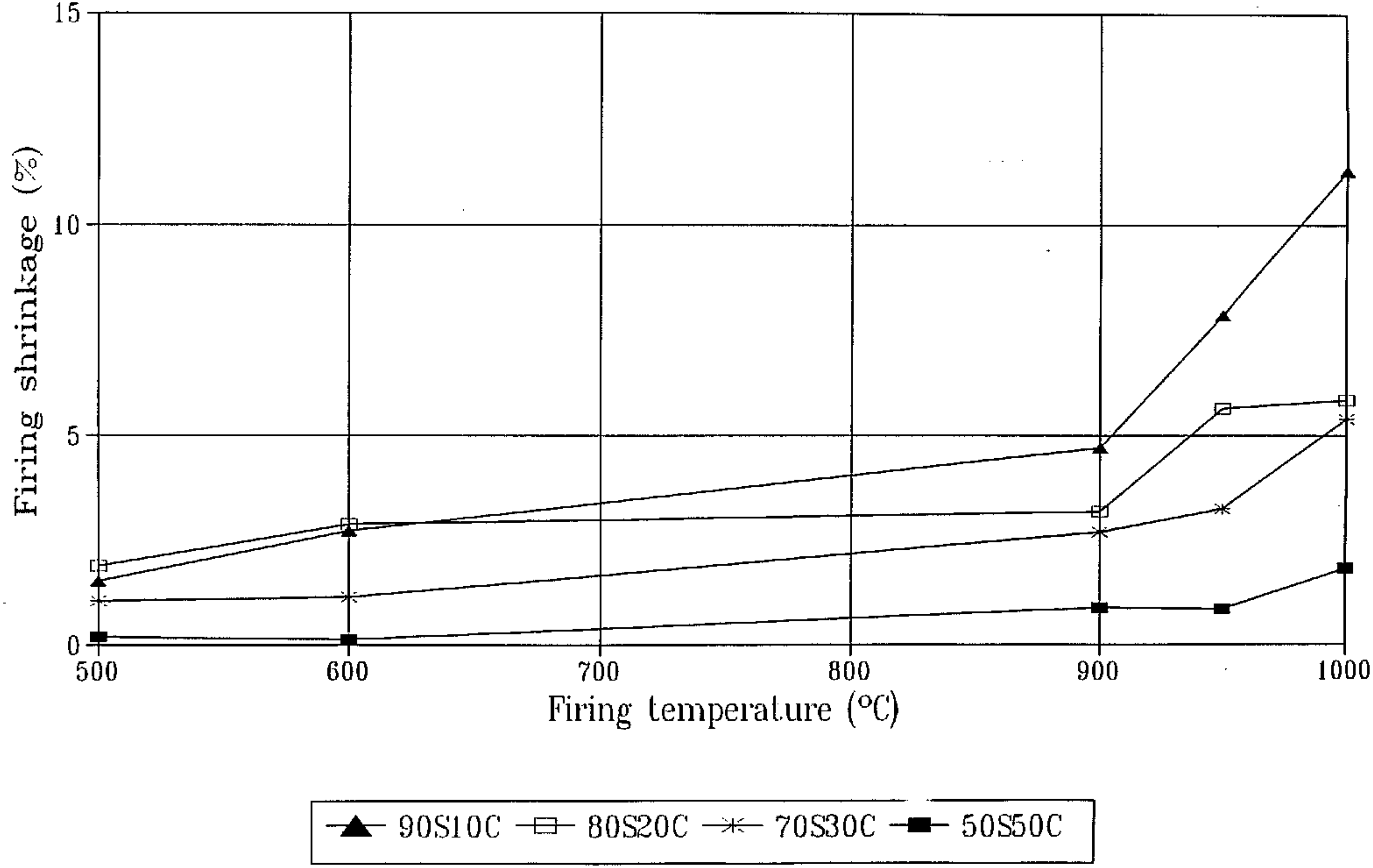
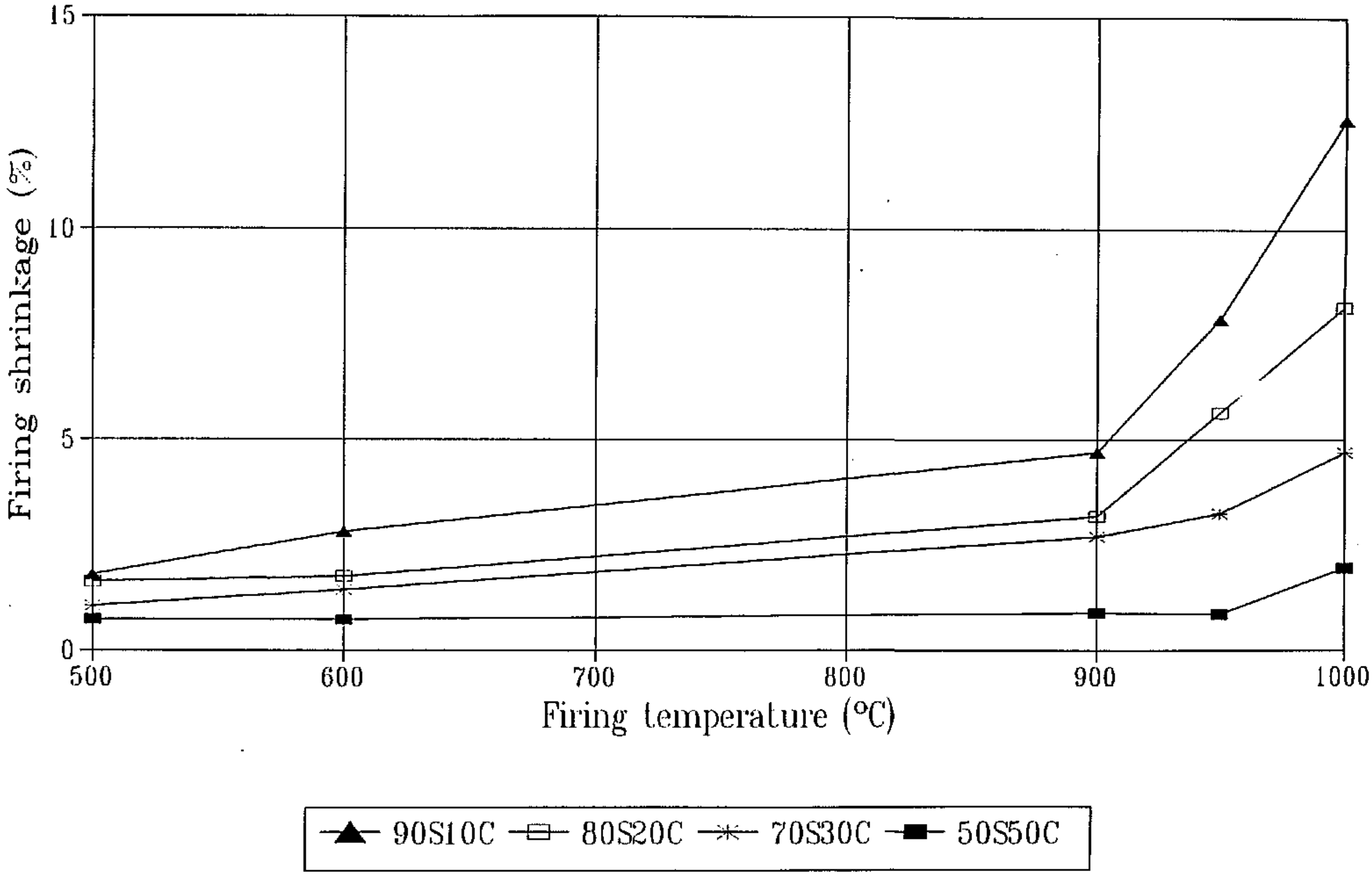


Figure 5.4 : Shrinkage vs firing temp.
F10 tiles fired 600/24hrs 1050 calcines



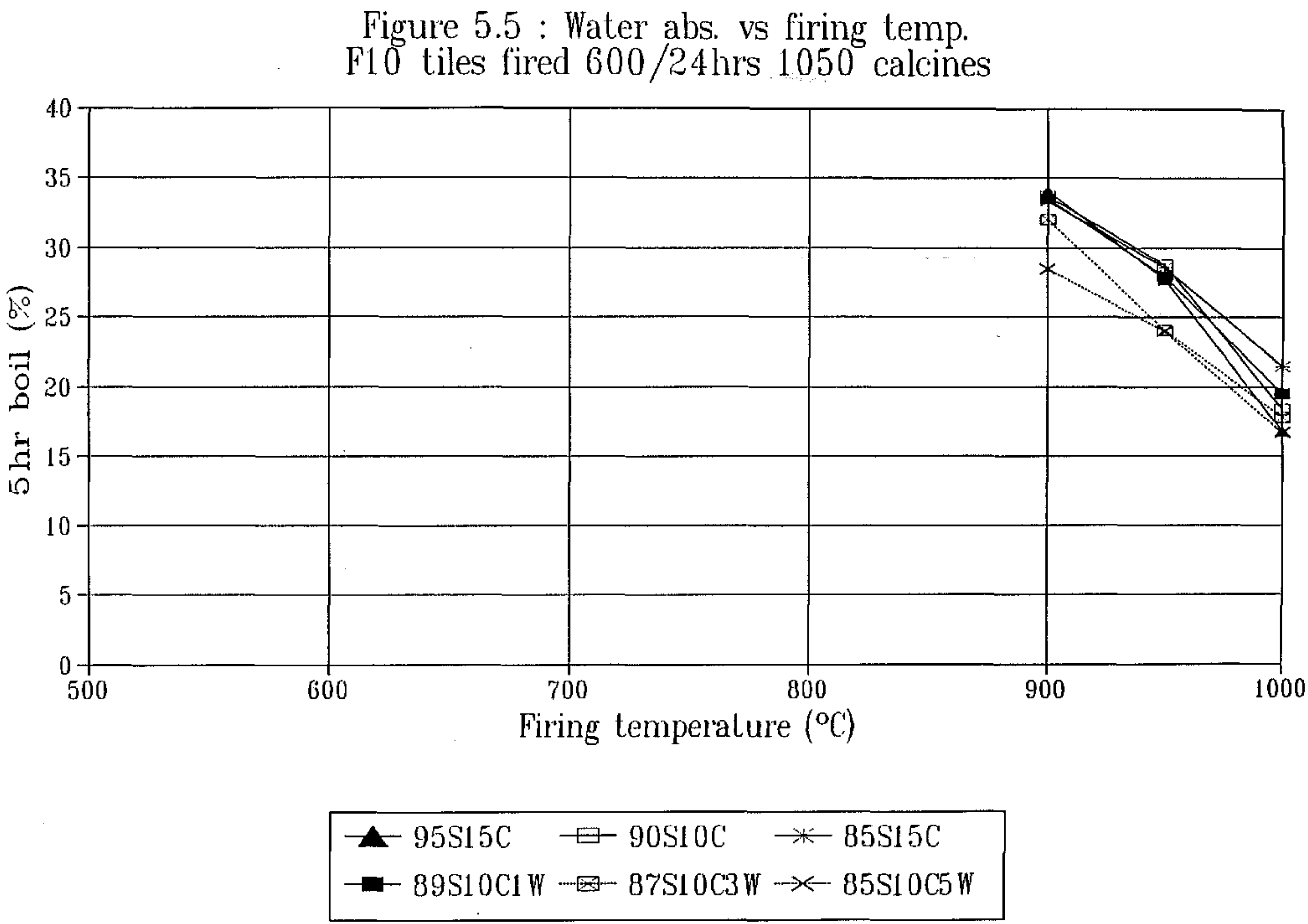


Figure 5.5 : Water abs. vs firing temp.
F10 tiles fired 600/24hrs 1050 calcines
5hr boil (%)
Firing temperature (°C)
0
5
10
15
20
25
30
35
40
500
600
700
800
900
1000
95S15C
90S10C
85S15C
89S10C1W
87S10C3W
85S10C5W

69

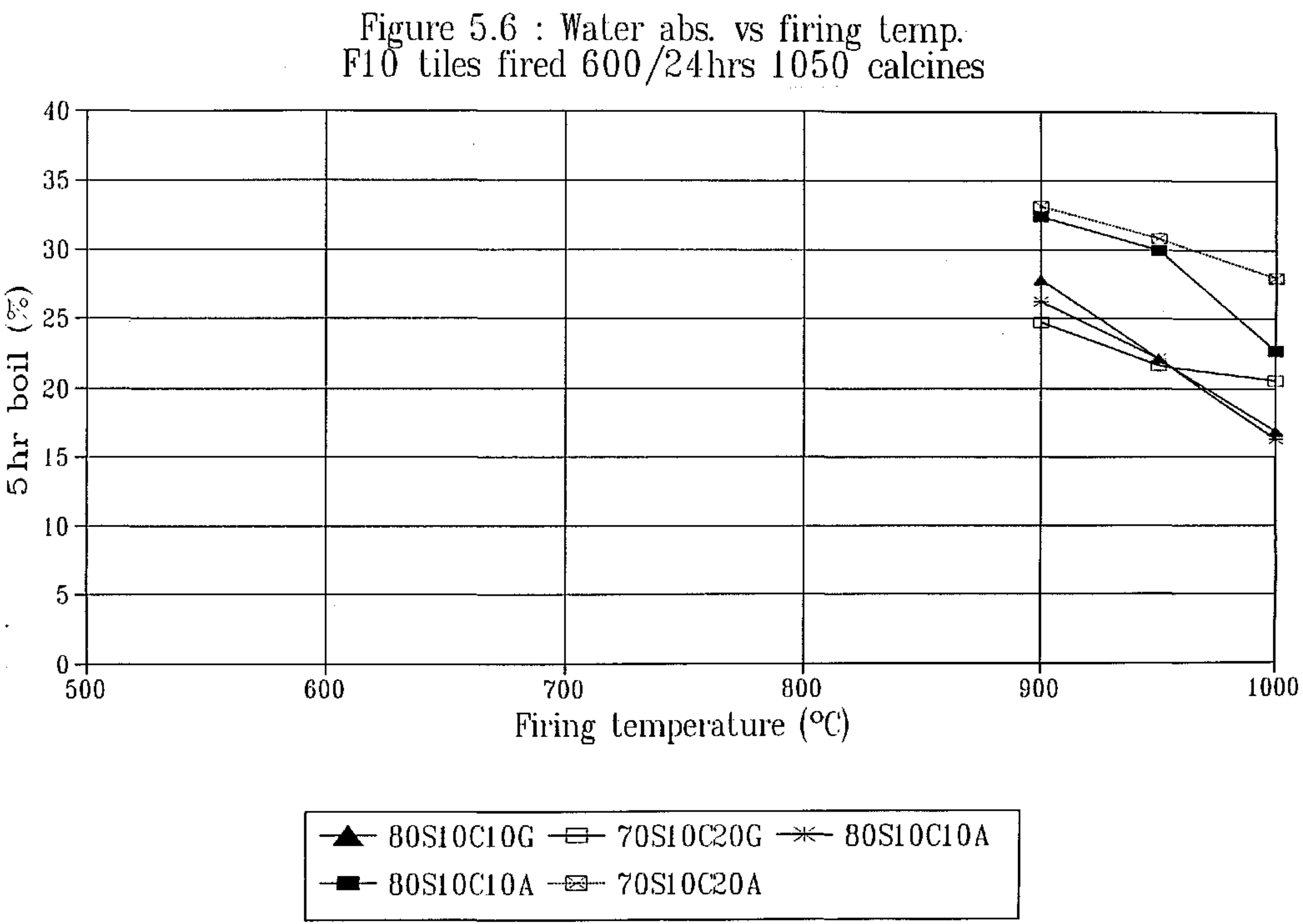


Figure 5.6 : Water abs. vs firing temp.
F10 tiles fired 600/24hrs 1050 calcines
5 hr boil (%)
0
5
10
15
20
25
30
35
40
500
600
700
800
900
1000
Firing temperature (°C)
80S10C10G
70S10C20G
80S10C10A
80S10C10A
70S10C20A

5.4 DISCUSSION OF RESULTS

5.4.1 Forming properties

Comments on the brickmaking forming of the tiles are all good.

Drying shrinkage or expansion values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. All the tiles have drying shrinkages varying between 0 and 2 %; which is very good.

All the tiles which were dried at room temperature showed no signs of cracking. The samples are thus not sensitive to drying and precautions do not have to be taken in practice to ensure that tiles made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

5.4.2 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks.

Table 5.24 gives the firing range of the cored tiles based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 5.5 to 5.23 and Figures 5.1, 5.2, 5.5 and 5.6.

5.4.3 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long respectively.

Table 5.25 gives the firing range of the different series of tiles based upon an acceptable firing shrinkage range as described above. This Table is derived from the data given in Tables 5.5 to 5.23 and Figures 5.3 to 5.4.

Table 5.24 : Water absorption firing range of cored tiles

Sample	5hr WA range ($\sim 20 \geq WA \geq \sim 8$) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
1000°C calcines				
100 S*	15 - 8	1000 - 1050	very short	poor
90S10C	16	1000	very short	bad
80S20C	-	-	-	-
70S30C	-	-	-	-
50S50C	19	1000	very short	bad
1050°C calcines				
100 S*	15 - 8	1000 - 1050	very short	poor
90S10C	16	1000	very short	bad
80S20C	20	1000	very short	bad
70S30C	-	-	-	-
50S50C	-	-	-	-
1050°C calcines Series II				
100 S*	15 - 8	1000 - 1050	very short	poor
95S05C	17	1000	very short	bad
90S10C	18	1000	very short	bad
85S15C	-	-	-	-
10G	17	1000	very short	bad
20G	-	-	-	-
10A	16	1000	very short	bad
10A	-	-	-	-
20A	-	-	-	-
1W	20	1000	very short	bad
3W	18	1000	very short	bad
5W	17	1000	very short	bad

* 100 S is the cored F5F 400 see Table 4.16

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable.

All the samples would make poor or bad clamp brickmaking materials from the water absorption firing range point of view. All the poor and bad clamp brickmaking samples would make acceptable and poor, respectively, kiln brickmaking materials from the water absorption firing range point of view.

Table 5.25 : Firing shrinkage firing range of cored tiles

Sample	Firing shrinkage ($0 \leq FS \leq 7$) (%)	Firing range (°C)	Firing range comments	Brickmaking comments
1000°C calcines				
100 S*	7	up to 950	medium	good
90S10C	3 - 5	600 - 930	very long	good
80S20C	3 - 6	600 - 1000	very long	good
70S30C	1 - 5	600 - 1000	very long	good
50S50C	0 - 2	600 - 1000	very long	very good
1050°C calcines				
100 S*	7	up to 950	medium	good
90S10C	3 - 5	600 - 900	very long	good
80S20C	2 - 6	600 - 920	very long	good
70S30C	1 - 5	600 - 1000	very long	good
50S50C	1 - 2	600 - 1000	very long	very good
1050°C calcines Series II				
100 S*	7	up to 950	medium	good
95S5C	1 - 7	600 - 950	very long	good
90S10C	1 - 6	600 - 950	very long	good
85S15C	1 - 7	600 - 1000	very long	good
10G	1 - 7	600 - 950	very long	good
20G	1 - 6	600 - 1000	very long	good
10A	1 - 7	600 - 950	very long	good
10A	1 - 7	600 - 1000	very long	good
20A	1 - 4	600 - 1000	very long	good
1W	1 - 6	600 - 950	very long	good
3W	1 - 4	600 - 900	very long	good
5W	1 - 7	600 - 950	very long	good

* 100 S is the cored F5F 400 see Table 4.17, firing range is estimated

All the tiles would make good to very good brickmaking materials from the firing shrinkage point of view.

5.4.4 Brickmaking

Based upon the combination of the water absorption and firing shrinkage firing ranges given in Tables 5.24 and 5.25 there is no common brickmaking firing range for the different series of tiles, except for sample 50S50C at 1000°C. Sample 50S50C at 1000°C has an acceptable firing range for a kiln brickmaking material but it's condition after firing of bad and friable make it unacceptable. All the samples are therefore unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

The colours of the fired tiles should be acceptable for bricks.

It is again recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

As expected the calcine additions reduce firing shrinkage and keep water absorption from decreasing. The flux additions have no great effect on the water absorption and firing shrinkage.

5.4.5 Tilemaking

According to the water absorption floor tile requirements given in Section 3.4.5 all the cored tiles would be classified as BIV floor tiles and would have to be glazed.

According to the water absorption glaze wall tile requirements given in Section 3.4.5 all the tiles meeting the brickmaking water absorption range given in Table 5.24, if fired in a ceramic kiln where the temperature can be controlled accurately, could possibly meet these tile requirements.

The dimensional tolerances given in Section 3.4.5 are quite stringent and it is believed that all the tiles meeting the brickmaking firing shrinkage range given in Table 5.25, if fired in a ceramic kiln where the temperature can be controlled, accurately could possibly meet these tile requirements.

In Table 5.26 the tiles which had good remarks and only slight defects after firing at or above 900°C from Tables 5.5 to 5.23 are given.

The colours of the fired tiles should be acceptable for tiles.

It is recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

Table 5.26 : Tiles which had good remarks after firing

Sample	Firing temperature (°C)
1000°C calcines	
100 S*	1000
90S10C	930, 950
80S20C	-
70S30C	-
50S50C	-
1050°C calcines	
100 S*	1000
90S10C	920, 1000
80S20C	900
70S30C	900, 920
50S50C	-
1050°C calcines Series II	
100 S*	1000
95S5C	900, 950, 1000
90S10C	900
85S15C	900
10G	900
20G	-
10A	900, 950, 1000
10A	900
20A	-
1W	900
3W	900
5W	900

* 100 S is the cored F5F 400 see Table 4.9

Considering all the above it is concluded that the cored tiles may make fair glazed floor and wall tile materials and it is recommended that glazed trials be undertaken on the successful compositions.

Though the tiles do not at this stage meet the specified requirements for unglazed tiles it is believed that it may be possible to produce a lower grade, such as quarry or rustic tiles, from these materials. We conclude that the sludge may be suitable for producing rustic tiles.

5.5 CONCLUSIONS

a) None of the samples are sensitive to drying and no precautions have to be taken in practice to ensure that tiles made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

b) All the samples are unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

c) The calcine additions reduce firing shrinkage and keep water absorption from decreasing. The flux additions have no great effect on the water absorption and firing shrinkage.

d) The colours of the fired tiles should be acceptable for bricks or tiles.

e) The cored tiles may make fair glazed floor and wall tile materials.

f) It is concluded that the sludge may be suitable for producing rustic tiles.

5.6 RECOMMENDATIONS

a) It is recommended that attempts be made to obtain a common water absorption and firing range for the bricks and tiles.

b) It is recommended that glazed trials be undertaken on the successful compositions.

6. EFFECT OF PARTICLE SIZE ON 150 MM PRESSED TILES

6.1 SCOPE

To determine the effects of particle size on the fired properties of floor tiles.

6.2 EXPERIMENTAL WORK

6.2.1 Sample preparation

For the preparation of tiles the dried sludge and addition were first hammer milled until all material passed through a 1 mm sieve. The addition used was 1050°C sludge calcines. The materials were ground and sieved till they reached the required particle size of either 1000, 850, 425, 125 and 63 μm. They were then mixed by hand with 5, 7 or 10% water. Tiles were then pressed at the required pressure. The dimension of the tile die was 150 mm square. The tiles were then dried in air at room temperature for at least one week followed by drying overnight at 110°C.

6.2.2 Firing

The dried tiles were fired in a laboratory electric furnace. A coring cycle (see Section 4.2.2) was adopted of one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature or left to cool. A tile of each composition was fired at most of the following temperatures: 600, 950, 970, 980 and 1000°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

6.3 RESULTS

6.3.1 Forming properties

The results obtained on the pressed tiles are given in Table 6.1.

Table 6.1 : Summary of forming properties of tiles

Sample	Drying shrinkage (%)	Water content (%)	Pressing & drying behaviour
F10 90S10C (1050°C calcines)			
hammer milled	1,0	11,2	good
1000 μm sieved	0,9	10,5	good
125 μm sieved	1,0	9,8	good
63 μm sieved	-	10,9	-
F5 850 μm			
100 kp/cm^2	1,2	8,0	good
200 kp/cm^2	1,3	8,0	good
300 kp/cm^2	0,8	8,1	good
F5 425 μm			
100 kp/cm^2	2,2	9,3	good
200 kp/cm^2	2,0	9,3	good, <crack
300 kp/cm^2	1,4	9,3	good, <crack
F7 850 μm			
100 kp/cm^2	1,4	9,9	good
200 kp/cm^2	1,3	10,0	good
300 kp/cm^2	1,2	10,0	good
F7 425 μm			
100 kp/cm^2	2,3	10,8	good
200 kp/cm^2	1,8	11,0	good
300 kp/cm^2	1,3	11,1	good, <crack
F10 850 μm			
100 kp/cm^2	1,4	12,8	good
200 kp/cm^2	1,5	13,2	good
300 kp/cm^2	1,4	13,1	good
F10 425 μm			
100 kp/cm^2	2,8	13,8	good
200 kp/cm^2	1,7	14,0	good
300 kp/cm^2	1,6	14,4	good, <crack

The dial readings of 100, 200 and 300 kp/cm^2 correspond to pressures of 7,4 MPa (167 000 N/22 500 mm^2), 15,1 MPa (340 000 N/22 500 mm^2) and 22,8 MPa (513 000 N/22 500 mm^2), respectively. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F5 850 μm means 5% water on a dry basis was added to unfired sludge F sieved through a 850 μm sieve.

6.3.2 Fired properties

The results obtained on the fired tiles are given in Tables 6.2 to 6.11 and are graphically presented in Figures 6.1 to 6.4.

Table 6.2 : Fired properties of cored tiles for sample F10 90% sludge / 10% calcines (1050°C) hammer milled

PROPERTY	TEMPERATURE (°C)				
	500	600	930	950 (250*)	1000
Firing shrinkage (%)		2,0		7,3	10,5
Mass loss (%)		17,6		18,3	16,8
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient				26,2 27,6 0,95	15,9 17,7 0,90
Colour		brown		brown	brown
Remarks after firing		good, <cracks		good, <cracks	good, <cracks

* units are kp/cm^2

Table 6.3 : Fired properties of cored tiles for sample F10 90% sludge / 10% calcines (1050°C) 1000 μm sieved

PROPERTY	TEMPERATURE (°C)				
	500	600	930	950	1000
Firing shrinkage (%)		2,9		9,0	14,1
Mass loss (%)		20,2		20,9	19,4
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient				26,9 28,2 0,95	14,3 15,7 0,91
Colour		brown		brown	brown
Remarks after firing		good, <cracks		good, <cracks	good, <cracks

Table 6.4 : Fired properties of cored tiles for sample F10
90% sludge / 10% calcines (1050°C) 125 μm sieved

PROPERTY	TEMPERATURE (°C)					
	500	600	930	950 (300*)	1000 (250)	1000 (300)
Firing shrinkage (%)		2,8		8,6	12,8	
Mass loss (%)		19,4		20,2	18,8	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient				25,1 26,6 0,94	14,7 16,1 0,92	
Colour		brown		brown	brown	brown
Remarks after firing		good		good, press cracks	excellent	good, <cracks

* units are kp/cm²

Table 6.5 : Fired properties of cored tiles for sample F10
90% sludge / 10% calcines (1050°C) 63 μm sieved

PROPERTY	TEMPERATURE (°C)				
	500	600	930	950 (250*)	1000 (300)
Firing shrinkage (%)		-		8,3	12,6
Mass loss (%)		19,4		20,3	18,8
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient				26,6 27,6 0,96	14,2 15,2 0,93
Colour		brown		brown	brown
Remarks after firing		good		cracked	good, <cracks

* units are kp/cm²

Table 6.6 : Fired properties of 850 μm milled sample F5

PROPERTY	TEMPERATURE (°C)					
	600 (100*)	1000 (100)	600 (200)	1000 (200)	600 (300)	1000 (300)
Firing shrinkage (%)	1,6	15,0	1,4	14,3	1,6	14,0
Mass loss (%)	18,3	19,8	18,4	19,5	18,6	19,7
24hr cold absorption (%)	49,6	20,0	45,5	16,7	41,6	15,3
5hr boil absorption (%)	52,9	21,7	47,9	18,1	43,5	16,5
Saturation coefficient	0,94	0,92	0,95	0,92	0,96	0,93
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good

* units are kp/cm²

Table 6.7 : Fired properties of 425 μm milled sample F5

PROPERTY	TEMPERATURE (°C)							
	600 (100*)	1000 (100)	600 (200)	970 (200)	1000 (200)	600 (300)	980 (300)	1000 (300)
Firing shrinkage (%)	2,1	14,7	1,6	8,8	14,1	1,2	10,3	13,6
Mass loss (%)	18,1	19,6	18,1	-	19,4	18,4	-	19,4
24hr cold absorption (%)	50,9	21,5	44,6	25,7	18,1	40,8	20,5	15,0
5hr boil absorption (%)	54,6	23,3	47,3	27,7	19,4	43,0	22,1	16,2
Saturation coefficient	0,93	0,92	0,94	0,93	0,93	0,95	0,93	0,93
Colour	brown	brown	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good	good	good

* units are kp/cm²

Table 6.8 : Fired properties of 850 μm milled sample F7

PROPERTY	TEMPERATURE (°C)					
	600 (100*)	1000 (100)	600 (200)	1000 (200)	600 (300)	1000 (300)
Firing shrinkage (%)	2,0	13,9	1,3	15,2	1,5	14,2
Mass loss (%)	18,3	19,6	18,5	20,5	18,5	18,6
24hr cold absorption (%)	49,2	21,0	44,2	16,5	40,4	13,1
5hr boil absorption (%)	60,5	23,0	46,6	17,7	42,1	14,1
Saturation coefficient	0,81	0,91	0,95	0,93	0,96	0,92
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good

* units are kp/cm^2

Table 6.9 : Fired properties of 425 μm milled sample F7

PROPERTY	TEMPERATURE (°C)							
	600 (100*)	1000 (100)	600 (200)	970 (200)	1000 (200)	600 (300)	980 (300)	1000 (300)
Firing shrinkage (%)	1,8	14,8	1,3	7,7	14,3	1,3	10,4	13,7
Mass loss (%)	18,3	19,6	19,9	-	19,4	18,6	-	19,4
24hr cold absorption (%)	49,7	20,3	43,0	25,5	15,6	38,7	18,9	14,1
5hr boil absorption (%)	53,2	22,2	45,6	27,2	17,0	40,8	20,5	15,2
Saturation coefficient	0,93	0,91	0,94	0,94	0,92	0,95	0,92	0,93
Colour	brown	brown	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good	good	good

* units are kp/cm^2

Table 6.10 : Fired properties of 850 μm milled sample F10

PROPERTY	TEMPERATURE (°C)					
	600 (100*)	1000 (100)	600 (200)	1000 (200)	600 (300)	1000 (300)
Firing shrinkage (%)	11,8	14,4	1,4	14,3	1,3	14,2
Mass loss (%)	18,3	19,6	18,3	22,0	18,6	17,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	48,4 51,7 0,94	20,1 21,9 0,92	43,0 45,1 0,95	18,0 19,3 0,93	39,3 40,9 0,96	11,5 12,5 0,92
Colour	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good

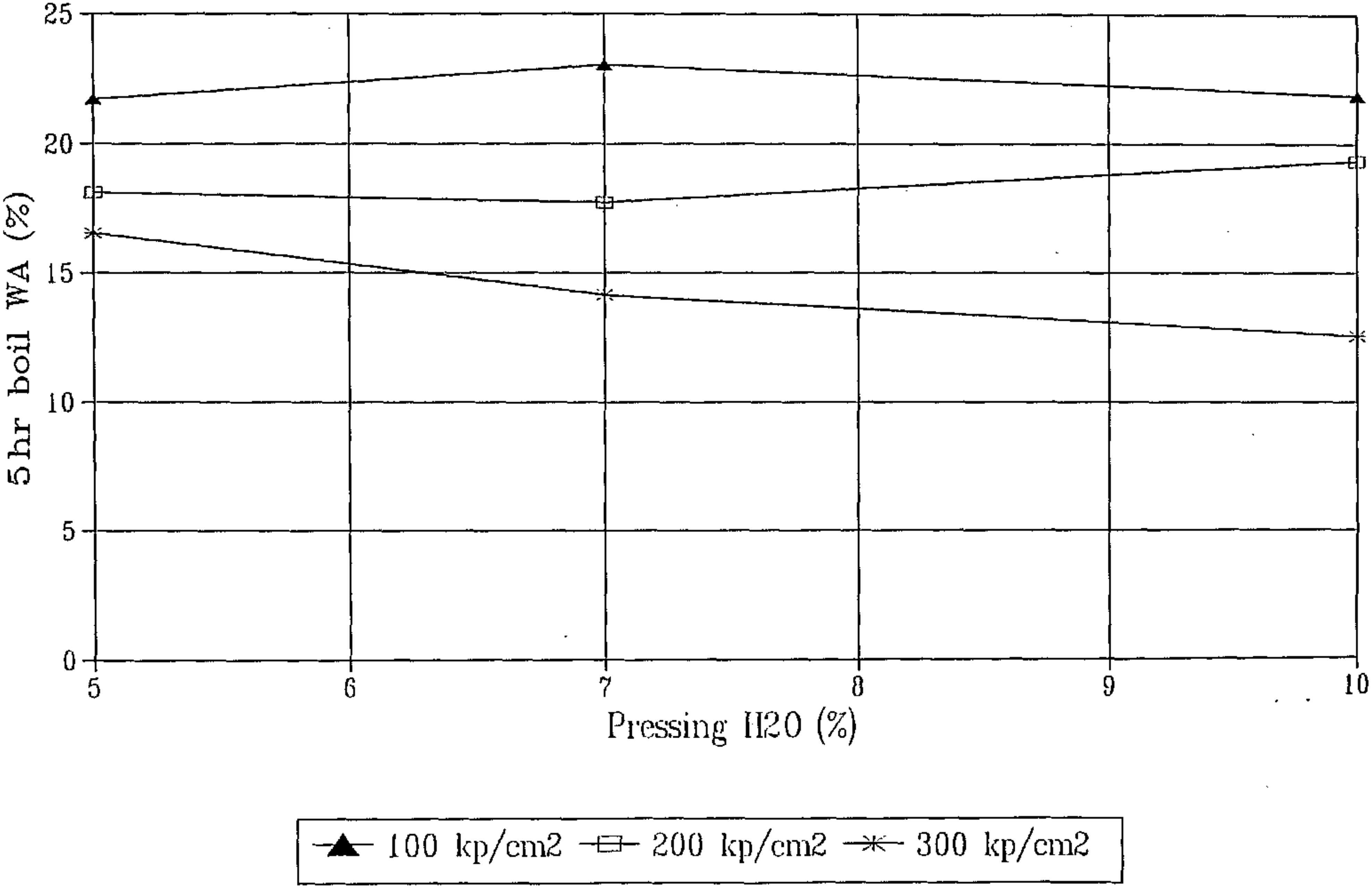
* units are kp/cm^2

Table 6.11 : Fired properties of 425 μm milled sample F10

PROPERTY	TEMPERATURE (°C)							
	600 (100*)	1000 (100)	600 (200)	970 (200)	1000 (200)	600 (300)	980 (300)	1000 (300)
Firing shrinkage (%)	1,5	14,0	1,5	8,5	-	1,1	9,3	14,2
Mass loss (%)	18,3	19,5	20,0	-	19,5	18,7	-	19,4
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	48,8 52,2 0,93	20,4 22,4 0,91	41,8 44,4 0,94	24,4 26,2 0,93	14,9 16,2 0,92	37,0 38,8 0,95	18,6 20,1 0,93	12,1 13,1 0,92
Colour	brown	brown	brown	brown	brown	brown	brown	brown
Remarks after firing	good	good	good	good	good	good	good	good

* units are kp/cm^2

Figure 6.1 : Water abs. vs pressing
water 850μ floor tiles fired 1000°C



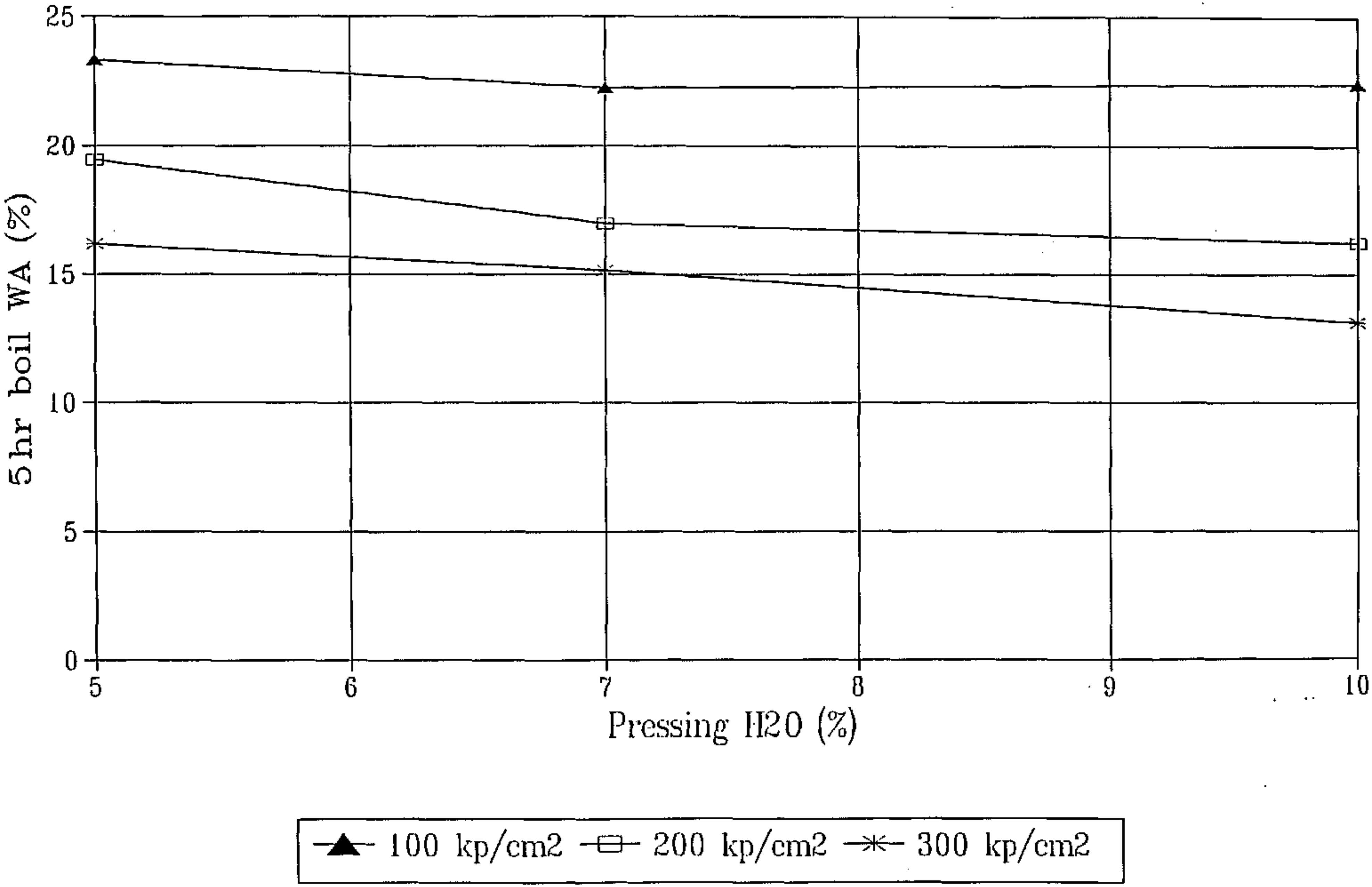


Figure 6.2 : Water abs. vs pressing
water 425μ floor tiles fired 1000°C

Figure 6.3 : Water abs. vs pressure
850μ floor tiles fired 1000°C

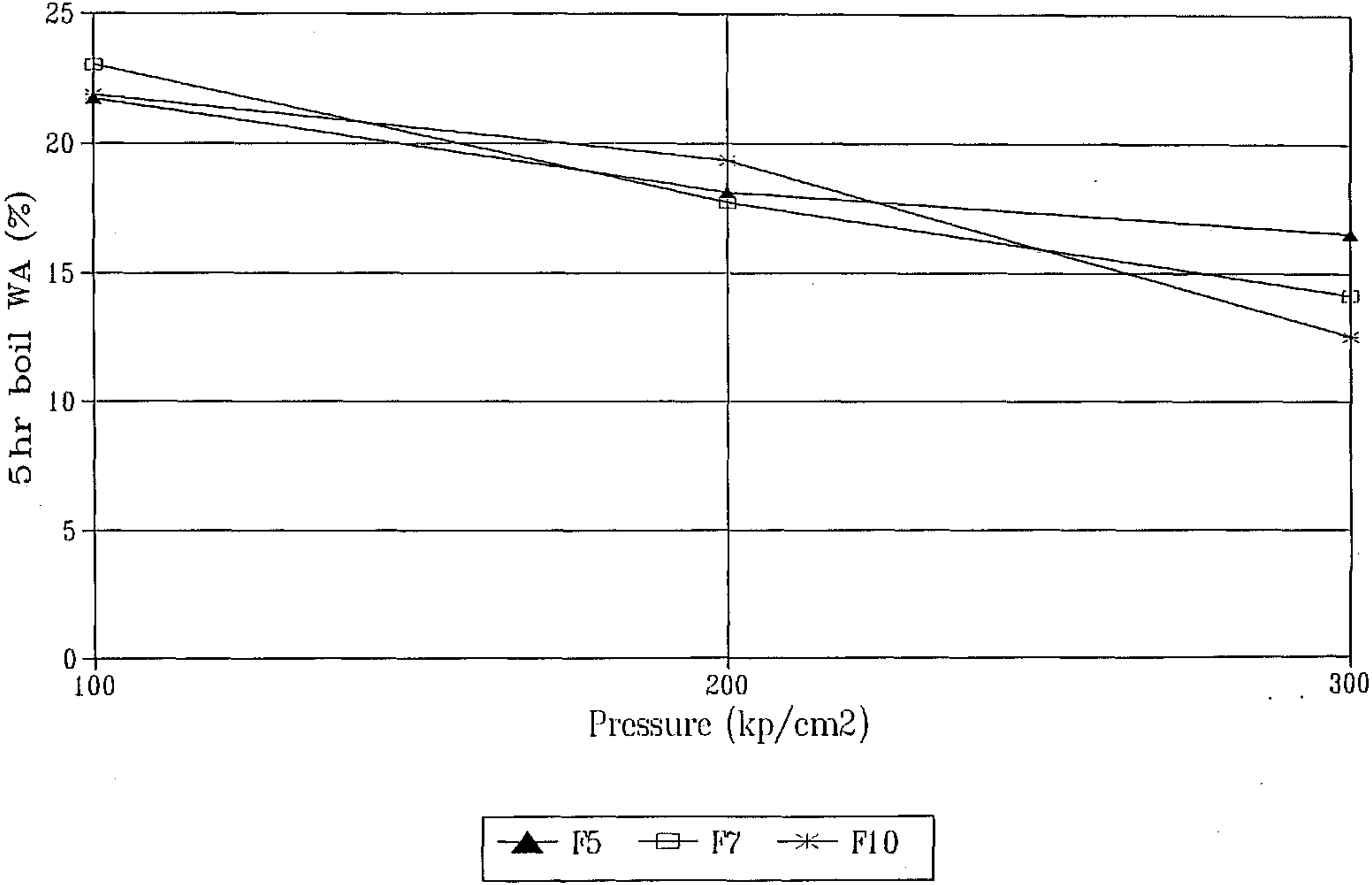
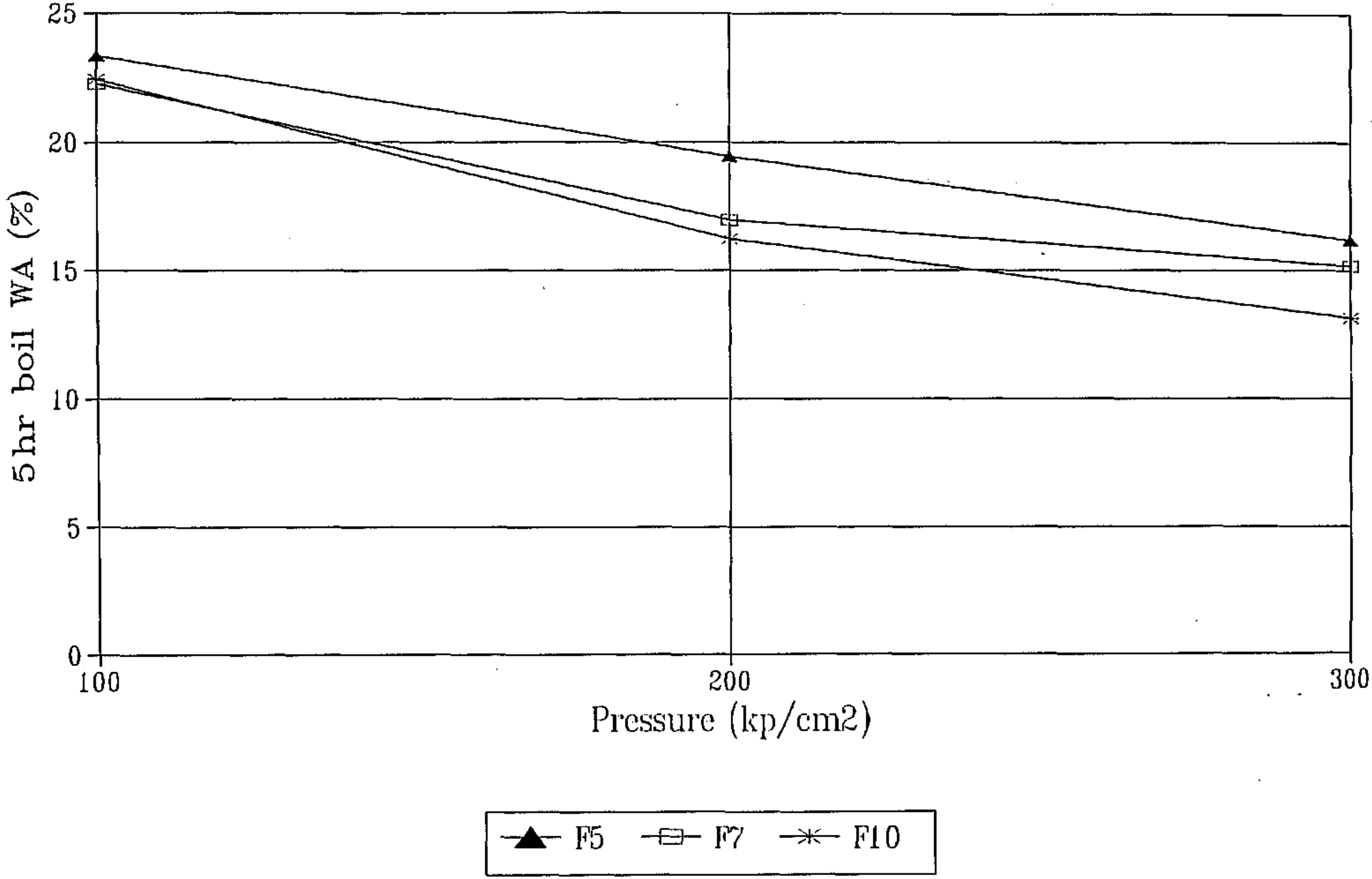


Figure 6.4 : Water abs. vs pressure
425µ floor tiles fired 1000°C



6.4 DISCUSSION OF RESULTS

6.4.1 Forming properties

Comments on the brickmaking forming of the tiles are all good.

Drying shrinkage or expansion values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. All the tiles have drying shrinkages varying between 1 and 3 % - which is good.

All the tiles except four of them which were dried at room temperature showed no signs of cracking. The four tiles mentioned above were all 425 μm tiles i.e. F5 200 and 300 kp/cm^2, F7 300 kp/cm^2 and F10 300 kp/cm^2. The samples are thus not very sensitive to drying and precautions do not have to be taken in practice to ensure that bricks or tiles made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

6.4.2 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks. From Tables 6.2 to 6.11 all the calcines addition samples and all the 100% sludge samples pressed at 200 and 300 kp/cm^2, fired at 1000°C have acceptable water absorptions. From Tables 6.6 to 6.11 all the 100% sludge samples pressed at 100 kp/cm^2, have unacceptable water absorptions.

Table 6.12 gives the firing range of the cored tiles based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 6.2 to 6.11.

Clamp firing ranges of 50, 100, 150, 200 and $\geq$300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln, where temperatures can be controlled accurately, a very short clamp firing range of 50°C is acceptable.

All the samples would make bad or unacceptable clamp brickmaking materials from the water absorption firing range point of view. All the bad clamp brickmaking samples would make poor kiln brickmaking materials from the water absorption firing range point of view.

Table 6.12 : Water absorption firing range of cored tiles

Sample	5hr WA range ($\sim20 \geq WA \geq \sim8$) (%)	Firing range (°C)	Firing range comments	Brickmaking comments
F10 90S10C (1050°C calcines)				
hammer milled	18	1000	too short	bad
1000 μm sieved	16	1000	too short	bad
125 μm sieved	16	1000	too short	bad
63 μm sieved	15	1000	too short	bad
F5 850 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	18	1000	too short	bad
300 kp/cm^2	16	1000	too short	bad
F5 425 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	19	1000	too short	bad
300 kp/cm^2	16	1000	too short	bad
F7 850 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	18	1000	too short	bad
300 kp/cm^2	14	1000	too short	bad
F7 425 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	17	1000	too short	bad
300 kp/cm^2	15	1000	too short	bad
F10 850 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	19	1000	too short	bad
300 kp/cm^2	12	1000	too short	bad
F10 425 μm				
100 kp/cm^2	-	-	-	-
200 kp/cm^2	16	1000	too short	bad
300 kp/cm^2	20 - 13	980 - 1000	too short	bad

6.4.3 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. From Tables 6.2 to 6.11 only the hammer milled calcines addition sample fired at 950°C has just an acceptable firing shrinkage.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. Table 6.13 gives the firing range of the different series of tiles based upon an acceptable firing shrinkage range as described above. This Table is derived from the data given in Tables 6.2 to 6.5. There are no acceptable firing shrinkages in the data given in Tables 6.6 to 6.12.

Table 6.13 : Firing shrinkage firing range of cored tiles

Sample	Firing shrinkage $(0 \leq FS \leq 7)$ (%)	Firing range (°C)	Firing range comments	Brickmaking comments
F10 90S10C (1050°C calcines)				
hammer milled	7	950	too short	bad
1000 μm sieved	-	-	-	-
125 μm sieved	-	-	-	-
63 μm sieved	-	-	-	-

All the tiles investigated would make bad or unacceptable clamp brickmaking materials from the firing shrinkage firing range point of view. The bad clamp brickmaking sample investigated would make a poor kiln brickmaking material from the firing shrinkage firing range point of view. These conclusions are not as serious as they seem since we have no data between 600 and 950°C. It is believed that the firing shrinkage would be acceptable below 950°C from results from Sections 4 and 5, but this would have to be investigated.

6.4.4 Brickmaking

Based upon the combination of the water absorption and firing shrinkage firing ranges given in Tables 6.12 and 6.13 there is no common brickmaking firing range for the different series of tiles. All the samples are therefore unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

It is again recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

The colours of the fired tiles should be acceptable for bricks.

The controlled particle sizes less than 1000 μm do have a beneficial effect on the quality of the tiles, however, they do not have a great effect on obtaining a common water absorption and firing shrinkage firing range.

6.4.5 Tilemaking

According to the water absorption floor tile requirements given in Section 3.4.5 all the floor tiles would be classified as BIV floor tiles and would have to be glazed.

According to the water absorption glaze wall tile requirements given in Section 3.4.5 all the tiles meeting the brickmaking water absorption range given in Table 6.12 if fired in a ceramic kiln where the temperature can be controlled accurately could possibly meet these tile requirements.

The dimensional tolerances given in Section 3.4.5 are quite stringent and it is believed that they may be met if the tiles are fired in a ceramic kiln where the temperature can be controlled accurately.

From Tables 6.2 to 6.11 of the 55 tiles produced all have good remarks after firing except the F10.90S10C 63 μm sample fired at 950°C. This excellent result is mainly due to the effect of controlled particle size.

The colours of the fired tiles should be acceptable for tiles.

Considering all the above it is concluded that the cored tiles may make fair glazed floor and wall tile materials and it is recommended that glazed trials be undertaken on the successful compositions.

It is recommended that attempts be made to obtain a common water absorption and firing range for the tiles.

Though the tiles do not at this stage meet the specified requirements for unglazed tiles we believed that it may be possible to produce a lower grade such as quarry or rustic tiles from these materials. We conclude that the sludge may be suitable for producing rustic tiles.

6.5 CONCLUSIONS

a) None of the samples are very sensitive to drying and no precautions have to be taken in practice to ensure that tiles made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

b) All the samples are unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

c) The controlled particle sizes less than 1000 μm do have a beneficial effect on the quality of the tiles. However, they do not have a great effect on obtaining a common water absorption and firing shrinkage firing range.

d) Of the 55 tiles produced all have good remarks after firing, except one. This excellent result is mainly due to the effect of controlled particle size.

e) The colours of the fired tiles should be acceptable for bricks or tiles.

f) The cored tiles may make fair glazed floor and wall tile materials.

g) We conclude that the sludge may be suitable for producing rustic tiles.

6.6 RECOMMENDATIONS

a) It is recommended that attempts be made to obtain a common water absorption and firing range for the bricks and tiles.

b) It is recommended that glazed trials be undertaken on the successful compositions.

7. PRESSING OF PAVERS

7.1 SCOPE

To determine the effects of size, pressing pressure, water and soap contents, particle size and calcines on the fired properties of pavers.

7.2 EXPERIMENTAL WORK

7.2.1 Sample preparation

For the preparation of pavers the dried sludge and calcines were first hammer milled until all material passed through a 0,425 or 0,850 mm sieve. The 1050°C sludge calcines were used. It was then mixed by hand with 7 or 10% water. In some cases a solution of 20% (m/m) liquid soap (Teepol) was used for 15% water/soap mixtures. In all cases after mixing in the water or the water/soap solution the mixtures were sealed overnight to temper. The pavers were then pressed at the required pressure with 0,5, 1 or 2 kg of the different materials. The dimensions of the paver die was 116,4 by 243,3 mm (surface area 28 320 mm^2). The pavers were then dried in air at room temperature for at least one week followed by drying overnight at 110°C.

7.2.2 Firing

The dried pavers were fired in a laboratory electric furnace. A coring cycle (see Section 4.2.2) was adopted of one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature or left to cool. A paver of each composition was fired at most of the following temperatures: 600, 950, 980 and 1000°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

7.3 RESULTS

7.3.1 Forming properties

The results obtained on the pressed tiles are given in Tables 7.1 to 7.7.

Table 7.1 : Summary of forming properties of 100 kp/cm^2 pavers F7

FORMING PROPERTY	MASS (kg)					
	0,5 (425 μm)	1,0 (425 μm)	2,0 (425 μm)	0,5 (850 μm)	1,0 (850 μm)	2,0 (850 μm)
Pressing	good	good	good	good	good	good
Mixing water (%)	11,1	11,4	11,1	10,8	11,1	10,9
Drying shrinkage (%)	0,7	0,8	0,6	1,4	1,4	1,4
Drying	good	good	bad, cracked	good	good	good
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

Table 7.2 : Summary of forming properties of 200 kp/cm^2 pavers F7

FORMING PROPERTY	MASS (kg)					
	0,5 (425 μm)	1,0 (425 μm)	2,0 (425 μm)	0,5 (850 μm)	1,0 (850 μm)	2,0 (850 μm)
Pressing	good	good	good	good	good	good
Mixing water (%)	11,3	11,5	10,9	11,0	10,9	10,9
Drying shrinkage (%)	1,0	1,5	0,7	1,5	1,5	1,9
Drying	good	good	bad, cracked	good	good	fair, <cracks
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

Table 7.3 : Summary of forming properties of 100 kp/cm² pavers F10

FORMING PROPERTY	MASS (kg)					
	0,5 (425 μm)	1,0 (425 μm)	2,0 (425 μm)	0,5 (850 μm)	1,0 (850 μm)	2,0 (850 μm)
Pressing	good	good	good	good	good	good
Mixing water (%)	12,6	12,2	12,8	14,5	15,6	15,7
Drying shrinkage (%)	0,1	1,7	1,4	1,7	1,9	2,8
Drying	good	good	bad, cracked	good, <crack	good	fair, <cracks
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

Table 7.4 : Summary of forming properties of 200 kp/cm² pavers F10

FORMING PROPERTY	MASS (kg)					
	0,5 (425 μm)	1,0 (425 μm)	2,0 (425 μm)	0,5 (850 μm)	1,0 (850 μm)	2,0 (850 μm)
Pressing	good	good	good	good	good	good
Mixing water (%)	15,8	15,6	14,2	15,8	15,8	15,6
Drying shrinkage (%)	1,8	2,0	0,8	1,8	2,0	1,8
Drying	good	good	fair, <crack	good	good	good, <cracks
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

Table 7.5 : Summary of forming properties of 200 kp/cm² 850 μm pavers F10

FORMING PROPERTY	MASS (kg)		
	0,5	1,0	2,0
Pressing	good	good	good, edge press damage
Mixing water (%)	15,4	16,1	15,6
Drying shrinkage (%)	1,7	1,7	1,7
Drying	good	good	good
Colour	grey brown	grey brown	grey brown

Table 7.6 : Summary of forming properties of 200 kp/cm² 850 μm pavers F15

FORMING PROPERTY	MASS (kg)		
	0,5	1,0	2,0
Pressing	good	good	good
Mixing water (%)	21,4	21,8	21,5
Drying shrinkage (%)	2,2	2,8	2,4
Drying	good	good	good
Colour	grey brown	grey brown	grey brown

Table 7.7 : Summary of forming properties of 200 kp/cm² 850 μm pavers F20

FORMING PROPERTY	MASS (kg)		
	0,5	1,0	2,0
Pressing	good	good	good
Mixing water (%)	26,9	27,8	27,4
Drying shrinkage (%)	3,3	3,5	3,1
Drying	good	good, <crack	good, <cracks
Colour	grey brown	grey brown	grey brown

The dial readings of 100 and 200 kp/cm² correspond to pressures of 5,9 (167 000 N/28 320 mm²) and 12,0 MPa (340 000 N/28 320 mm²), respectively.

7.3.2 Fired properties

The results obtained on the fired pavers are given in Tables 7.4 to 7.27. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F7 means 7% water was added to unfired sludge F on a dry basis.

Table 7.8 : Fired properties of 0,5kg cored pavers 100 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	2,0	11,8	2,1	14,8
Mass loss (%)	18,7	-	19,2	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	27,6 29,3 0,94	- - -	21,5 23,0 0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, < soft	good	fair, press crack, soft	poor, cracked

Table 7.9 : Fired properties of 0,5kg cored pavers 200 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,6	11,1	1,7	14,2
Mass loss (%)	18,8	-	18,8	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	23,3 24,7 0,94	- - -	17,3 18,6 0,93
Colour	brown	brown	brown	brown
Remarks after firing	excellent	excellent	good	excellent

Table 7.10 : Fired properties of 1kg cored pavers 100 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,6	11,0	11,6	13,9
Mass loss (%)	18,7	-	19,1	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	28,1 30,2 0,93	- - -	21,8 23,4 0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, <cracks, <red core	poor, cracks	good, <cracks, red core	fair, cracks

Table 7.11 : Fired properties of 1kg cored pavers 200 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,4	10,9	1,6	13,9
Mass loss (%)	18,7	-	19,0	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	23,5 25,3 0,93	- - -	18,8 20,2 0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, <<crack <<red core	good, <crack	good, red core	good

Table 7.12 : Fired properties of 2kg cored pavers 100 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,8	11,4	1,8	14,5
Mass loss (%)	18,8	-	19,4	-
24hr cold absorption (%)	-	27,1	-	20,8
5hr boil absorption (%)	-	29,2	-	22,4
Saturation coefficient	-	0,93	-	0,93
Colour	brown	brown	brown	brown
Remarks after firing	bad, >cracks	unacceptable, drying cracks	poor, <cracks, soft, >red core	bad, drying cracks

Table 7.13 : Fired properties of 2kg cored pavers 200 kp/cm² for sample F7

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,4	10,8	1,8	13.6
Mass loss (%)	19,1	-	20,0	-
24hr cold absorption (%)	-	24,6	-	19,9
5hr boil absorption (%)	-	26,6	-	21,4
Saturation coefficient	-	0,93	-	0,93
Colour	brown	brown	brown	brown
Remarks after firing	bad, >cracks	broken, unacceptable, drying cracks	bad, >cracks	unacceptable, drying cracks

Table 7.14 : Fired properties of 0,5kg cored pavers 100 kp/cm² for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	2,1	11,5	2,1	14,7
Mass loss (%)	19,0	-	18,9	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	28,4 30,5 0,93	- - -	19,5 20,4 0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, <soft	good	fair, press crack, soft	good, <crack

Table 7.15 : Fired properties of 0,5kg cored pavers 200 kp/cm² for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,4	11,0	1,6	14,3
Mass loss (%)	18,6	-	18,7	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	23,1 24,6 0,94	- - -	16,6 17,6 0,94
Colour	brown	brown	brown	brown
Remarks after firing	excellent	excellent	good	good

Table 7.16 : Fired properties of 1kg cored pavers 100 kp/cm^2 for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,2	11,2	1,5	13,7
Mass loss (%)	18,8	-	19,0	-
24hr cold absorption (%)	-	27,7	-	20,6
5hr boil absorption (%)	-	30,1	-	22,2
Saturation coefficient	-	0,92	-	0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, <cracks, <red core	fair, crazed, <warp	good, <craze, red core	good, <cracks

Table 7.17 : Fired properties of 1kg cored pavers 200 kp/cm^2 for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,3	10,9	1,2	13,6
Mass loss (%)	18,6	-	18,9	-
24hr cold absorption (%)	-	22,4	-	16,8
5hr boil absorption (%)	-	24,2	-	18,1
Saturation coefficient	-	0,92	-	0,93
Colour	brown	brown	brown	brown
Remarks after firing	good, < <cracks, < <red core	good, <warp, <craze	good, red core	good

Table 7.18 : Fired properties of 2kg cored pavers 100 kp/cm² for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,8	11,1	1,4	14,3
Mass loss (%)	19,0	-	19,4	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	29,4 32,0 0,92	- - -	18,3 19,9 0,92
Colour	brown	brown	brown	brown
Remarks after firing	bad, >cracks	bad, drying cracks	fair, <cracks	bad, drying cracks

Table 7.19 : Fired properties of 2kg cored pavers 200 kp/cm² for sample F10

PROPERTY	TEMPERATURE (°C)			
	600 (425 μm)	980 (425 μm)	600 (850 μm)	1000 (850 μm)
Firing shrinkage (%)	1,3	10,7	1,3	13,3
Mass loss (%)	17,8	-	19,5	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	23,2 25,0 0,93	- - -	15,0 16,3 0,92
Colour	brown	brown	brown	brown
Remarks after firing	poor, crazing	bad, drying cracks	fair, <cracks	bad, drying cracks

Table 7.20 : Fired properties of 0,5kg cored pavers for sample F10 II (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	3,3			13,9
Mass loss (%)	18,1			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			13,8 15,5 0,89
Colour	brown			dark brown
Remarks after firing	good			excellent

Table 7.21 : Fired properties of 1kg cored pavers for sample F10 II (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	3,5			13,5
Mass loss (%)	18,5			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			16,6 18,7 0,88
Colour	brown			dark brown
Remarks after firing	good			good, <cracks

Table 7.22 : Fired properties of 2kg cored pavers for sample F10 II (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	3,4			12,7
Mass loss (%)	18,7			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			15,2 17,4 0,87
Colour	brown			dark brown
Remarks after firing				bad, cracked

Table 7.23 : Fired properties of 0,5kg cored pavers for sample F15 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950*	1000
Firing shrinkage (%)	3,5		-	14,0
Mass loss (%)	18,5			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -		23,7 24,9 0,95	11,0 12,4 0,89
Colour	brown		brown	brown
Remarks after firing	good		good	good

* Teepol used as with calcined samples

Table 7.24 : Fired properties of 1kg cored pavers for sample F15 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950*	1000
Firing shrinkage (%)	3,8	2,7	-	13,0
Mass loss (%)	18,7			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -		25,8 27,1 0,95	12,4 14,0 0,88
Colour	brown		brown	brown
Remarks after firing	good		good, <craze	good

* Teepol used as with calcined samples

Table 7.25 : Fired properties of 2kg cored pavers for sample F15 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950*	1000
Firing shrinkage (%)	3,5		-	13,8
Mass loss (%)	19,1			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -		25,5 26,9 0,95	12,5 14,1 0,88
Colour	brown		brown	brown
Remarks after firing	-		bad, cracked	bad, cracked

* Teepol used as calcined samples

Table 7.26 : Fired properties of 0,5kg cored pavers for sample F15T (850 μm) 80% sludge / 20% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)			-	6,2
Mass loss (%)				-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient			25,5 27,8 0,92	22,0 23,1 0,95
Colour			brown	brown
Remarks after firing			fair, crazed	fair, crazed

Table 7.27 : Fired properties of 1kg cored pavers for sample F15T (850 μm) 80% sludge / 20% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)			-	6,2
Mass loss (%)				-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient			26,7 29,2 0,91	22,2 23,4 0,95
Colour			brown	brown
Remarks after firing			fair, crazed	fair, crazed

Table 7.28 : Fired properties of 2kg cored pavers for sample F15T (850 μm) 80% sludge / 20% calcines (1050°C)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)			-	7,5
Mass loss (%)				-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient			26,3 29,1 0,90	21,0 22,2 0,95
Colour			brown	brown
Remarks after firing			fair, crazed	fair, crazed

Table 7.29 : Fired properties of 0,5kg cored pavers for sample F20 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	4,5			13,9
Mass loss (%)	18,4			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			9,7 10,9 0,89
Colour	brown			dark brown
Remarks after firing	good			good, <cracks

Table 7.30 : Fired properties of 1kg cored pavers for sample F20 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	5,1			14,1
Mass loss (%)	18,8			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			10,4 11,7 0,89
Colour	brown			dark brown
Remarks after firing	-			poor, cracked, crazed

Table 7.31 : Fired properties of 2kg cored pavers for sample F20 (850 μm)

PROPERTY	TEMPERATURE (°C)			
	600	900	950	1000
Firing shrinkage (%)	4,2			13,6
Mass loss (%)	19,5			-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -			10,7 12,0 0,89
Colour	brown			dark brown
Remarks after firing	-			bad, cracked

7.4 DISCUSSION OF RESULTS

7.4.1 Forming properties

Drying shrinkage or expansion values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. Tables 7.1 to 7.6 show that the pavers with water contents up to 15% have drying shrinkages below 3% which is low and good. Table 7.7 shows that the F20 pavers drying shrinkages are below 4% which is medium and good.

All the 0,5 and 1kg pavers which were dried at room temperature showed no signs of cracking. These samples are thus not sensitive to drying and precautions do not have to be taken in practice to ensure that bricks or tiles made from these materials do not dry out too quickly. However, all the 2 kg 425 μm, and the 2kg 850 μm F7 200 kp/cm^2 and F10 100 kp/cm^2 pavers were either bad or fair. These are particle size and mass effects. The brickmaking forming comments on the pavers are all good except for the 2 kg 425 μm, and the 2kg 850 μm F7 200 kp/cm^2 and F10 100 kp/cm^2 pavers.

7.4.2 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks. From Tables 7.8 to 7.19 samples F7 0,5 and 1 kg 200 kp/cm^2, F10 0,5 and 2kg 100 kp/cm^2, and F10 0,5, 1 and 2 kg 200 kp/cm^2 fired at 1000°C and having 850 μm particle size have acceptable water absorptions.

Table 7.32 gives the firing range of the cored pavers based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 7.8 to 7.19.

Table 7.32 : Water absorption firing range of cored pavers

Sample	5hr WA range (~20 ≥ WA ≥ ~8) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
F7 (850 μm)				
0,5 kg 100 kp/cm²	-	-	-	-
1,0 kg 100 kp/cm²	-	-	-	-
2,0 kg 100 kp/cm²	-	-	-	-
0,5 kg 200 kp/cm²	19	1000	too short	bad
1,0 kg 200 kp/cm²	20	1000	too short	bad
2,0 kg 200 kp/cm²	-	-	-	-
F10 (850 μm)				
0,5 kg 100 kp/cm²	20	1000	too short	bad
1,0 kg 100 kp/cm²	-	-	-	-
2,0 kg 100 kp/cm²	20	1000	too short	-
0,5 kg 200 kp/cm²	18	1000	too short	bad
1,0 kg 200 kp/cm²	18	1000	too short	bad
2,0 kg 200 kp/cm²	16	1000	too short	bad

From Tables 7.20 to 7.31 all the samples, except the ones with 20% calcines, when fired at 1000°C and having 850 μm particle size have acceptable water absorptions. Table 7.33 gives the firing range of these cored pavers based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 7.20 to 7.31.

Table 7.33 : Water absorption firing range of cored pavers

Sample	5hr WA range ($\sim 20 \geq WA \geq \sim 8$) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
F10 II (850 μm)				
0,5 kg 200 kp/cm²	16	1000	too short	bad
1,0 kg 200 kp/cm²	19	1000	too short	bad
2,0 kg 200 kp/cm²	17	1000	too short	bad
F15 (850 μm)				
0,5 kg 200 kp/cm²	12	1000	too short	bad
1,0 kg 200 kp/cm²	14	1000	too short	bad
2,0 kg 200 kp/cm²	14	1000	too short	bad
F15T (850 μm) 80% sludge / 20% calcines				
0,5 kg 200 kp/cm²	-	-	-	-
1,0 kg 200 kp/cm²	-	-	-	-
2,0 kg 200 kp/cm²	-	-	-	-
F20 (850 μm)				
0,5 kg 200 kp/cm²	11	1000	too short	bad
1,0 kg 200 kp/cm²	12	1000	too short	bad
2,0 kg 200 kp/cm²	12	1000	too short	bad

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable.

All the samples given in Tables 7.23 and 7.24 would make bad or unacceptable clamp brickmaking materials from the water absorption firing range point of view. All the bad clamp brickmaking samples would make poor kiln brickmaking materials from the water absorption firing range point of view.

7.4.3 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. From the data given in Tables 7.8 to 7.31 all the samples fired above 900°C not containing calcines have firing shrinkages greater than 10% which is unacceptable, these samples fired at 600°C are all below 6% firing shrinkage. From the data given in Tables 7.26 to 7.28 the 0,5 and 1 kg pavers containing calcines fired at 1000°C have firing shrinkages of 6% which is acceptable, the 2 kg sample fired at 1000°C has a shrinkage of 8% which is very high. The addition of calcines can control firing shrinkage.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. No acceptable firing range for the different series of pavers based upon an acceptable firing shrinkage range has so far been found.

All the pavers investigated except the 0,5 and 1 kg pavers containing calcines would make bad or unacceptable clamp brickmaking materials from the firing shrinkage firing range point of view. The bad clamp brickmaking samples investigated would make poor kiln brickmaking materials from the firing shrinkage firing range point of view. These conclusions are not as serious as they seem since we have no data between 600 and 980°C. It is believed that the firing shrinkage would be acceptable below 950°C from results from Sections 4 and 5, but this would have to be investigated.

7.4.4 Brickmaking

Based upon the combination of the water absorption and firing shrinkage firing ranges given in Tables 7.32 and 7.33, and Section 7.4.3 there is no common brickmaking firing range for the different series of pavers. All the samples are therefore unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

It is noted that in Tables 7.8 to 7.31 and Table 7.34 all the pavers fired above 900°C and containing no calcines, of the 0,5 kg pavers 11 of the 12 are good, of the 1 kg pavers 7 of the 12 are good, and none of the twelve 2 kg pavers are good. This indicates that there is a relationship between the quality of the paver and the mass of the paver. It is recommended that the relationship between the quality of the paver and the mass of the paver be further investigated.

The colours of the fired pavers should be acceptable for pavers.

It is recommended that attempts be made to obtain a common water absorption and firing range for the pavers.

7.4.5 Tilemaking

According to the water absorption floor tile requirements given in Section 3.4.5 all the cored pavers would be classified as BIV floor tiles and would have to be glazed.

According to the water absorption glaze wall tile requirements given in Section 3.4.5 all the pavers meeting the brickmaking water absorption range given in Tables 7.32 and 7.33, if fired in a ceramic kiln where the temperature can be controlled accurately, could possibly meet these tile requirements.

The dimensional tolerances given in Section 3.4.5 are quite stringent and it is believed that the pavers meeting the brickmaking firing shrinkage range given in Section 7.4.3, if fired in a ceramic kiln where the temperature can be controlled accurately, could possibly meet these tile requirements.

In Table 7.34 the pavers which had good or excellent remarks and only slight defects after firing from Tables 7.4 to 7.31 are given.

The colours of the fired pavers should be acceptable for tiles.

It is recommended that attempts be made to obtain a common water absorption and firing range for the pavers.

Table 7.34 : Pavers which had good or excellent remarks after firing

Sample	Firing temperature (°C)
F7 0,5 kg 100 kp/cm² (425 μm)	600, 980
F7 0,5 kg 200 kp/cm² (425 μm & 850 μm)	600, 980, 1000
F7 1,0 kg 100 kp/cm² (425 μm & 850 μm)	600
F7 1,0 kg 200 kp/cm² (425 μm & 850 μm)	600, 980, 1000
F10 0,5 kg 100 kp/cm² (425 μm)	600, 980, 1000
F10 0,5 kg 100 kp/cm² (850 μm)	1000
F10 1,0 kg 100 kp/cm² (425 μm & 850 μm)	600, 1000
F10 0,5 kg 200 kp/cm² (425 μm & 850 μm)	600, 980, 1000
F10 1,0 kg 200 kp/cm² (425 μm & 850 μm)	600, 980, 1000
F10 II (850 μm)	
0,5 kg 200 kp/cm²	600, 1000
1,0 kg 200 kp/cm²	600, 1000
F15 (850 μm)	
0,5 kg 200 kp/cm²	600, 950, 1000
1,0 kg 200 kp/cm²	600, 950, 1000
F20 (850 μm)	
0,5 kg 200 kp/cm²	600, 1000

Considering all the above it is concluded that the cored pavers may make fair glazed floor and wall tile materials and it is recommended that glazed trials be undertaken on the successful compositions.

Though the pavers do not at this stage meet the specified requirements for unglazed tiles we believed that it may be possible to produce a lower grade such as quarry or rustic tiles from these materials. We conclude that the sludge may be suitable for producing rustic tiles.

7.5 CONCLUSIONS

a) None of the pavers except the 2 kg 425 μm, and the 2kg 850 μm F7 200 kp/cm^2 and F10 100 kp/cm^2 ones are sensitive to drying and no precautions have to be taken in practice to ensure that pavers made from these materials do not dry out too quickly. To extrapolate this behaviour to bricks would be premature at this stage.

b) All the pavers are unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

c) There is a relationship between the quality of the paver and the mass of the paver.

d) The addition of calcines can control firing shrinkage.

e) The colours of the fired pavers should be acceptable for pavers or tiles.

f) The cored pavers may make fair glazed floor and wall tile materials.

g) We conclude that the sludge may be suitable for producing rustic tiles.

7.6 RECOMMENDATIONS

a) It is recommended that the relationship between the quality of the paver and the mass of the paver be further investigated.

b) It is recommended that attempts be made to obtain a common water absorption and firing range for the pavers and tiles.

c) It is recommended that glazed trials be undertaken on the successful compositions.

8. PRESSING OF BRICKS

8.1 SCOPE

To determine the effects of water and pressure used for pressing, and the effects of coring and calcine additions on the fired properties of sludge bricks.

8.2 EXPERIMENTAL WORK

8.2.1 Sample preparation

For the preparation of bricks the dried material was first hammer milled until all material passed through the relevant size sieve or if not mentioned it is 1 mm. It was then mixed by hand with 7, 10, and 15% water. The dimensions of the brick die were 116,4 by 243,3 mm (surface area 28 320 mm^2). The bricks were then dried in air at room temperature for one week followed by drying overnight at 110°C.

8.2.2 Firing

The dried bricks were fired in a laboratory electric furnace. The bricks were fired at the stated temperatures. Where mentioned a coring cycle (see Section 4.2.2) was adopted of one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature or left to cool. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

8.3 RESULTS

8.3.1 Forming properties

The results obtained on the pressed bricks are given in Tables 8.1 to 8.3.

Table 8.1 : Summary of forming properties of bricks F15

FORMING PROPERTY	PRESSURE (kp/cm²)			
	350	400	450	500
Pressing	fair, crazed	fair, crazed	good, <craze	good, <craze
Mixing water (%)	13,8	13,3	13,5	14,2
Drying shrinkage (%)	1,5	1,3	1,3	1,8
Colour	grey brown	grey brown	grey brown	grey brown

Table 8.2 : Summary of forming properties of bricks F10 pressed at 400 kp/cm²

FORMING PROPERTY	COMPOSITION			
	90S10C	80S20C	70S30C	50S50C
Pressing	good	good	good	good
Mixing water (%)	14,9	14,2	12,2	12,6
Drying shrinkage (%)	2,1	1,2	0,9	1,0
Colour	grey brown	grey brown	grey brown	grey brown

Table 8.3 : Summary of forming properties of bricks made from ½ to 2 mm fines and pressed at 450 kp/cm²

FORMING PROPERTY	WATER CONTENT (%)	
	F7	F10
Pressing	good, <cracks	good
Mixing water (%)	21,6	27,7
Drying shrinkage (%)	2,3	3,2
Colour	grey brown	grey brown

8.3.2 Fired properties

The results obtained on the fired bricks are given in Tables 8.4 to 8.9. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F10 means 10% water was added to unfired sludge F on a dry basis.

Table 8.4 : Properties of uncored bricks of sample F7 and F10 pressed at 450 and 500 kp/cm² respectively

PROPERTY	TEMPERATURE (°C)	
	F7 1050	F10 1000
Firing shrinkage (%)	10,3	1,4
Mass loss (%)	32,2	6,5
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	9,4* 11,1* 0,85*	- - -
Colour	dark brown	brown
Remarks after firing	unacceptable, cracked	unacceptable, cracked

* unreliable data due to cracking

Table 8.5 : Properties of cored bricks of sample F10 pressed at 400 kp/cm²

PROPERTY	COMPOSITION						
	600°C			950°C			
	90S10C	80S20C	70S30C	90S10C	80S20C	70S30C	50S50C
Firing shrinkage (%)	0,5	0,3	0,3	5,1	2,6	1,7	1,5
Mass loss (%)	16,1	14,0	12,0	0,9*	0,7*	0,5*	9,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	- - -	- - -	24,6 28,3 0,87	25,8 29,5 0,88	26,3 29,9 0,88	24,3 27,6 0,88
Colour	brown	brown	brown	brown	brown	brown	brown
Remarks after firing	-	-	-	poor, crazed	poor, crazed	poor, crazed	fair, edge loss

* mass loss from 600°C

Table 8.6 : **Properties of cored bricks of sample F10 pressed at 400 kp/cm²**

PROPERTY	COMPOSITION 1000°C	
	75S25C	50S50C
Firing shrinkage (%)	7,5	6,4
Mass loss (%)	35,3	30,3
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	- - -
Colour	brown	brown
Remarks after firing	fair	fair

Table 8.7 : **Properties of cored bricks of sample F7 made from ½ to 2 mm fines and pressed at 450 kp/cm²**

PROPERTY	TEMPERATURE (°C)	
	900	950
Firing shrinkage (%)	6,2	7,2
Mass loss (%)	22,5	21,2
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	16,5 18,6 0,89	15,9 17,5 0,91
Colour	brown	brown
Remarks after firing	poor, cracked	unacceptable, cracked

Table 8.8 : Properties of cored bricks of sample F10 made from ½ to 2 mm fines and pressed at 450 kp/cm²

PROPERTY	TEMPERATURE (°C)	
	850	900
Firing shrinkage (%)	-	-
Mass loss (%)	19,8	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	20,8 21,9 0,96	- - -
Colour	brown	brown
Remarks after firing	unacceptable, broken	poor, cracked, cored

Table 8.9 : Properties of cored brick of sample F10 made from 2 to 5 mm fines and pressed at 450 kp/cm²

PROPERTY	TEMPERATURE (°C)
	900
Firing shrinkage (%)	4,9
Mass loss (%)	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	31,8 35,4 0,90
Colour	brown
Remarks after firing	good

8.4 DISCUSSION OF RESULTS

8.4.1 Forming properties

Drying shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. The brick drying shrinkages given in Tables 8.1 to 8.3 are at 3% or lower and are good.

The pressing of bricks proved to be the greatest area of difficulty of the whole investigation. The bricks pressed well in the die, but after having been removed from the die for several hours they might crack badly. It is believed that this difficulty has been overcome see Conclusion 10.5 a) but it has not been possible at this stage to produce full size bricks to prove conclusive success due to time and financial constraints of the investigation. Most of the prepared bricks are not discussed because they were never fired because of this cracking.

In the area of drying of the pressed bricks it was originally thought that no difficulties were present but it was eventually found that this was the cause of the cracking of the unfired and some of the fired bricks. This was unexpected and very unusual because the drying shrinkage of the bricks is very low and would normally in the case of extruded bricks never cause such a problem. The drying cracks in some cases were not noticed because they were so fine and they closed up upon drying but they opened up again upon firing see Section 8.4.2.

The method to overcome the difficulty is to perforate the brick and we have been successful in producing rings of approximately 50 mm in height, a diameter of 100 mm and a thickness of 20 mm see Conclusion 10.5 a).

8.4.2 Brickmaking

In the area of firing it was found that if bricks were fired in a normal manner up to top temperature usually in the range of 900 to 1050°C they would crack badly (see Tables 8.4. to 8.8.) This difficulty was overcome in the case of tiles by introducing a calcination step at 600°C to burn off deflocculants and organic matter see Section 4. The firing cycle adopted was one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature. Though this had solved the cracking and warping of the tiles it was not successful with the solid bricks. It is believed that the main reason for this was that the bricks were already cracked after drying.

In Table 8.9 the 2 to 5 mm fines F7 brick fired at 900°C dried and fired well. However, the coarseness and the excessive water absorption of the brick would make it unsuitable as a normal building brick.

It is essential that attempts to eliminate the cracking of the bricks before and after firing be undertaken.

The colours of the fired bricks should be acceptable for bricks.

Based on the results for Section 10 we recommend that full-size perforated bricks be investigated before pilot plant trials are undertaken.

8.5 CONCLUSIONS

a) The pressing of bricks proved to be the greatest area of difficulty of the whole investigation. The bricks pressed well in the die but after being removed from the die for several hours they might crack badly. It is believed that this difficulty has been overcome see Conclusion 10.5 a) but it has not been possible at this stage to produce full size bricks to prove conclusive success due to time and financial constraints of the investigation.

b) In the area of drying of the pressed bricks it was originally thought that no difficulties were present but it was eventually found that this was the cause of the cracking of the unfired bricks. This was unexpected and very unusual because the drying shrinkage of the bricks is very low and would normally in the case of extruded bricks never cause such a problem. The method to overcome the difficulty is to perforate the brick and we have been successful in producing rings of approximately 50 mm in height, a diameter of 100 mm and a thickness of 20 mm see Conclusion 10.5 a).

c) In the area of firing it was found that if bricks were fired in a normal manner up to top temperature usually in the range of 900 to 1000°C they would crack badly. This difficulty was overcome in the case of tiles by introducing a calcination step at 600°C to burn off deflocculants and organic matter. The firing cycle adopted was one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature. Though this had solved the cracking and warping of the tiles it was not successful with the solid bricks.

d) The colours of the fired bricks should be acceptable for bricks.

8.6 RECOMMENDATIONS

a) It is essential that attempts be made to eliminate the cracking of the bricks before and after firing.

b) We recommend that full-size perforated bricks be investigated before pilot plant trials are undertaken.

9. DRYING OF TILES, PAVERS AND BRICKS

9.1 SCOPE

To determine the effects of drying on tiles, pavers and bricks. To implement Recommendation 7.6 a) i.e. that the relationship between the quality of the paver and the mass of the paver be further investigated.

9.2 EXPERIMENTAL WORK

9.2.1 Sample preparation

For the preparation of the units dried sludge was first hammer milled until all material passed through a 0,85 mm sieve. It was then mixed by hand with a 5 or 15% solution of Teepol and water and left overnight. The solution contains 20% by mass of Teepol. The dimensions of the die was 116,4 by 243,3 mm (surface area 28 320 mm^2). Tiles of 0,5 kg, pavers of 1 and 2 kg, and bricks of 3 kg were then pressed at a pressure of 200 kp/cm^2. The samples were sealed in plastic bags after pressing awaiting the drying experiments.

9.2.2 Drying

A set of samples were dried in air at room laboratory temperature for at least one week followed by drying overnight at 110°C. Two sets of samples were dried in a humidity and temperature controlled dryer at 70% relative humidity (RH) and 40°C, and 100% RH and 25°C, respectively. Another set of samples were dried in a humidity and temperature controlled incubator at 100% RH and 35°C. Another two sets of samples were also dried in a humidity and temperature controlled room at 70% RH and 20°C. In these experiments when drying stopped the relative humidities were decreased.

9.3 RESULTS

9.3.1 Forming properties

The results obtained on the F15T pressed units are given in Table 9.1. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F15T means 15% water/Teepol was added to unfired sludge F on a dry basis.

Table 9.1 : Summary of forming properties of sample F15T used for drying at 70% relative humidity and 40°C

FORMING PROPERTY	UNITS (kg)				
	0,5	1	2	3	3
Pressing	good	good	good	bad, cracke d	bad, cracke d
Water content (%)	21,4	21,5	21,8	21,8	20,9
Drying shrinkage (%)	3,0	2,6	2,4	2,3	2,2
Colour	grey brown	grey brown	grey brown	grey brown	grey brown

The dial reading of 200 kp/cm^2 corresponds to a pressure 12,0 MPa (340 000 N/28 320 mm^2).

9.3.2 Drying properties

The results obtained on the dried units are given in Tables 9.2 to 9.8 and are graphically presented in Figures 9.1 to 9.5.

Table 9.2 : Drying properties of F15T units in air

Time (hrs)	0,5 kg tile		1 kg paver		2 kg paver		3 kg brick		3 kg brick with 20% calcines	
	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)
0	511,9	14,2	1175,5	14,1	2355,0	14,3	3529,0	14,2	3502,5	13,3
1	508,1	13,4	1171,5	13,7	2349,9	14,1	3523,5	14,0	3496,1	13,1
2	505,7	12,8	1169,0	13,5	2346,9	13,9	3518,6	13,9	3491,7	13,0
3	503,0	12,2	1166,0	13,2	2343,0	13,7	3513,7	13,7	3486,0	12,8
4	500,6	11,7*	1163,4	13,0*	2339,7	13,6*	3508,8	13,6*	3480,4	12,6*
5	498,3	11,2	1160,9	12,7	2336,4	13,4	3503,7	13,4	3474,9	12,5
6	495,8	10,6	1158,3	12,5	2332,9	13,2	3498,8	13,2	3469,1	12,3
7	493,3	10,1	1155,6	12,2	2329,1	13,1	3492,7	13,0	3463,23	12,1
8	491,2	9,6	1153,2	12,0	2325,5	12,9	3487,9	12,9	457,7	11,9
21	477,6	6,6	1134,3	10,1	2295,5	11,4	3446,0	11,5	3409,5	10,3
22	476,4	6,3	1132,8	10,0	2292,4	11,3	3443,0	11,4	3406,4	10,2
23	475,5	6,1	1131,6	9,9	2290,8	11,2	3440,9	11,4	3403,6	10,1
24	473,9	5,7	1129,1	9,6	2287,9	11,1	3435,6	11,2	3397,4	9,9
25	473,0	5,5	1127,6	9,5	2286,0	11,0	3433,3	11,1	3394,7	9,9
26	471,7	5,2	1125,6	9,3	2283,2	10,8	3429,9	11,0	3390,6	9,7
27	469,1	4,7	1122,0	8,9	2279,3	10,6	3423,9	10,8	3384,1	9,5
28	466,4	4,1	1118,4	8,6	2274,3	10,4	3416,1	10,6	3375,8	9,2

* cracked

Table 9.3 : Drying properties of F15T units at 70% relative humidity and 40°C

Time (hrs)	0,5 kg tile		1 kg paver		2 kg paver		3 kg brick		3 kg brick with 20% calcines	
	mass	H_2O	mass	H_2O	mass	H_2O	mass	H_2O	mass	H_2O
0	577,3	21,3	1176,01	21,5	2354,7	21,7	3532,7	21,8	3533,2	20,8
17	533,7	12,1	115,1	15,2	2296,6	18,7*	3465,4	19,4*	3448,3	17,9*
24	522,7	9,8	1096,7	13,3	2269,2	17,3	3433,2	18,3	3406,3	16,5
49	501,3	5,3	1048,0	8,2	2186,7	13,0	3325,8	14,6	3292,5	12,6
77	501,3	5,3	1035,5	7,0	2153,8	11,3	3281,5	13,1	3245,7	11,0
89	500,0	5,0	1029,3	6,3	2139,7	10,6	3261,6	12,4	3225,6	10,3

* cracked

Table 9.4 : Drying properties of F15T units at 100% RH and 25°C*

Time (hrs)	0,5 kg tile		1 kg paver		2 kg paver		3 kg brick	
	mass	H_2O	mass	H_2O	mass	H_2O	mass	H_2O
0	594,4	19,5	1176,6	22,1	2354,5	22,0	3534,2	22,1
17	583,8	17,4	1154,9	19,8	2332,9	20,8	3505,9	21,1
24	579,1	16,5	1145,8	18,9	2322,7	20,3	3492,9	20,6
42	567,9	14,2	1125,5	16,8	2294,7	18,9	3457,8	19,4
48	566,0	13,8	1120,7	16,3	2287,5	18,5	3448,5	19,1
67	564,4	13,5	1113,2	15,5	2273,4	17,8	3429,3	18,4
96	571,9	15,0	1120,5	16,3	2279,5	18,1	3433,4	18,6
114	565,6	13,8	1111,7	15,3	2267,4	17,4	3417,3	18,0
120	563,0	13,2	1107,7	14,9	2260,9	17,1	3408,5	17,7
137	558,4	12,3	1098,1	13,9	2244,9	16,3	3387,6	17,0
144	559,2	12,5	1098,5	14,0	2243,2	16,2	3385,4	16,9
161	553,6	11,3	1088,6	12,9	2226,9	15,3	3363,7	16,2
169	549,9	10,6	1082,5	12,3	2216,8	14,8	3350,7	15,7
185	544,3	9,5	1072,3	11,3	2199,5	13,9	3328,5	15,0
193	542,6	9,1	1068,4	10,9	2192,3	13,6	3318,5	14,6
210	539,1	8,4	1060,9	10,1	2178,2	12,8	3300,1	14,0
216	541,1	8,8	1061,7	10,2	2177,6	12,8	3298,9	13,9
234	538,7	8,3	1056,4	9,6	2167,0	12,2	3284,1	13,4
281	532,3	7,1	1041,9	8,1	2136,7	10,7	3242,8	12,0
305	530,4	6,7	1036,3	7,5	2124,2	10,0	3225,2	11,4
312	530,2	6,6	1035,0	7,4	2120,7	9,8	3220,6	11,2
331	527,0	6,0	1029,0	6,8	2108,8	9,2	3204,2	10,7
355	525,2	5,6	1024,5	6,3	2097,0	8,6	3187,5	10,1
379	524,1	5,4	1020,5	5,9	2087,0	8,1	3173,2	9,6
456	523,3	5,2	1016,1	5,4	2066,9	7,1	3141,2	8,5
480	522,7	5,1	1014,4	5,3	2060,6	6,7	3131,2	8,1
504	522,3	5,0	10,13,3	5,1	2054,9	6,4	3122,2	7,8
522	518,3	4,2	1007,7	4,6	2043,8	5,9	3106,0	7,3
525	516,2	3,8	1004,8	4,3	2039,3	5,6	3099,8	7,1
528	513,0	3,2	1000,1	3,8	2032,3	5,3	3090,9	6,7
530,5	511,0	2,8	997,5	3,5	2026,8	5,0	3082,9	6,5
545,5	505,7	1,7	983,9	2,1	1994,8	3,3	3036,4	4,9
547,5	504,2	1,4	981,4	1,8	1989,3	3,0	3028,3	4,6
550,5	503,1	1,2	978,9	1,6	1981,5	2,6	3015,9	4,2
552,5	502,9	1,1	977,9	1,5	1977,3	2,4	3008,4	3,9
553,75	501,0	0,8	975,4	1,2	1972,3	2,2	3000,3	3,6
622,5	497,2	0,0	963,8	0,0	1930,6	0,0	2895,5	0,0

* no cracking occurred in the humidity dryer, after 622 hrs the samples were placed in a normal dryer and the 2 and 3 kg samples cracked

Table 9.5 : Drying properties of F15T units at 100% relative humidity and 35°C*

Time (hrs)	0,5 kg tiles		1 kg paver		2 kg paver		3 kg brick	
	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)
0	585,6	13,7	1173,5	13,9	2353,0	14,2	3498,2	13,2
19	584,6	13,5	1172,2	13,8	2356,5	14,4	3501,6	13,3
25	583,6	13,3	1171,2	13,7	2355,2	14,3	3499,3	13,2
43	582,9	13,2	1169,7	13,6	2350,4	14,1	3495,6	13,1
48	582,3	13,1	1168,6	13,5	2347,8	14,0	3492,8	13,0
115	575,3	11,7	1155,4	12,2	2319,6	12,6	3468,6	12,3
139	573,2	11,3	1152,0	11,8	2311,9	12,2	3460,4	12,0
145	571,1	11,0	1149,9	11,6	2308,4	12,1	3456,5	11,9
164	569,5	10,6	1145,9	11,3	2300,4	11,7	3447,3	11,6
188	564,7	9,7	1138,2	10,5	2287,2	11,0	3434,0	11,1
212	561,9	9,1	1133,3	10,0	2277,9	10,6	3422,9	10,8
284	559,7	8,7	1126,5	9,4	2261,0	9,8	3401,0	10,1
288	557,5	8,3	1123,9	9,1	2257,7	9,6	3396,5	9,9
312	554,2	7,6	1117,8	8,5	2247,3	9,1	3384,0	9,5
336	551,8	7,1	1112,9	8,0	2241,2	8,8	3366,9	9,0
360	548,8	6,6	1107,3	7,5	2231,3	8,3	3354,7	8,6
456	541,4	5,1	1090,6	5,9	2197,2	6,7	3311,4	7,2

* did not crack after 456 hours

Table 9.6 : Drying properties of F5T units at 100% relative humidity and 35°C*

Time (hrs)	0,5 kg tile		1 kg paver		2 kg paver		3 kg brick	
	mass	H_2O	mass	H_2O	mass	H_2O	mass	H_2O
0	513,6	1,7	1050,8	4,0	2112,3	4,6	3167,2	4,5
19	527,0	4,4	1065,4	5,5	2135,3	5,7	3193,6	5,4
25	529,4	4,8	1067,9	5,7	2138,7	5,9	3197,1	5,5
43	534,2	5,8	1075,8	6,5	2149,4	6,4	3209,4	5,9
48	534,7	5,9	1076,8	6,6	2150,3	6,5	3210,0	5,9
115	537,0	6,3	1083,8	7,3	2164,9	7,2	3227,8	6,5
139	536,1	6,2	1083,7	7,3	2167,6	7,3	3230,5	6,6
145	535,7	6,1	1082,7	7,2	2167,0	7,3	3230,0	6,6
164	534,8	5,9	1081,0	7,0	2166,1	7,2	3230,5	6,6
188	533,3	5,6	1077,9	6,7	2163,9	7,1	3229,2	6,6
212	532,5	5,4	1076,3	6,6	2163,3	7,1	3228,6	6,6
284	531,6	5,3	1075,9	6,5	2161,0	7,0	3221,0	6,3
288	530,3	5,0	1073,2	6,3	2158,7	6,9	3225,6	6,5
312	528,6	4,7	1070,7	6,0	2155,7	6,7	3221,1	6,3
336	527,1	4,4	1068,3	5,8	2151,9	6,5	3216,5	6,2
360	525,6	4,1	1065,9	5,5	2147,8	6,3	3211,9	6,0
456	522,3	3,4	1059,2	4,9	2136,6	5,8	3195,8	5,5

Table 9.7 : Drying properties of F5T units at 67% relative humidity and 21°C*

Time (hrs)	0,5 kg tiles		1 kg paver		2 kg paver		3 kg brick	
	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)	mass	H_2O (%)
0	482,5	4,3	1049,5	3,9	2112,5	4,6	3170,7	4,6
24	482,2	4,3	1048,5	3,8	2112,2	4,5	3168,4	4,6
48	481,3	4,1	1048,0	3,8	2110,3	4,5	3166,6	4,5
72	481,2	4,0	1047,9	3,8	2109,9	4,5	3165,8	4,5
96	481,2	4,0	1047,8	3,7	2109,5	4,4	3165,0	4,5
100	480,8	4,0	1047,1	3,7	2108,9	4,4	3163,9	4,4
116	481,8	4,2	1048,8	3,8	2110,7	4,5	3165,5	4,5
163	482,4	4,3	1050,3	4,0	2112,6	4,6	3167,5	4,5
166	481,7	4,2	1049,6	3,9	2111,6	4,5	3166,2	4,5
168	481,4	4,1	1048,8	3,8	2110,8	4,5	3165,6	4,5
170	480,4	3,9	1048,0	3,8	2109,4	4,4	3163,6	4,4

* did not crack after 170 hours

Table 9.8 : Drying properties of F15T units at 67% relative humidity and 21°C

Time (hrs)	0,5 kg tile		1 kg paver		2 kg paver		3 kg brick	
	mass	H_2O	mass	H_2O	mass	H_2O	mass	H_2O
0	585,5	13,7	1176,1	14,2	2114,7	14,1	3173,3	14,1
24	551,3	7,0	1132,9	10,0	2066,6	11,5*	3113,7	11,9*
48	539,6	4,8	1112,7	8,0	2040,0	10,0	3080,6	10,7
72	535,1	3,9	1099,8	6,8	2021,4	9,0	3057,4	9,9
96	533,3	3,6	1091,2	5,9	2007,0	8,3	3038,9	9.2
168	534,5	3,8	1082,5	5,1	1983,4	7,0	3006,8	8,1

* cracked

Figure 9.1 : Moisture content vs time
F15T units, air drying

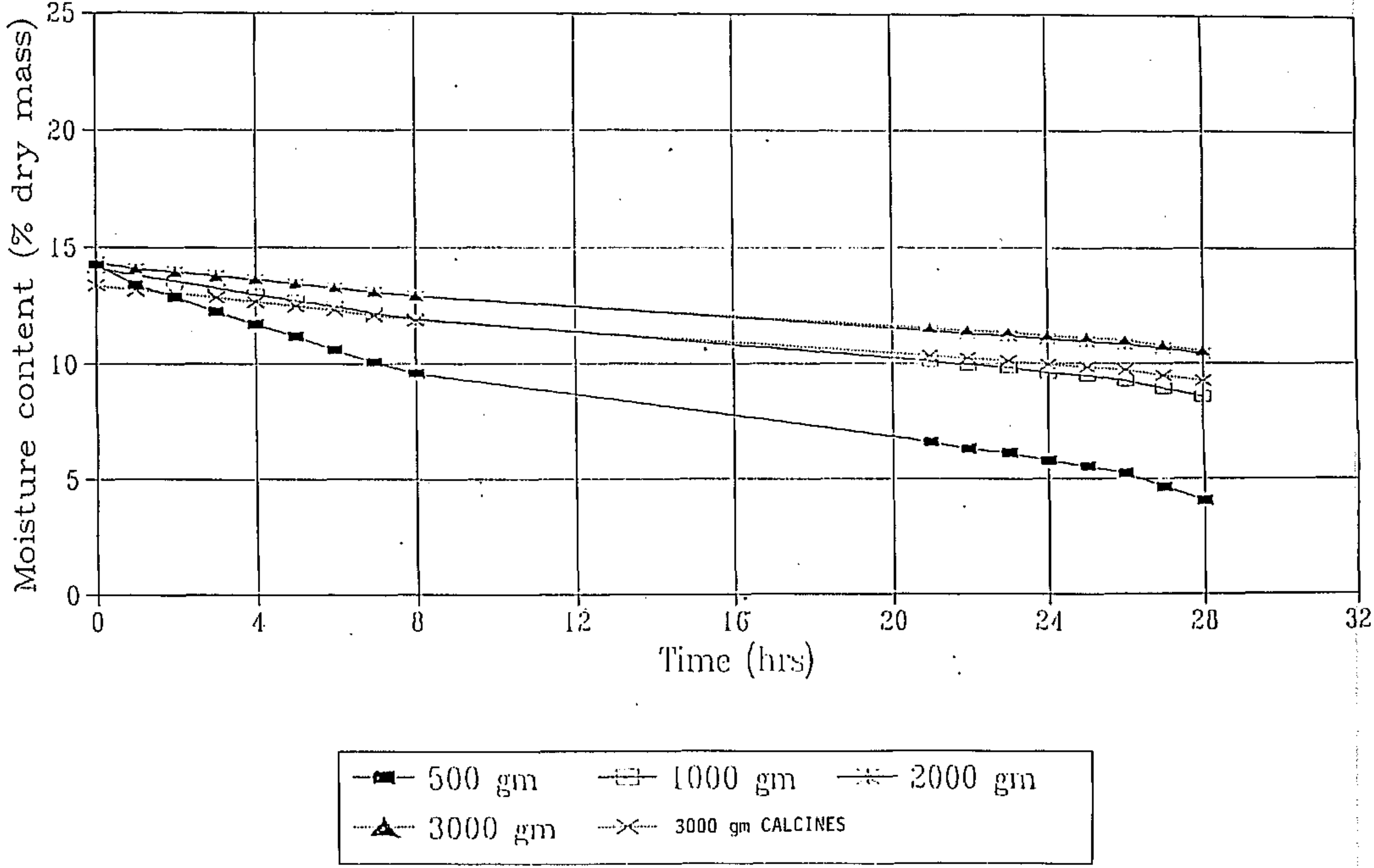


Figure 9.2 : Moisture content vs time
F15T units, 70% RH @ 40°C

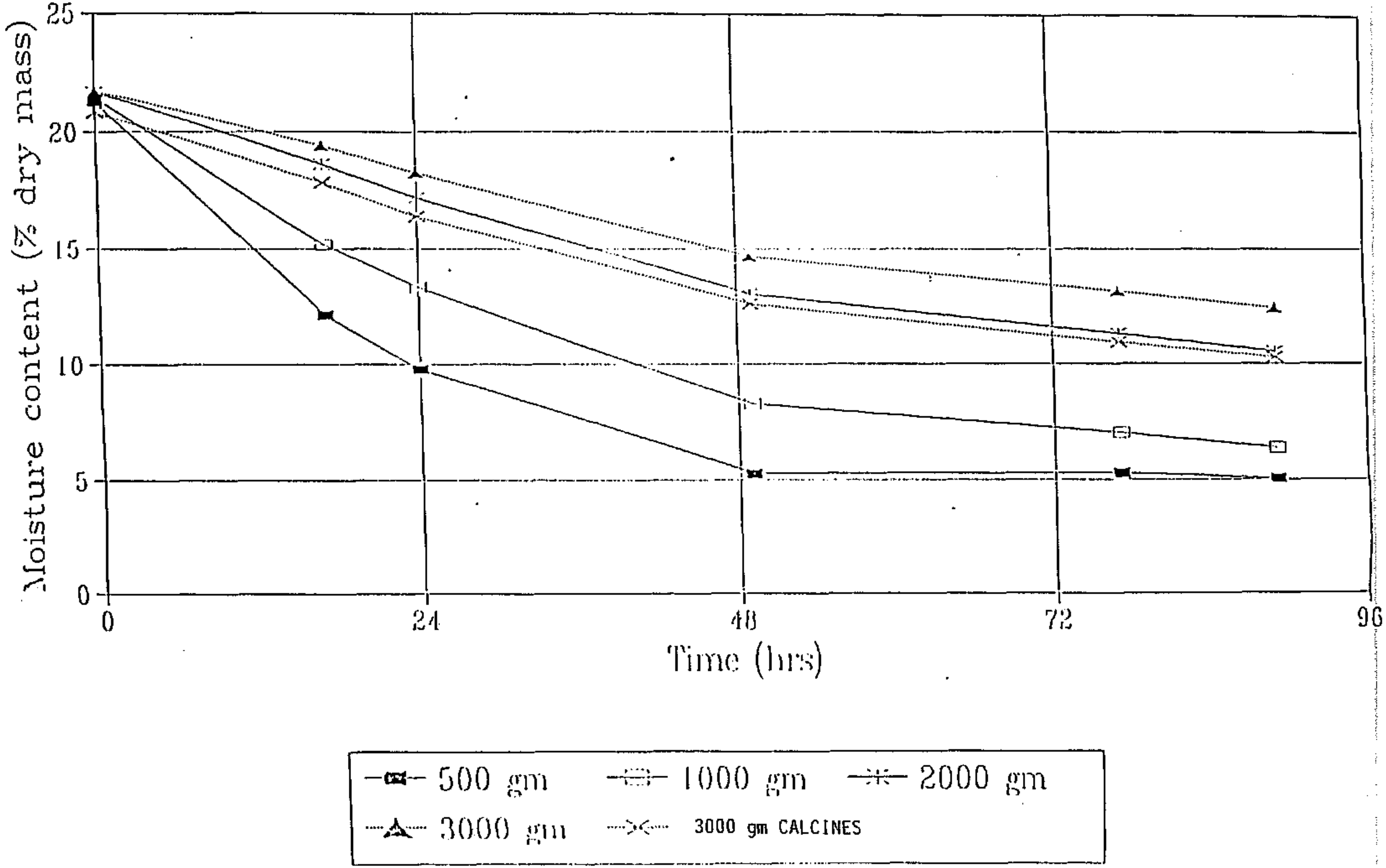


Figure 9.3 : Moisture content vs time
F15T units, 100% RH @ 25°C

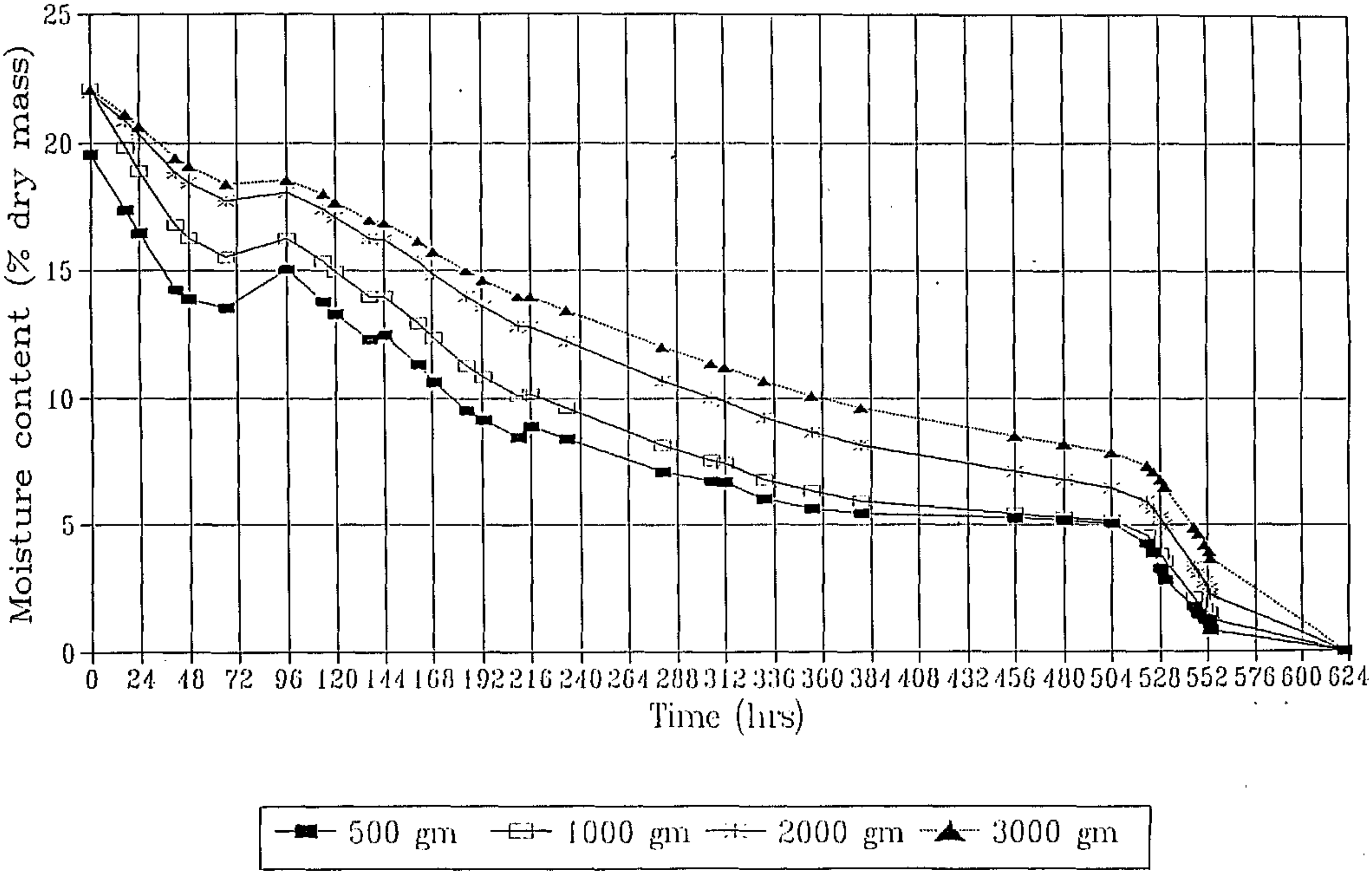


Figure 9.4 : Moisture content vs time
F15T units, 100% RH @ 35°C

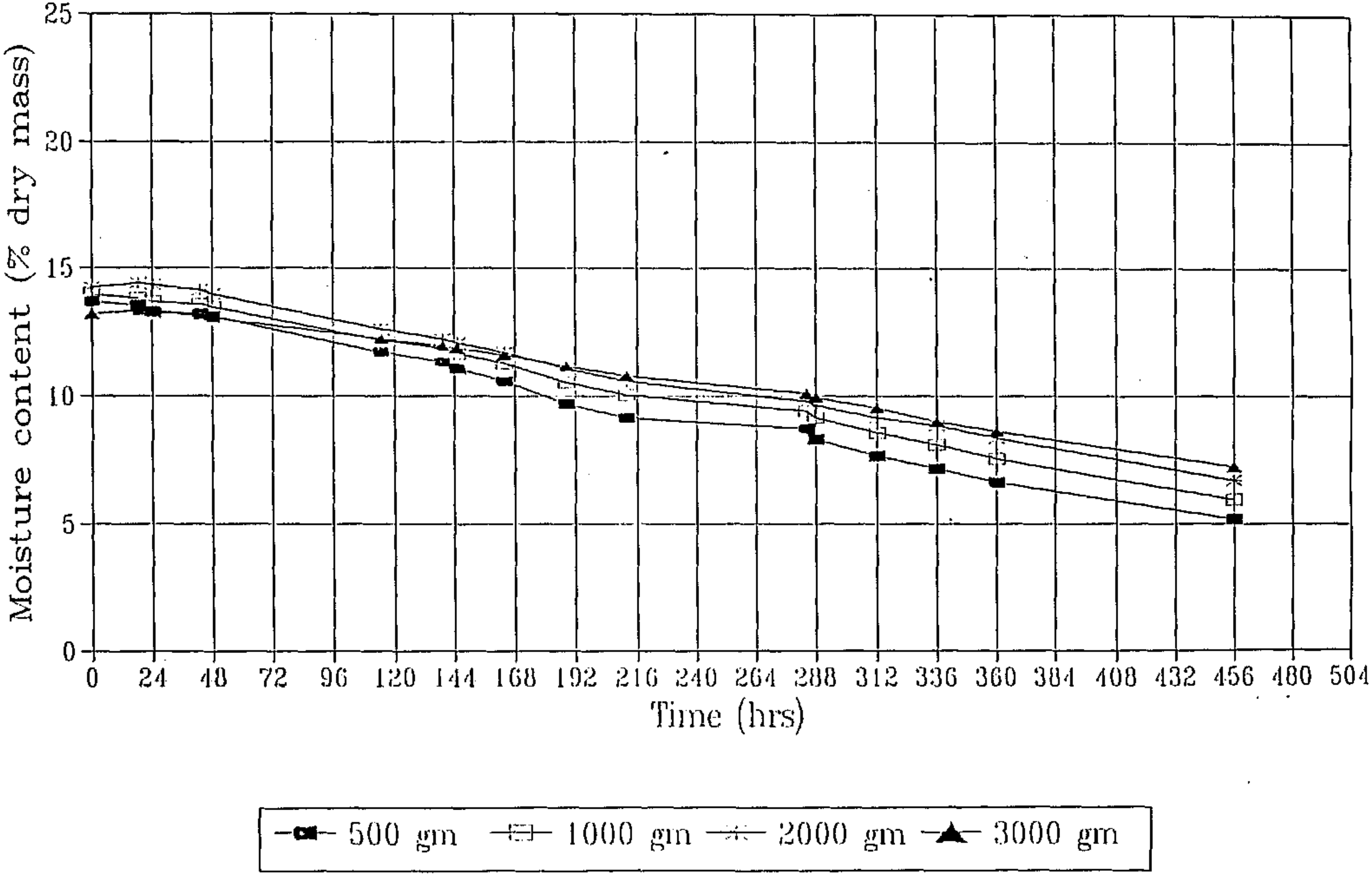
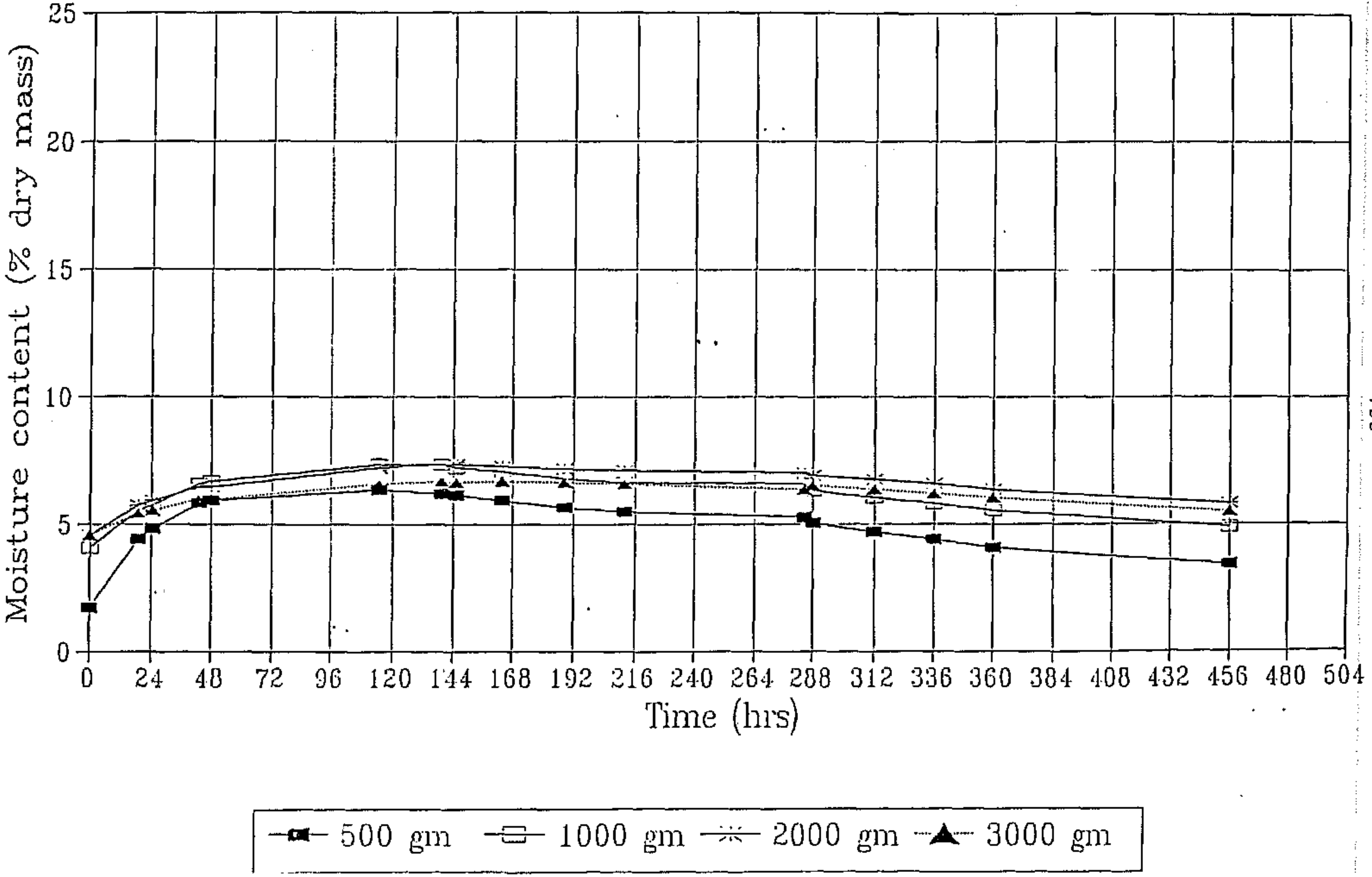


Figure 9.5 : Moisture content vs time
F5T units, 100% @ 35°C



9.4 DISCUSSION OF RESULTS

9.4.1 Drying properties

All the F15T samples which were dried at room temperature showed signs of cracking after 4 hours.

At 67% relative humidity and 21°C none of the F5T samples cracked after 170 hours. However, the 2 and 3 kg samples of F15T cracked after 24 hours.

At 70% relative humidity and 40°C the 2 and 3 kg F15T samples cracked after being left overnight.

At 100% relative humidity and 25°C after 622 hours no cracking of the F15T samples occurred in the humidity dryer but when the samples were placed in a normal dryer the 2 and 3 kg samples cracked. The samples are thus sensitive to temperature rise and precautions will have to be taken in practice to ensure that bricks made from these materials are not subjected to thermal shocks.

At 100% relative humidity and 35°C none of the F5T and F15T samples cracked after 456 hours.

It was found that humidity drying was very successful in stopping the drying cracking of the pavers and the bricks. Though humidity drying works it may not be economical to dry bricks in this way because of the length of time it takes.

An economical way to solve drying problems may be to perforate the brick.

We recommend that full-size perforated bricks be investigated before pilot plant trials are undertaken.

9.5 CONCLUSIONS

a) It was found that humidity drying was very successful in stopping the drying cracking of the pavers and the bricks. Though humidity drying works it may not be economical to dry bricks in this way.

b) An economical way to solve drying problems may be to perforate the brick.

9.6 RECOMMENDATION

a) We recommend that full size perforated bricks be investigated before pilot plant trials are undertaken.

10. PRESSING OF RINGS

10.1 SCOPE

To determine drying properties and firing range of pressed rings; and the effect of water and calcines used for pressing on the fired properties of these rings. To implement Recommendation 9.6 a) i.e. that full size perforated bricks be investigated before pilot plant trials are undertaken.

10.2 EXPERIMENTAL WORK

10.2.1 Sample preparation

For the preparation of rings the dried material and calcined material at 1050°C for 5 hours, were first hammer milled until all material passed through a 0,85 mm sieve. It was then mixed by hand with water varying from 5 up to 40% and left to temper overnight. Two similar series (I and II) of rings were then pressed with the tile press. The inner diameter dimension of the outer ring cylindrical die was 105,46 mm. The outer diameter dimension of the inner ring cylindrical die was 66,04 mm. The rings were then dried in air at room temperature for approximately two weeks (corresponded to high ambient humidity conditions) followed by drying 5 to 10°C per hour up to 110°C.

10.2.2 Firing

The dried rings were fired in a laboratory electric furnace. All the rings were cored out at 600°C, the time to reach this temperature was 24 hours and the samples were kept at this temperature for 24 hours and then they were allowed to cool to room temperature. A ring of each water content was fired at one or both of the following temperatures: 950 and 1000°C. The calcine rings were also fired at 1020°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

10.3 RESULTS

10.3.1 Forming properties

The results obtained on the Series II pressed rings are given in Table 10.1.

Table 10.1 : Summary of forming properties of Series II rings

Forming property	F5	F10	F15	F20	F25	F30	F35	F40
Pressing	good	good	good	good	good	good	good	poor
Dial reading (kp/cm^2)	50	50	50	50	50	50	50	50
Mixing water (%)	6,8	13,2	16,3	24,9	29,8	33,1	35,8	46,0
Green strength	good	good	good	good	good	good	good	good
Drying shrinkage (%)	1,1	2,1	2,0	3,4	3,2	4,3	4,9	4,9
Drying	good	good	good	good	good	good	good	good
Colour	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown	grey brown

The dial readings of 50 and 112 kp/cm^2 correspond to pressures of 15 (80 000 N/5 310 mm^2) and 35 MPa (188 000 N/5 310 mm^2) respectively.

The results obtained on the sludge/calcine pressed rings are given in Table 10.2. The number following the sample identification letter denotes the nominal percentage by mass of water used for pressing, eg. F20 80/20 means 20% water was added to the mix of 80% unfired sludge and 20% calcined sludge F on a dry basis.

Table 10.2 : Summary of forming properties of sludge/calcine rings

Forming property	F20 80/20	F20 90/10	F25 80/20	F25 90/10
Pressing	good	good	good	good
Dial reading (kp/cm^2)	50	50	50	50
Mixing water (%)	12,6	13,3	13,7	15,2
Green strength	good	good	good	good
Drying shrinkage (%)	0,8	1,3	1,2	1,8
Drying	good	good	good	good
Colour	grey brown	grey brown	grey brown	grey brown

10.3.2 Fired properties

The results obtained on the fired rings are given in Tables 10.3 to 10.14, and are graphically presented in Figures 10.1 to 10.10.

Table 10.3 : Fired properties of the rings for sample F5

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 I	1050
Firing shrinkage (%)	1,4	2,2	9,3	15,4	
Mass loss (%)	14,6	18,1	19,5	20,1	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	48,5 51,1 0,95	33,1 34,5 0,96	18,8 19,9 0,94	
Colour	orange brown	orange brown	orange brown	orange brown	
Remarks after firing	good	good	good	good, small vertical inner cracks	

Table 10.4 : Fired properties of the rings for sample F10

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 I	1050
Firing shrinkage (%)	1,6	1,5	8,7	15,3	
Mass loss (%)	14,6	18,1	19,4	20,1	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	36,3 44,4 0,82	30,6 31,8 0,96	18,3 19,3 0,95	
Colour	orange brown	orange brown	orange brown	orange brown	
Remarks after firing	good	good	good	good, small vertical inner cracks	

Table 10.5 : Fired properties of the rings for sample F15

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 I	1050
Firing shrinkage (%)	1,9	1,2	8,4	15,2	
Mass loss (%)	14,6	18,1	19,5	20,0	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	45,4 47,9 0,95	30,7 32,1 0,96	17,4 18,6 0,94	
Colour	orange brown	orange brown	orange brown	orange brown	
Remarks after firing	good	good	good	good	

Table 10.6 : Fired properties of the rings for sample F20

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 II	1050
Firing shrinkage (%)	1,5	2,7	8,7	14,7	
Mass loss (%)	14,7	17,9	19,7	20,4	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	- - -	28,3 29,3 0,97	14,0 15,5 0,90	
Colour	orange brown	orange brown	orange brown	brown	
Remarks after firing	good, (drying) cracks	good	fair, drying cracks, vertical inner cracks	excellent, small vertical inner cracks	

Table 10.7 : Fired properties of the rings for sample F25

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 II	1050
Firing shrinkage (%)	1,6	2,4	8,2	16,0	
Mass loss (%)	14,6	17,9	19,7	20,3	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	- - -	27,1 28,1 0,97	11,5 12,9 0,89	
Colour	orange brown	orange brown	orange brown	brown	
Remarks after firing	good, < drying cracks	good	fair, drying cracks, vertical inner cracks	excellent, vertical inner cracks	

Table 10.8 : Fired properties of the rings for sample F30

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 II	1050
Firing shrinkage (%)		1,6		16,0	
Mass loss (%)		18,2		20,5	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		- - -		11,4 13,0 0,88	
Colour		orange brown		brown	
Remarks after firing		good		good, vertical inner cracks	

Table 10.9 : Fired properties of the rings for sample F35

PROPERTY	TEMPERATURE (°C)				
	600 II	600 II*	950 I	1000 II	1050
Firing shrinkage (%)	2,0	2,0		15,6	
Mass loss (%)	18,2	18,2		20,5	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	- - -	41,2 43,6 0,95		12,8 14,8 0,87	
Colour	-	orange brown		brown	
Remarks after firing	-	good		good, vertical inner cracks	

* Pressed at 112 kp/cm[2] by error, not further processed

Table 10.10 : Fired properties of the rings for sample F40

PROPERTY	TEMPERATURE (°C)				
	600 I	600 II	950 I	1000 II	1050
Firing shrinkage (%)		2,3		16,1	
Mass loss (%)		18,1		20,7	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		- - -		18,2 21,7 0,84	
Colour		orange brown		brown	
Remarks after firing		good		poor, drying cracks	

Table 10.11 : Fired properties of the rings for sample F20 80/20

PROPERTY	TEMPERATURE (°C)				
	500	600	950	1000	1020
Firing shrinkage (%)		0,4		6,1	-
Mass loss (%)		13,0		15,0	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		35,4 38,0 0,93		25,1 29,1 0,86	24,6 28,5 0,86
		orange brown		brown	brown
Remarks after firing		good		poor, crazed	poor, crazed

Table 10.12 : Fired properties of the rings for sample F20 90/10

PROPERTY	TEMPERATURE (°C)				
	500	600	950	1000	1020
Firing shrinkage (%)		1,4		11,6	-
Mass loss (%)		17,0		19,6	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		41,5 44,0 0,94		20,6 22,8 0,90	18,1 20,1 0,90
Colour		orange brown		brown	brown
Remarks after firing		good		fair, crazed	fair, crazed

Table 10.13 : Fired properties of the rings for sample F25 80/20

PROPERTY	TEMPERATURE (°C)				
	500	600	950	1000	1020
Firing shrinkage (%)		0,0		3,5	-
Mass loss (%)		12,8		14,8	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		34,7 37,4 0,93		24,6 28,6 0,86	22,8 27,1 0,84
Colour		orange brown		brown	brown
Remarks after firing		good		poor, crazed	poor, crazed

Table 10.14 : Fired properties of the rings for sample F25 90/10

PROPERTY	TEMPERATURE (°C)				
	500	600	950	1000	1020
Firing shrinkage (%)		0,9		9,7	-
Mass loss (%)		16,9		19,4	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient		41,8 44,5 0,94		18,7 20,8 0,90	17,4 19,5 0,89
Colour		orange brown		brown	brown
Remarks after firing		good		fair, crazed	fair, crazed

Figure 10.1 : Water absorption vs mix water content (rings)

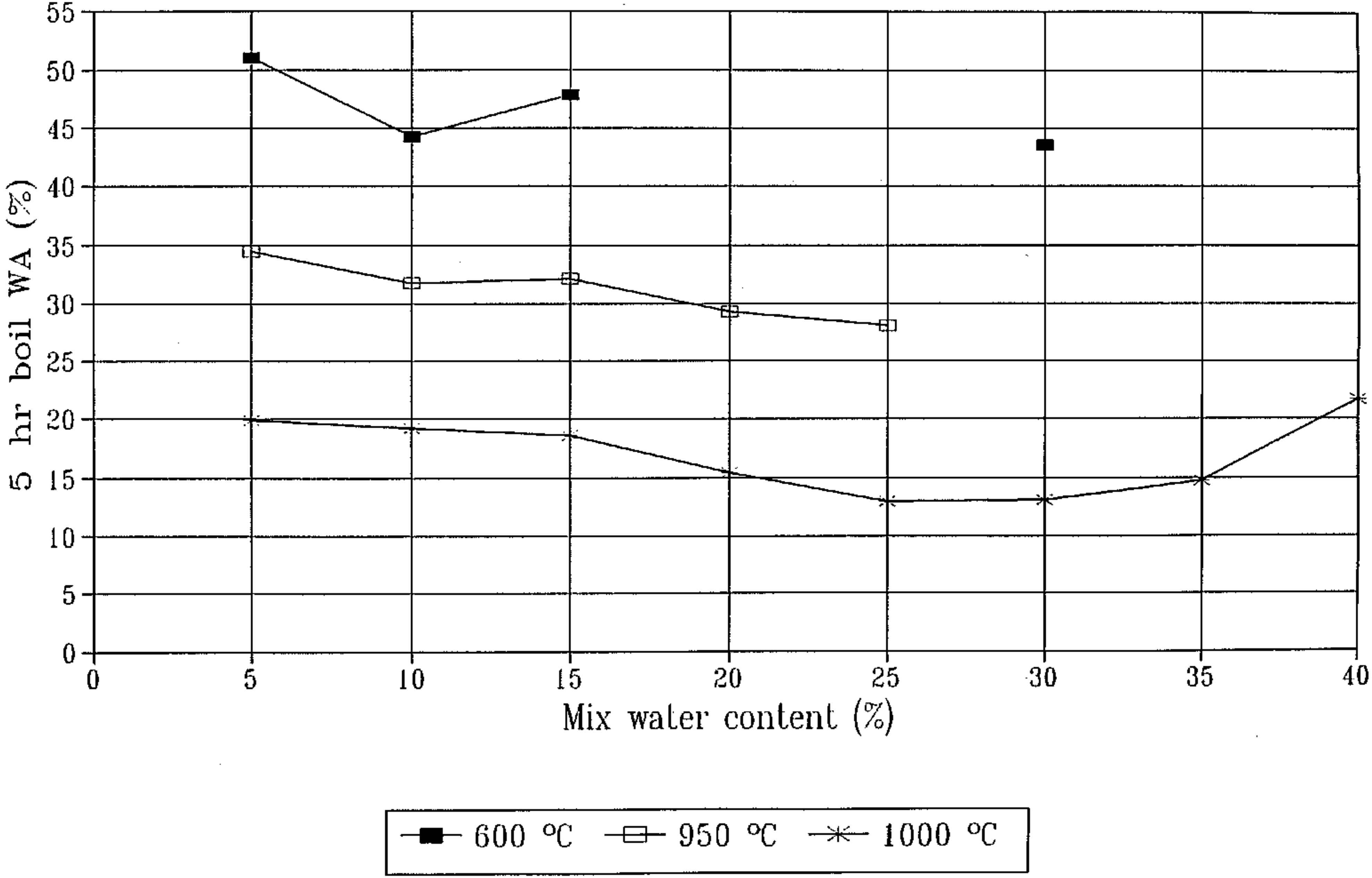


Figure 10.2 : Water absorption vs firing temperature (rings)

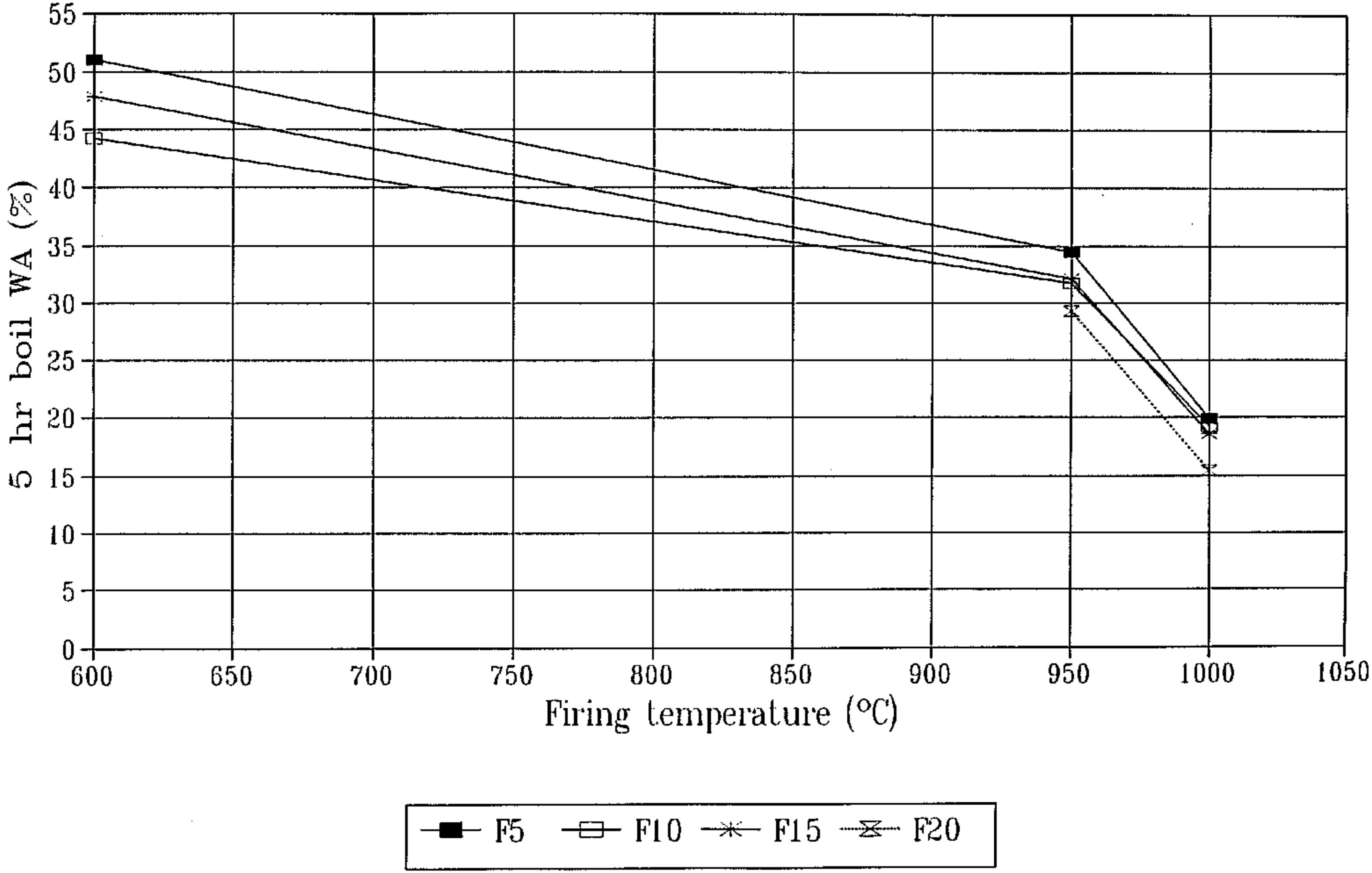


Figure 10.3 : Water absorption vs firing temperature (rings)

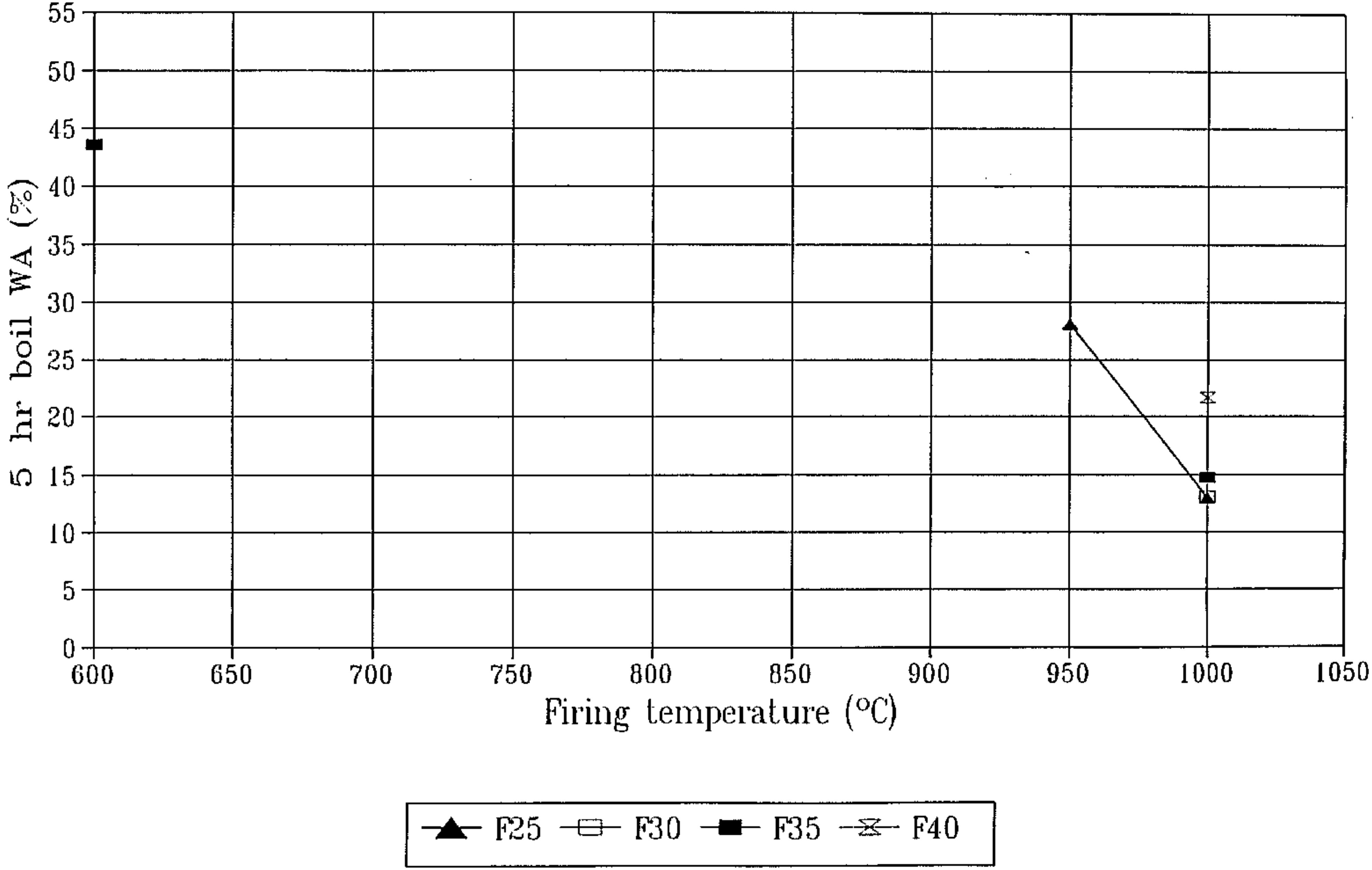


Figure 10.4 : Drying & firing
shrinkage vs mix water content (rings)

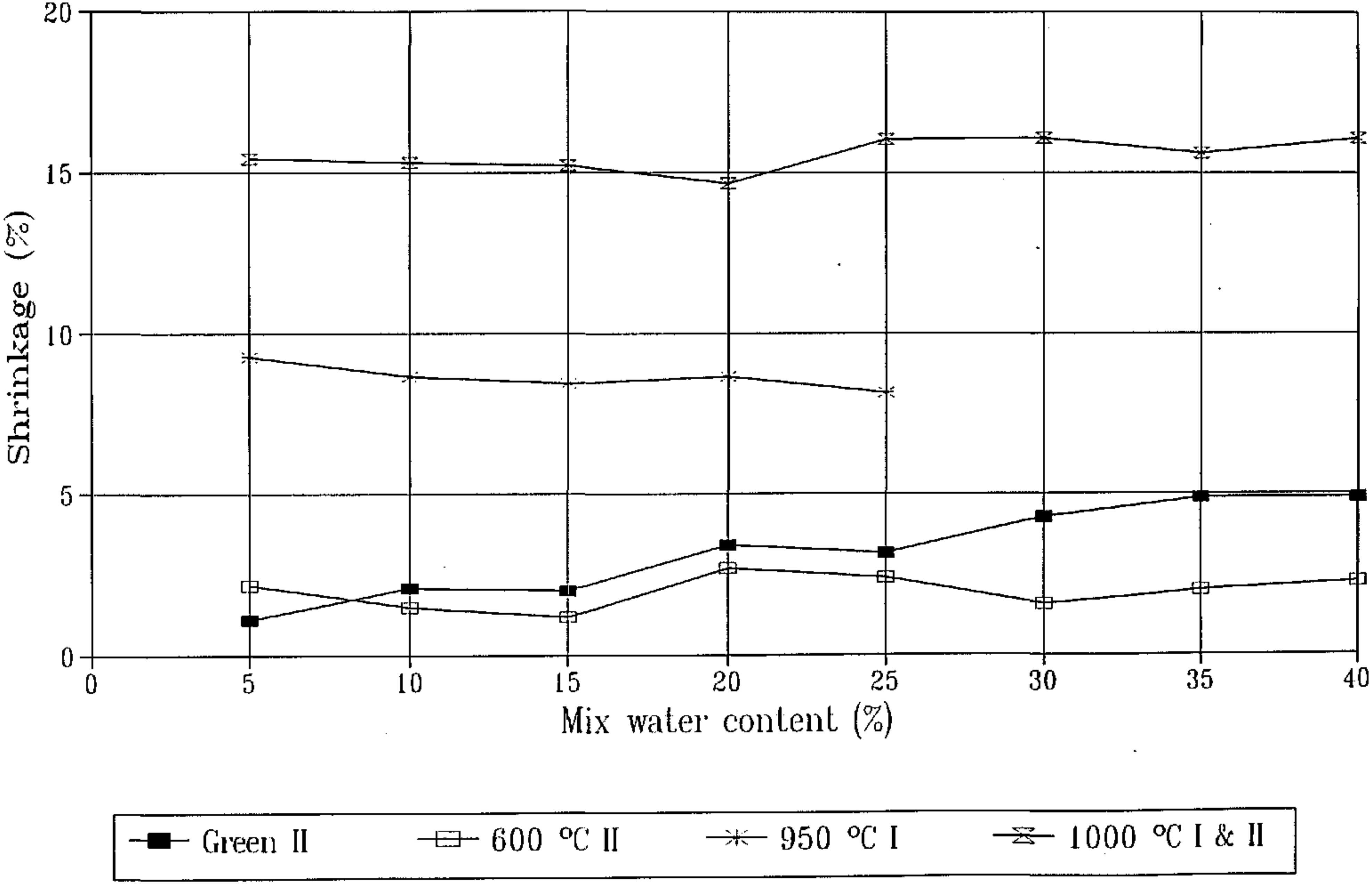


Figure 10.5 : Firing shrinkage vs firing temperature (rings)

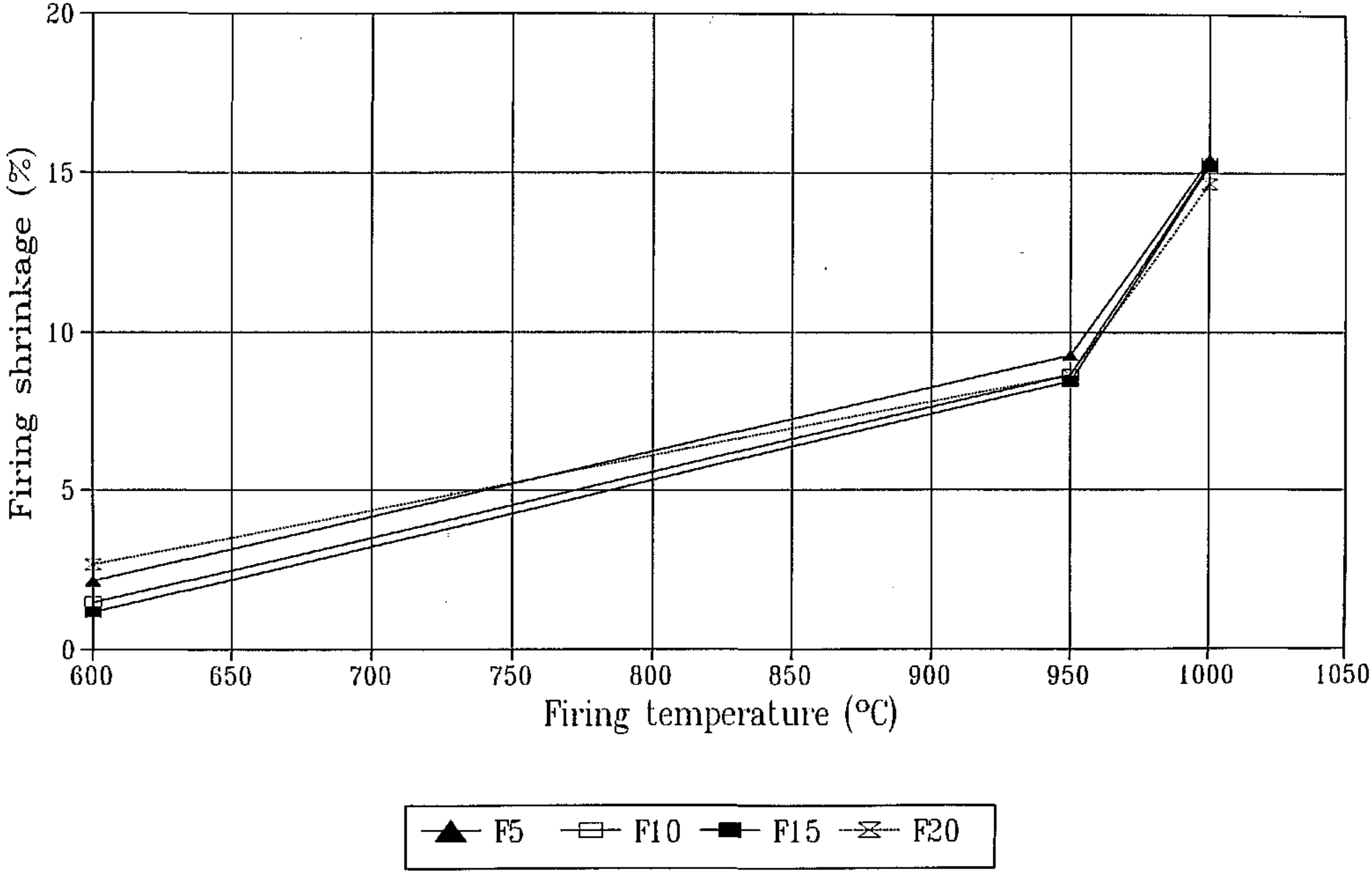


Figure 10.6 : Firing shrinkage vs firing temperature (rings)

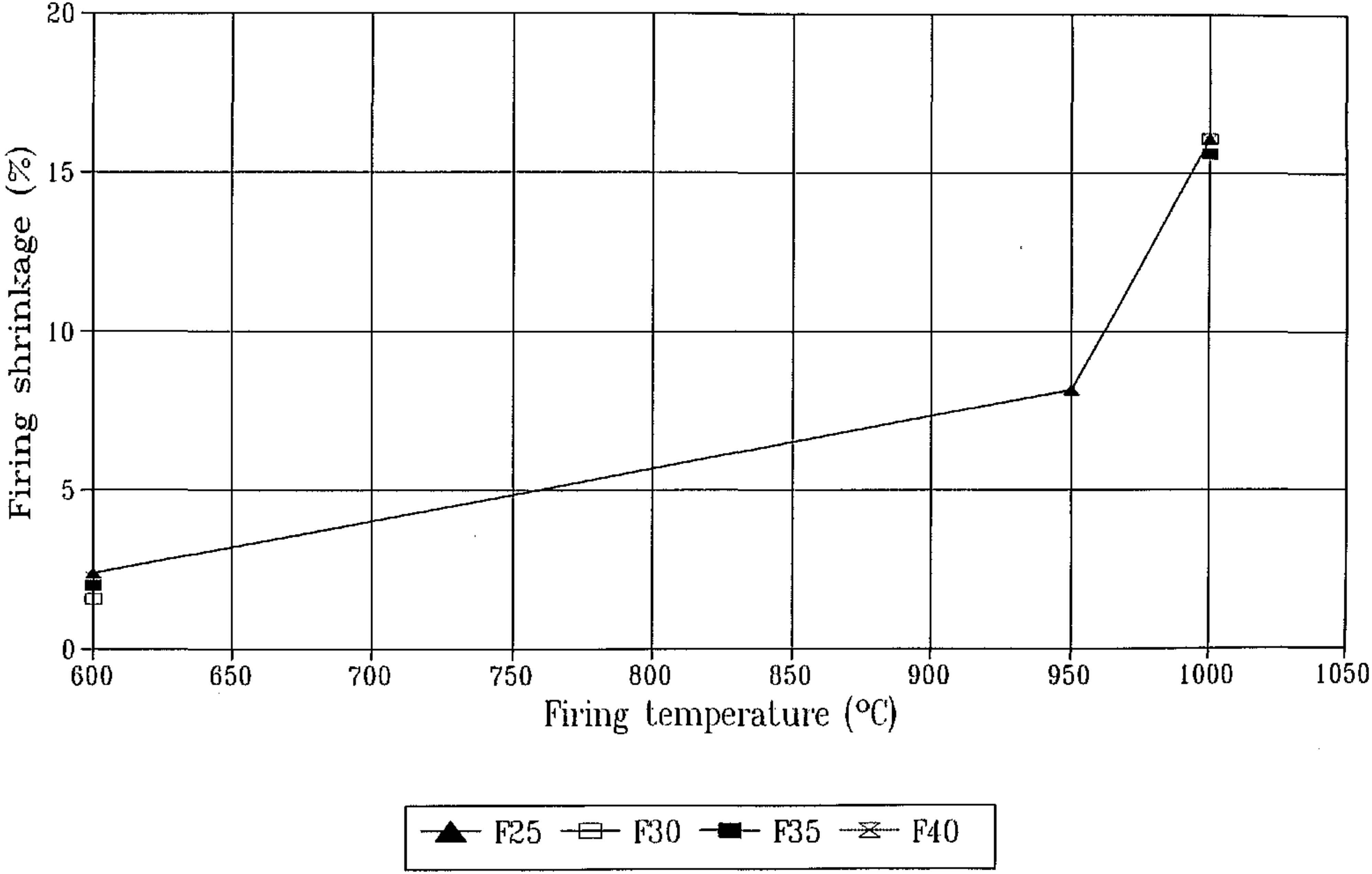


Figure 10.7 : Water absorption vs mix water content (calcine rings)

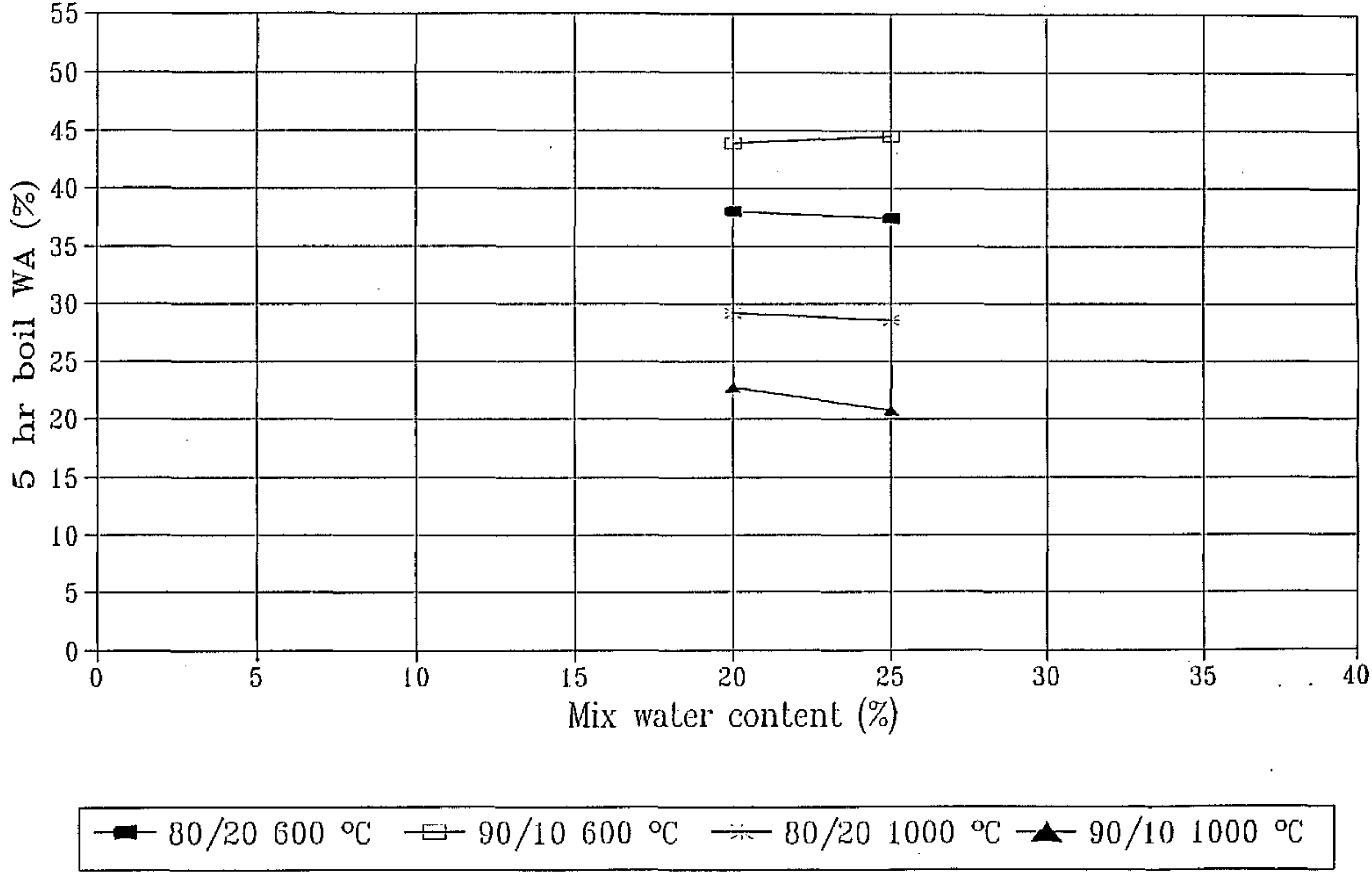


Figure 10.8 : Drying & firing shr.
vs mix water content (calcine rings)

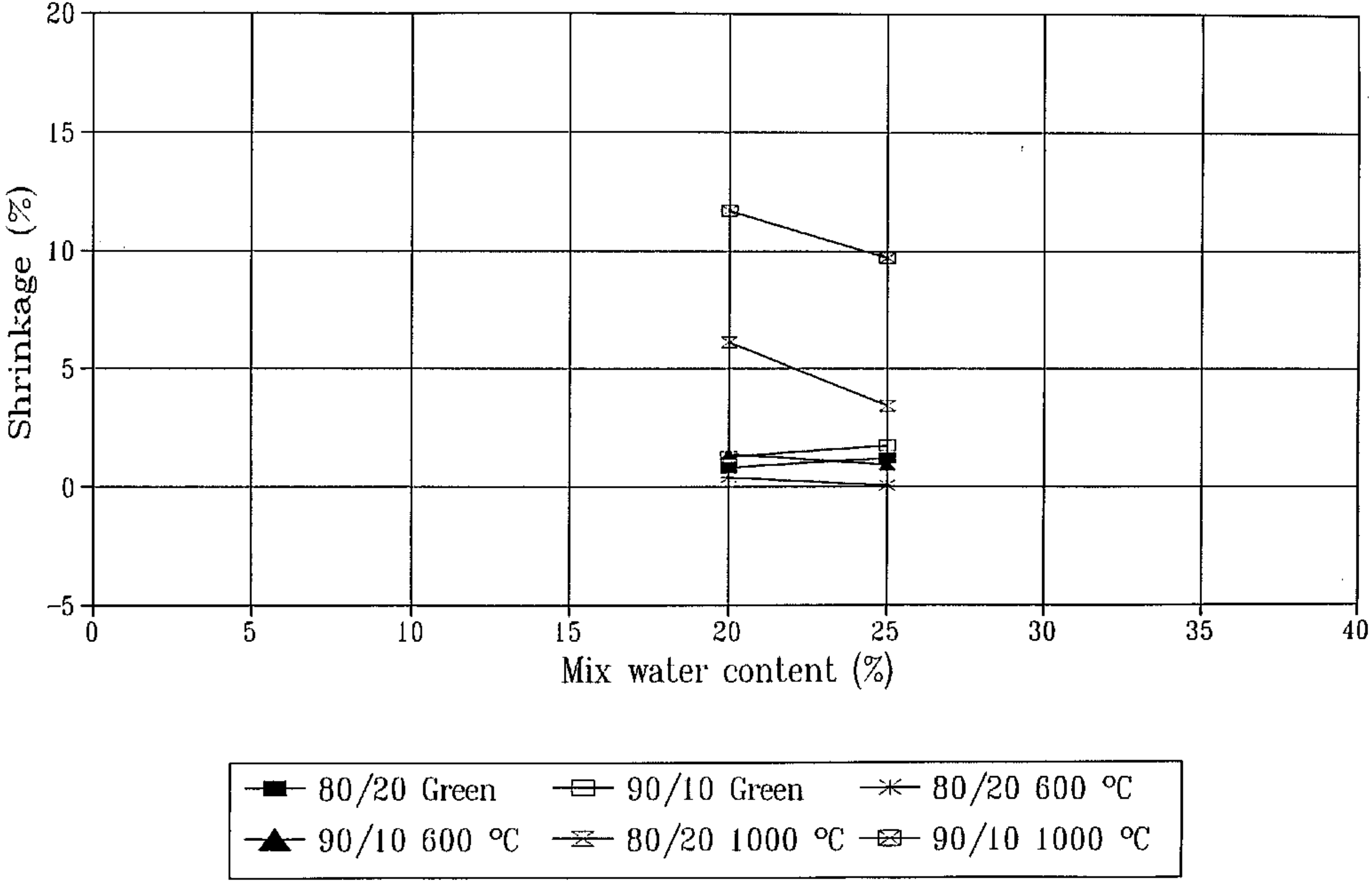


Figure 10.9 : Water absorption vs calcine content (calcine rings)

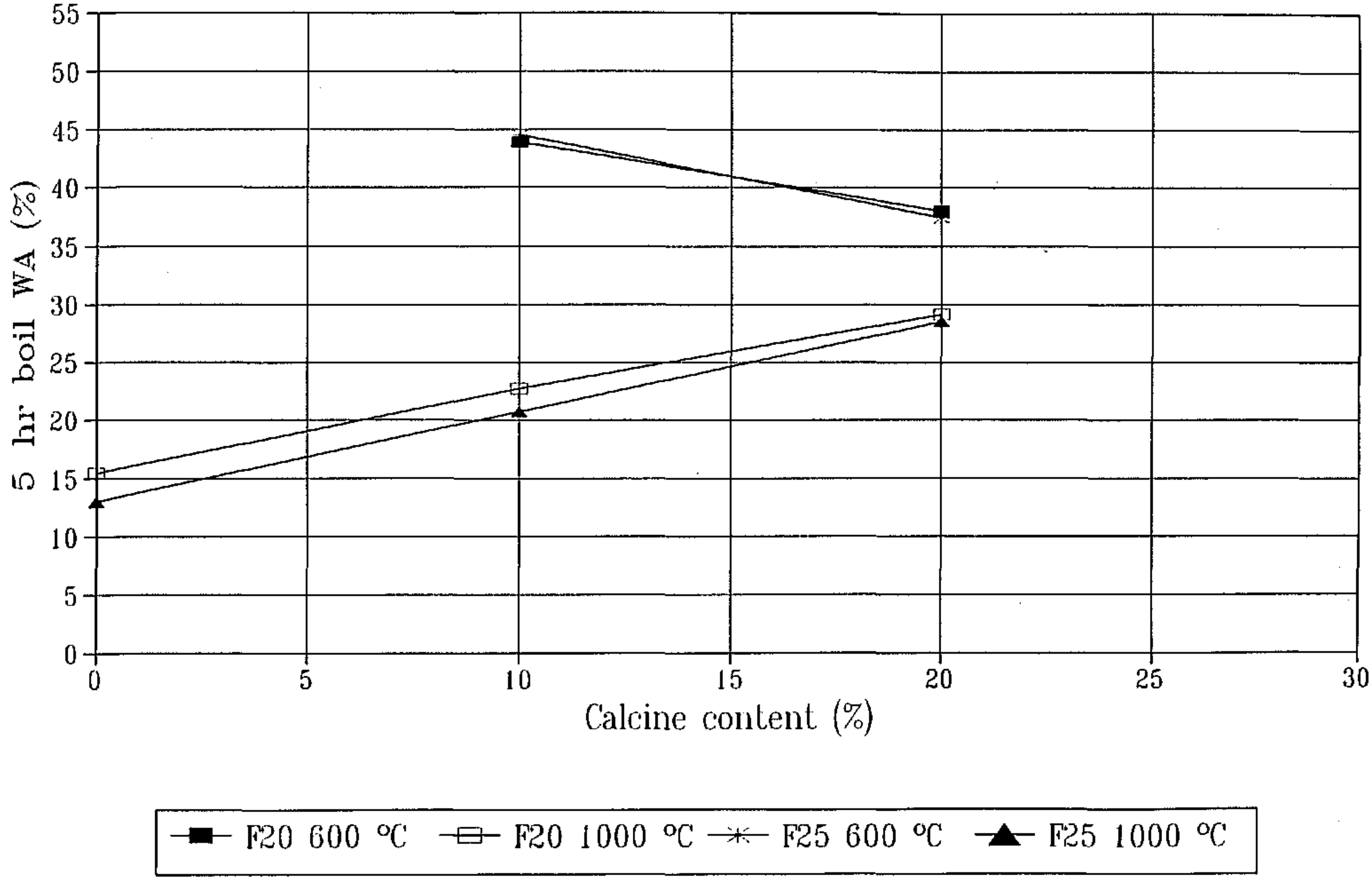
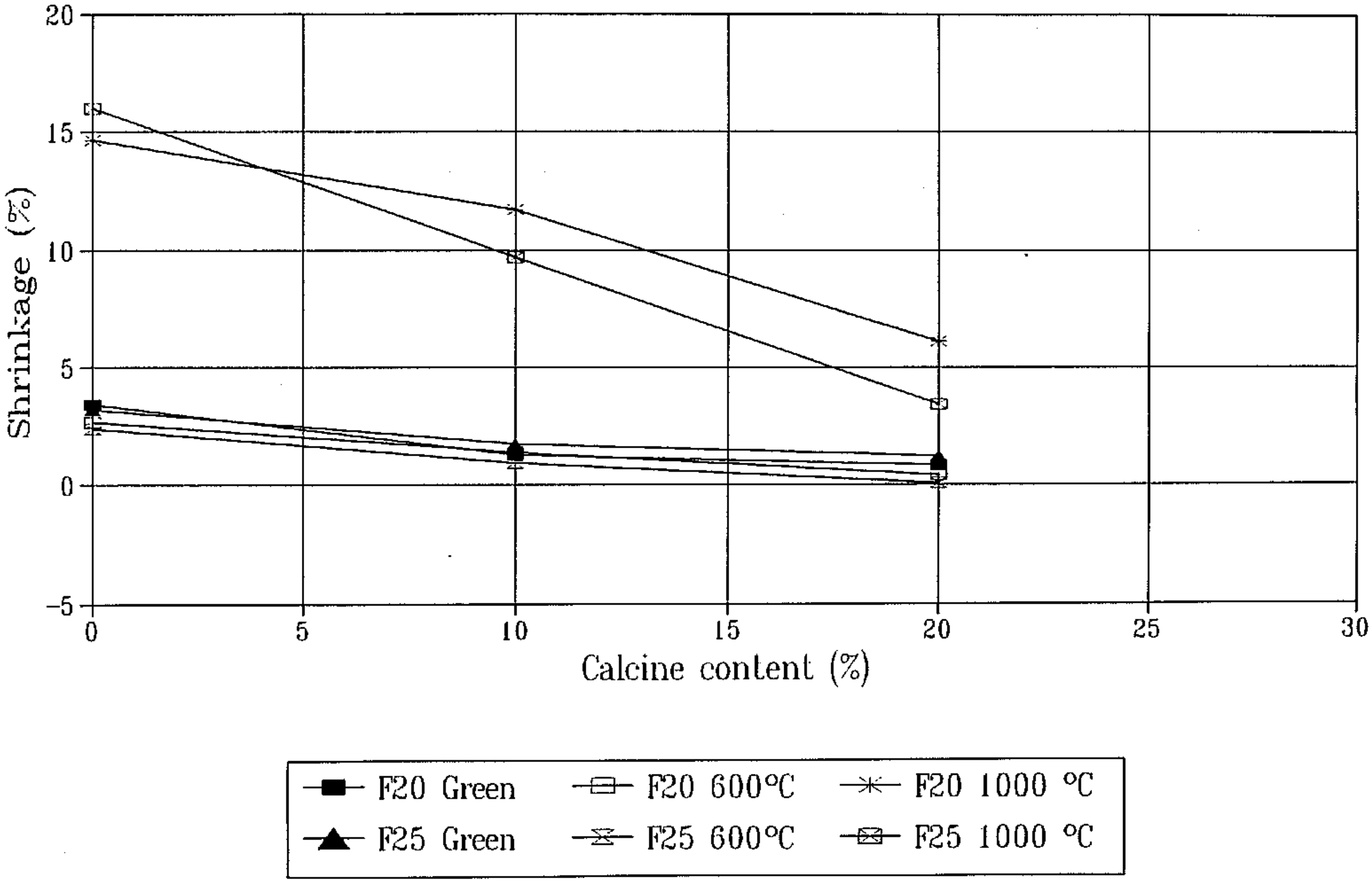


Figure 10.10 : Drying & firing shr. vs calcine content (calcine rings)



10.4 DISCUSSION OF RESULTS

10.4.1 Forming properties

Based upon Tables 10.1 and 10.2 the brickmaking forming comments on the rings with and without calcines are all good, except for the sample with 40% water. The F40 sample, because of the high water content, became sticky in the mould and could not be ejected properly. This raises the possibility of a soft mud mould filling process and it is recommended that this process be investigated.

10.4.2 Drying properties

Drying shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for face or stock bricks. Figure 10.4 shows that the dried rings drying shrinkages increase with water content to between 1 and 5% which is good. Figure 10.8 shows that the dried calcine rings drying shrinkages vary between 1 and 2%, which is excellent.

All the rings which were carefully dried at room temperature showed no signs of cracking. Two rings of Series I F20 and F25 were dried normally in an oven and they cracked slightly. All the rings which were dried carefully and slowly in an oven at 110°C did not crack. The samples are thus sensitive to temperature rise and precautions will have to be taken in practice to ensure that bricks made from these materials are not subjected to thermal shocks. It is essential that the whole drying process and rate of temperature rise be optimized for the rings.

The addition of calcines has a grogging (filler) effect on the material, reducing the drying shrinkage of the respective samples, which is good.

10.4.3 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks.

Figure 10.1 shows that rings fired at 600°C have water absorptions varying between 51 to 44% which is unacceptable and implies high porosity due to the evolution of volatile matter. Rings fired at 950°C have water absorptions decreasing from 34 to 28% which is unacceptable. Rings fired at 1000°C have water absorptions varying from 13 to 22% which is acceptable. The rings pressed with 20 to 35% water have water absorptions varying from 13 to 16% which is excellent.

Figure 10.7 shows that calcine rings fired at 600°C have water absorptions varying between 37 to 44% which is unacceptable and implies high porosity due to the evolution of volatile matter. The 20% calcine rings fired at 1000°C have water absorptions of 29% which is unacceptable. The 10% calcine rings fired at 1000°C have water absorptions varying from 21 to 23% which may be acceptable.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. Ceramic kiln firing ranges of 20, 40 and 50°C are regarded as short, medium and long, respectively.

All the samples would make poor clamp brickmaking materials from the water absorption firing range point of view. All the samples fired at 1000°C except the 20% calcine rings may make acceptable ceramic kiln brickmaking materials from the water absorption firing range point of view.

It is recommended that the low fired porosity be decreased eg by calcining or de-airing the unfired material.

10.4.4 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks.

Figure 10.4 shows that rings fired at 600°C have firing shrinkages varying between 1 to 3% which is excellent. Rings fired at 950°C have firing shrinkages decreasing from 9 to 8% which is very high. Rings fired at 1000°C have firing shrinkages varying from 15 to 16% which is unacceptable. The effect of mix water content on the fired rings is negligible.

Figure 10.8 shows that calcine rings fired at 600°C have firing shrinkages varying between 0 to 1% which is excellent. The 20% calcine rings fired at 1000°C have firing shrinkages decreasing from 6 to 4% which is good. The 10% calcine rings fired at 1000°C have firing shrinkages decreasing from 12 to 10% which is unacceptable. The grogging effect of the 20% calcine addition is marked compared to the 15 to 16% firing shrinkage when no calcines are added.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. Ceramic kiln firing ranges of 20, 40 and 50°C are regarded as short, medium and long respectively.

All the samples except the 20% calcine rings would make poor to bad clamp brickmaking materials from the firing shrinkage firing range point of view. The 20% calcine rings would make acceptable clamp and ceramic kiln brickmaking materials from the firing shrinkage firing range point of view.

10.4.5 Brickmaking

All the samples are unacceptable clamp and ceramic kiln brickmaking materials from the firing range and, the forming and firing properties points of view. In spite of this negative conclusion it is recommended that scaling up to full perforated brick size of the more successful compositions be undertaken because the presumed drying problems encountered seem to have been overcome.

The colours of the fired rings should be acceptable for bricks.

It is recommended that waterworks sludge processing be further investigated for brick production. The optimum amount of calcines which gives beneficial properties to sludge rings should be determined.

It is recommended that the patenting possibilities of sludge processing for brickmaking be investigated.

10.4.6 Tilemaking

According to the water absorption floor tile requirements given in Section 3.4.5 all the rings would be classified as BIV floor tiles and would have to be glazed.

According to the water absorption glaze wall tile requirements given in Section 3.4.5 only the rings not containing calcines fired at 1000°C except F40 are acceptable. The 10% calcine rings fired at 1020°C are also acceptable.

The dimensional tolerances given in Section 3.4.5 are quite stringent and it is believed that only the 20% calcine rings composition if fired in a ceramic kiln where the temperature can be controlled accurately could possibly meet these tile requirements.

The colours of the fired rings should be acceptable for tiles.

Considering all the above it is concluded that the rings may make fair glazed wall tile materials and it is recommended that glazed trials be undertaken.

It is recommended that pressing of tiles of successful compositions be undertaken.

10.5 CONCLUSIONS

a) All the rings which were carefully dried at room temperature showed no signs of cracking. The samples are, however, sensitive to temperature rise and precautions will have to be taken in practice to ensure that bricks made from these materials are not subjected to thermal shocks during the drying process.

b) The addition of calcines has a filler effect on the material, reducing the drying shrinkage of the respective samples, which is good.

c) All the samples are unacceptable clamp and ceramic kiln brickmaking materials from the firing range and, the forming and firing properties points of view.

d) It is concluded that the rings may make fair glazed wall tile materials.

10.6 RECOMMENDATIONS

a) It is recommended that the soft mud brick process be investigated.

b) It is recommended that the whole drying process and rate of temperature rise be optimized for the rings.

c) It is recommended that the low fired porosity of the rings be decreased eg by calcining or de-airing the unfired material.

d) It is recommended that waterworks sludge processing be further investigated for brick production. The optimum amount of calcines which gives beneficial properties to sludge rings should be determined.

e) It is recommended that scaling up to full perforated brick size of the more successful compositions be undertaken.

f) It is recommended that the patenting possibilities of sludge processing for brickmaking be investigated.

g) It is recommended that glazing trials be undertaken.

11. EXTRUSION OF BRIQUETTES

11.1 SCOPE

To determine the extrudability of the submitted sludge.

11.2 EXPERIMENTAL WORK

11.2.1 Sample preparation

A Pretoria area brickmaking clay (E) was used as an extrusion aid. For the preparation of briquettes the dried sludge and clay were first hammer milled until all the materials passed through a 1 mm sieve. They were then mixed as required in a laboratory cement mixer with the minimum quantity of water adequate for extrusion in a de-airing laboratory extruder under a vacuum of approximately 380 mm of mercury. The cross section of the extruded column was 38 mm by 25 mm. After extrusion 155 mm and 250 mm briquettes were cut from the column. The briquettes were then dried in air at room temperature for one week followed by drying overnight at 110°C. In one case a solution of 15% (m/m) liquid soap (Teepol) to 85% water was used for extrusion. In this case after mixing in the water/soap solution the mixture was sealed overnight to temper.

11.2.2 Firing

The dried samples were fired in a laboratory electric furnace. Groups of three briquettes were fired, where appropriate, at each of the following temperatures: 900, 950, 1000, 1050, 1100, 1150, 1200 and 1250°C. The heating rate was approximately 80°C per hour, and on attaining the required temperature the samples were kept at this temperature for 5 hours and then they were allowed to cool to room temperature.

11.3 RESULTS

11.3.1 Forming properties

The results obtained on the extruded briquettes are given in Table 11.1. The number preceding the sample identification letter denotes the nominal percentage by mass of material used for extruding, eg. 100F means 100% unfired sludge F was used on a dry basis.

Table 11.1 : Summary of forming properties

Forming property	100F	100FT*	50F50E	30F70E	100E
Extrusion	not possible	poor	not possible	good	good
Mixing water	-	56,5	-	45,9	29,9
Plasticity	-	poor	-	good	good
Dog earing	-	high	-	slight	slight
Green strength	-	fair	-	good	fair
Drying shrinkage	-	12,8	-	6,6	2,0
Normal drying	-	fair	-	good	good
Rapid drying	-	-	-	good	good
Colour	-	brown	-	light brown	light brown

* Teepol solution used

11.3.2 Fired properties

The results obtained on the fired briquettes are given in Tables 11.2 to 11.4, and are graphically presented in Figure 11.1.

Table 11.2 : Fired properties of the extruded briquettes for sample 100 FT

PROPERTY	TEMPERATURE (°C)					
	800	850	900	950	1000	1050
Firing shrinkage (%)			5,1		14,7	
Mass loss (%)			19,2		19,2	
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient			17,8 20,5 0,86		9,2 10,6 0,87	
Colour			brown		dark brown	
Remarks after firing			poor, crazed		fair, crazed	

Table 11.3 : Fired properties of the extruded briquettes for sample 30F70E

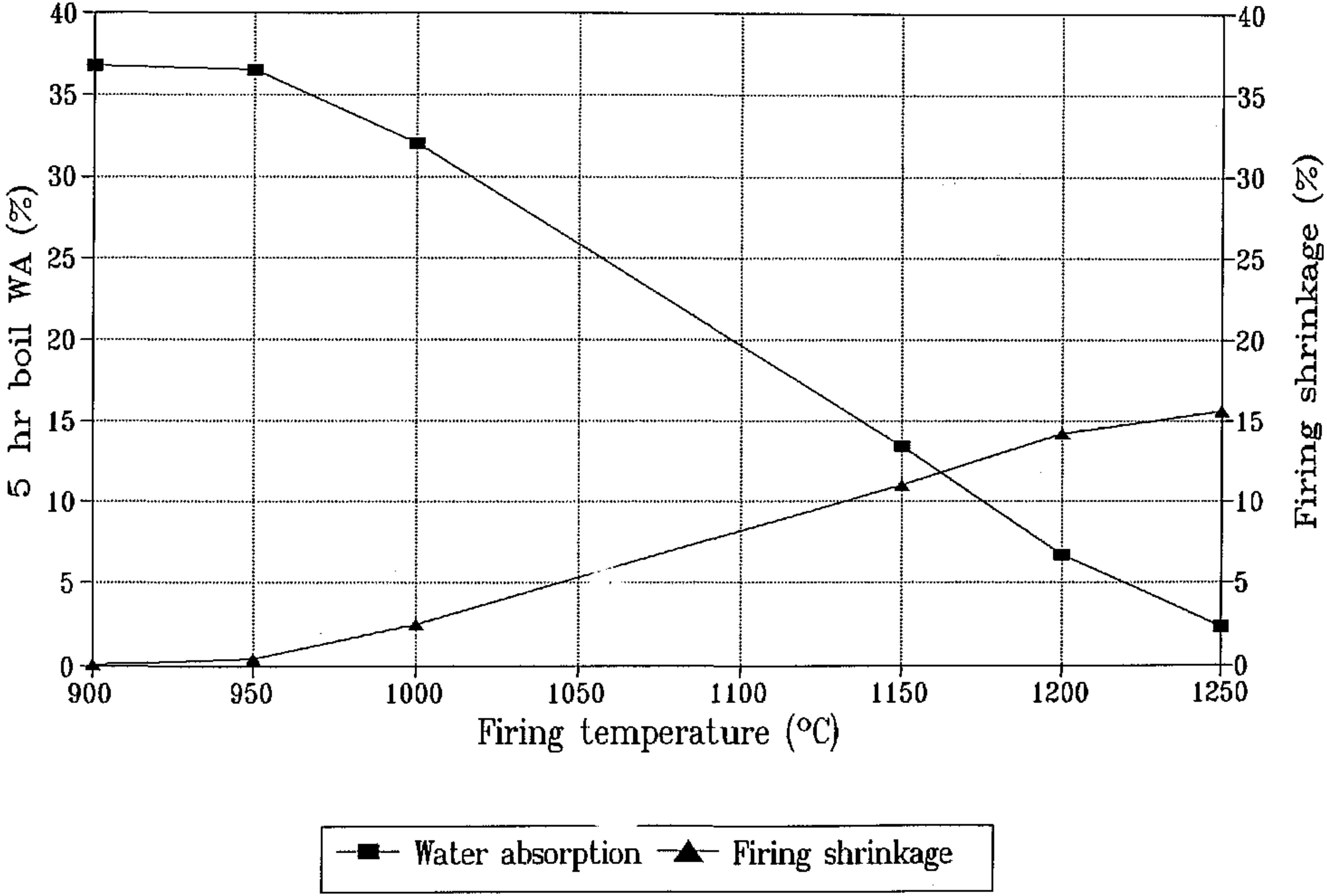
PROPERTY	TEMPERATURE (°C)						
	900	950	1000*	1100	1150	1200	1250
Firing shrinkage (%)	0,4	0,4	2,6	-	11,0	14,3	15,6
Mass loss (%)	11,9	11,7	11,6	-	12,6	10,6	13,1
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	33,4 36,7 0,91	32,2 36,5 0,88	27,0 32,0 0,84	- - -	9,6 13,4 0,72	4,0 6,7 0,59	1,2 2,3 0,52
Colour	salmon	salmon	salmon	-	dark brown	dark brown	brown black
Remarks after firing	good	good	good	-	good	good	good

* cored

Table 11.4 : Fired properties of the extruded briquettes for sample 100E

PROPERTY	TEMPERATURE (°C)							
	900	950	1000	1050	110 0	1150	1200	1250
Firing shrinkage (%)	0,1	0,1	0,3	2,4	5,8	7,2	8,6	-
Mass loss (%)	7,6	7,4	6,8	7,3	7,5	7,5	8,2	-
24hr cold absorption (%) 5hr boil absorption (%) Saturation coefficient	30,2 31,3 0,96	29,4 31,0 0,95	28,5 29,3 0,97	23,8 25,0 0,95	16,6 18,7 0,89	10,7 12,9 0,83	4,8 5,9 0,82	- - -
Colour	light salmon	light salmon	light salmon	light salmon	light red	red	dark red	-
Remarks after firing	good	good	good	good	good	good	good	-

Figure 11.1 : Water absorption and shrinkage vs firing temp. of 30F70E



11.4 DISCUSSION OF RESULTS

11.4.1 Forming properties

Table 11.5 gives the brickmaking forming comments on the briquettes. This Table is based upon the data given in Table 11.1.

Table 11.5 : Brickmaking forming comments on briquettes

Sample	Extrusion behaviour	Drying behaviour	Green strength	Brickmaking comments
100F	not possible	-	-	unacceptable
100FT	poor	good	fair	unacceptable
50F50E	not possible	-	-	unacceptable
30F70E	good	good	fair	good
100E	good	good	fair	good

The brickmaking forming comments on briquettes 100F and 50F50E are that they are unextrudable and therefore unacceptable. Briquettes 100FT extrude poorly and combined with an excessive drying shrinkage are unacceptable. The brickmaking forming comments on briquettes 30F70E and 100F are that they are good and acceptable.

It is not recommended that further attempts to extrude 100% sludge be undertaken at this stage.

Drying shrinkage or expansion values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium drying shrinkages are acceptable for tiles, face or stock bricks. The 30F70E and 100E briquettes drying shrinkages are acceptable and good, respectively.

The 30F70E and 100E briquettes which were dried at room temperature or in a oven at 110°C showed no signs of cracking. These samples are thus not sensitive to drying and precautions do not have to be taken in practice to ensure that bricks or tiles made from these materials do not dry out too quickly. The 100FT briquettes are slightly drying sensitive.

11.4.2 Water absorption

Water absorption values from 8 to 12% can be regarded as fairly low and from 12 to 16% as medium, and satisfactory for face or stock bricks. Water absorption values from 16 to 20% can be regarded as fairly high but still acceptable for face or stock bricks. From Tables 11.2 to 11.4 and Figure 11.1 the 100FT sample fired at 900°C has an acceptable water absorption and sample 30F70E fired at 1150°C has a good water absorption. Samples 100E fired at 1100 and 1150°C also have good water absorptions.

Table 11.6 gives the firing range of the briquettes based upon an acceptable water absorption range as described above. This Table is based upon the data given in Tables 11.2 to 11.4 and Figure 11.1.

Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively. In a fixed ceramic kiln where temperatures can be controlled accurately a very short clamp firing range of 50°C is acceptable.

Table 11.6 : Water absorption firing range of briquettes

Sample	5hr WA range (−20≥WA≥−8) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
100FT	20 - 11	900 - 1000	short	poor
30F70E	13 - 7	1150	too short	bad
100E	19 - 13	1100 - 1150	very short	poor

All the samples would make poor or bad clamp brickmaking materials from the water absorption firing range point of view. All the poor and bad clamp brickmaking samples would make acceptable and poor, respectively, kiln brickmaking materials from the water absorption firing range point of view.

11.4.3 Linear firing shrinkage

Firing shrinkage values below 3% are regarded as low, between 3 and 7% as medium and above 7% as high. Low and medium firing shrinkages are acceptable for face or stock bricks. Clamp firing ranges of 50, 100, 150, 200 and ≥300°C are regarded as very short, short, medium, long and very long, respectively.

Table 11.7 gives the firing range of the different series of briquettes based upon an acceptable firing shrinkage range as described above. This Table is derived from the data given in Tables 11.2 to 11.4 and Figure 11.1.

Table 11.7 : Firing shrinkage firing range of briquettes

Sample	Firing shrinkage (0≤FS≤7) (%)	Firing range (°C)	Firing range comments	Clamp brickmaking comments
100FT	5	900	short	poor
30F70E	0 - 3	900 - 1000	short	poor
100E	0 - 7	900 - 1150	long	good

The briquettes 100FT and 30F70E would make poor and acceptable clamp and kiln brickmaking materials, respectively, from the firing shrinkage point of view. The 100E briquettes would make as expected good brickmaking materials from the firing shrinkage point of view.

11.4.4 Brickmaking

Based upon the combination of the water absorption and firing shrinkage firing ranges given in Tables 11.6 and 11.7 there is no common brickmaking firing range for the different series of briquettes containing sludge. All the briquettes containing sludge are unacceptable clamp and kiln brickmaking materials from the common firing range point of view. As would be expected the 100% clay sample E is acceptable.

The colours of the fired briquettes should be acceptable for bricks.

It is recommended that attempts be made to obtain a common water absorption and firing range for the briquettes, if the extrusion problems are overcome.

11.5 CONCLUSIONS

a) The brickmaking forming comments on briquettes 100F and 50F50E are that they are unextrudable and unacceptable. Briquettes 100FT extrude poorly and are unacceptable. The brickmaking forming comments on briquettes 30F70E and 100F are that they are good and acceptable.

b) None of the 30F70E and 100E briquettes are sensitive to drying and no precautions have to be taken in practice to ensure that briquettes made from these materials do not dry out too quickly. The 100FT briquettes are slightly drying sensitive.

c) All the samples containing sludge are unacceptable clamp and kiln brickmaking materials from the common firing range point of view.

d) The colours of the fired briquettes should be acceptable for bricks or tiles.

11.6 RECOMMENDATIONS

a) It is not recommended that further attempts to extrude 100% sludge be undertaken at this stage.

b) It is recommended that pressing trials be undertaken.

12. GENERAL CONCLUSIONS

a) The Wiggins waterworks sludge may be suitable for producing rustic tiles and for manufacturing stock or face bricks.

b) Severe difficulties were experienced in all critical areas of ceramic processing, i.e. forming, drying and firing. Fortunately most of these difficulties have been overcome, to a greater or lesser extent.

c) In the area of forming it was found that extrusion is not possible at this stage but that pressing of tiles worked well. The pressing of bricks proved to be the greatest area of difficulty of the whole investigation. The bricks pressed well in the die but after being removed from the die for several hours they might crack badly. It is believed that this difficulty has been overcome but it has not been possible at this stage to produce full-size bricks to prove conclusive success due to time and financial constraints of the investigation.

d) In the area of drying of the pressed tiles and bricks it was originally thought that no difficulties were present but it was eventually found that this was the cause of the cracking of the unfired bricks. This was unexpected and very unusual because the drying shrinkage of the bricks is very low and would normally in the case of extruded bricks never cause such a problem. The method to overcome the difficulty is to perforate the brick and we have been successful in producing rings of approximately 50 mm in height, a diameter of 100 mm and a thickness of 20 mm.

e) In the area of firing it was found that if the brick or tiles were fired in a normal manner up to top temperature, usually in the range of 900 to 1000°C, they would crack or warp badly. This difficulty was overcome by introducing a calcination step at 600°C to burn off deflocculants and organic matter. The firing cycle adopted was one day to reach 600°C from room temperature, then left for one day at 600°C followed by an 80°C rise per hour to reach the required top temperature. This solved the cracking and warping, of the tiles and the rings but not for the solid bricks. Another area of concern is the firing range of the material. It is at this stage too short to clamp fire but could be adequate for a well controlled ceramic kiln if the porosity and firing shrinkage properties would be optimized. It was also found that noxious gases were evolved in the temperature range of 250 to 550°C. This can be overcome by venting or scrubbing the gases.

13. GENERAL RECOMMENDATIONS

a) We recommend that a techno-economic feasibility study of glazed and unglazed tiles including pilot plant trials be undertaken.

b) We recommend that full-size perforated bricks be investigated before pilot plant trials are undertaken.

14. REFERENCES

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