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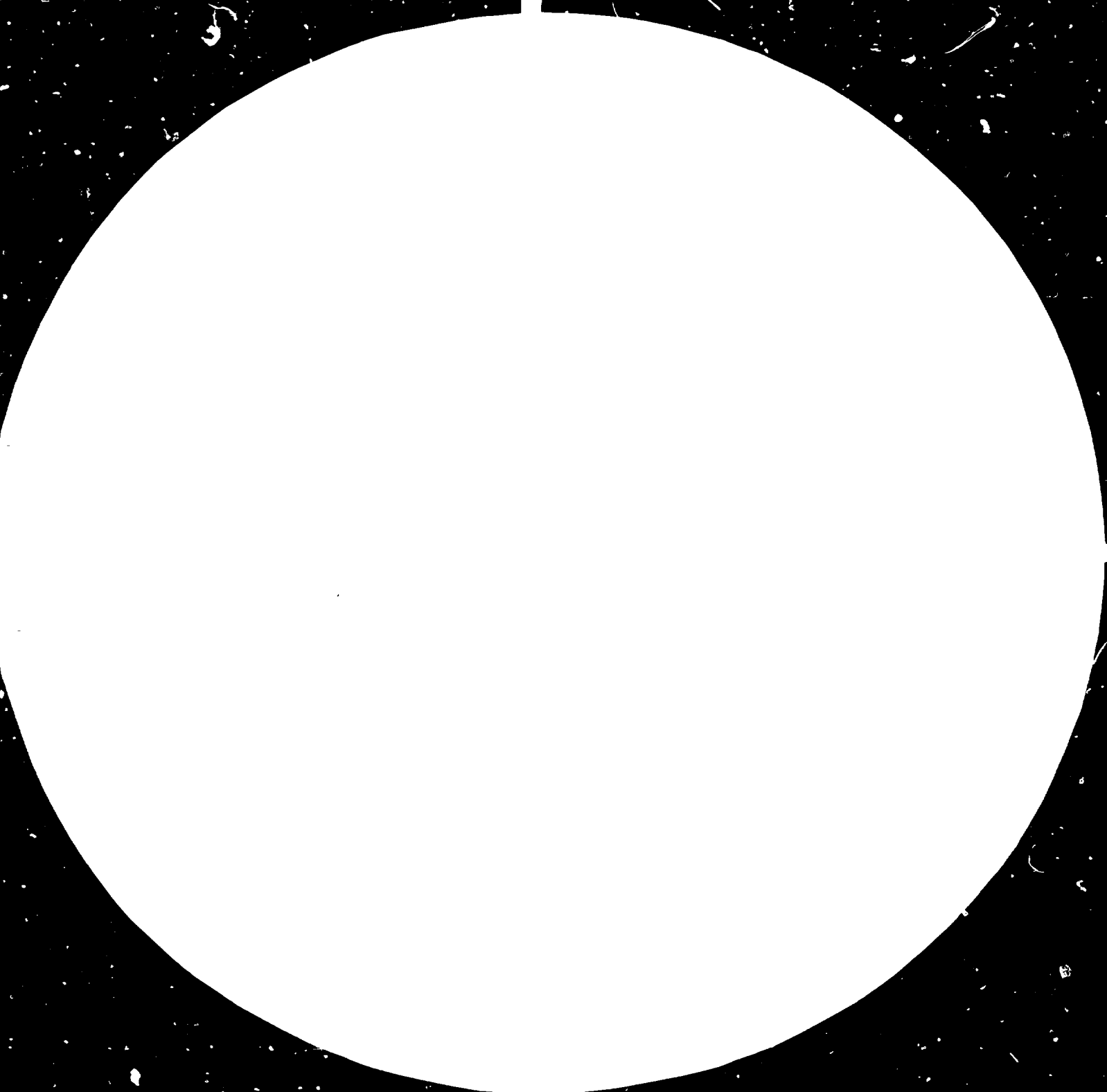
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Microcopy Resolution Test Chart

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ART OF COMPOSING WALL TILE BODIES

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## I. INTRODUCTION

The Third General Conference of UNIDO outlined the requirement for a more intensive training of technicians from developing and the least developed countries. This conclusion proved true already in the In-plant Training Workshop on the Exploitation and Beneficiation of Non-metallic Minerals organized by the UNIDO/Czechoslovakia Joint Programme for International Co-operation in the Field of Ceramics, Building Materials and Non-metallic Minerals Based Industries at Pilsen in April 1980.

The paper being presented defines the chief practical principles to be observed by a technician of any developing country wanting to assess the suitability of local non-metallic raw materials for the manufacture of double-fire porous wall tiles based upon the conducted geological exploration.

This publication respects the variability of local raw materials and, hence, it is focussed to a series of technological processes resulting in a product of equal utility values though the possibilities may often even strikingly change from the point of view of raw materials used.

The authors of this paper are convinced that this practical instruction, respecting fully the the essential theoretical knowledge, will contribute to the promotion of know-how of the technicians

from the developing and least developed countries, to the commercial utilization of local non-metallic raw materials and thereby to the industrialization of these countries as well as to the improvement of housing and living standards.



## II. TESTING EQUIPMENT

This paper covers only the equipment necessary for technological testing and verification of body compositions for porous wall tiles. Analytical silicate laboratory equipment is not described though the chemical analyses of raw materials having not yet been used are of high importance for a quick composition of bodies, particularly of the lime-siliceous type.

The equipment for the technological testing depends on the extent of tests to be made, whether only preliminary tests are to be performed on body composition from unknown raw materials or laboratory tests or a detail technological process should be elaborated and properties of the manufactured wall tiles verified, i. e. if semi-industrial test is involved.

The following equipment is necessary for the preliminary tests:

- a set of sieves, at least those ones of 0.063, 0.2 and 1.5 mm mesh sizes
- technical balance of 10 kg max. weighing capacity
- technical balance of 1 kg max. weighing capacity of 0.01 gr accuracy
- planetary or porcelain drum mill up to 1 kg volume
- porcelain grinding mortar with a pestle
- filter cloth or plaster dishes

- laboratory hydraulic or screw press
- laboratory kiln up to 1280°C temperature
- hand-glazing tank or a spray gun
- laboratory drier

In addition to the above mentioned instruments the following pieces of equipment are necessary for the verification testing, elaboration of a technological process and verification of properties of the manufactured wall tiles:

- drum mill of 100 to 400 litre capacity
- laboratory filter press
- laboratory pan mill with a screening path
- wall or floor tile press
- drier with temperature control and forced circulation of air
- laboratory kiln of 0.5 to 1 cu.m capacity up to 1250°C temperature

### III. COMPOSING OF BLENDS FOR WALL TILE BODIES

#### 1. Different Technological Types of Wall Tile Bodies

Basically, every wall tile body for porous wall tiles consists of two main groups of raw material, i. e. of two components. They are the plastic component, which enables the shaping of pressings, and the non-plastic component, which controls shrinkage and decreases the possibility of cracking of the pressings due to an excessive plasticity of the former one on one hand and renders prerequisites for the formation of phases during the firing process on the other hand being characteristic for the individual types of wall tile body.

Ball clays resulting in a porous body when fired at 1200 - 1250°C temperature as well as semivitrifying clays or even pronounced stoneware clays may be used as a plastic component because the properties of the body can be adapted by adding suitable non-plastic raw materials, i. e. grog or fluxes. Kaolinitic clays are suitable whereas illitic or montmorillonitic ones not due to their tendency to cracking during the drying and firing processes.

The different types of wall tile bodies can be classified according to the type and amount of non-plastic admixtures as shown in Table 1.

Table No. 1

Basic Types of Porous Wall Tile Bodies According to the Mineralogical Composition

		kaolinitic	semisili- ceous	siliceous			Talcum		Wollasto- nitic
				feldspatic	calcare- ous	combined	I.	II.	
Cly Substance /Kaolinite/	%	80	60-65	40-55	35-37	50	40	20-25	25-35
Silica	%	15	25-30	35-50	36-41	40	15	-	0-15
Feldspar	%	5	5-10	5-15	-	5	3	-	-
Limestone /chalk.shells/	%	-	-	-	13-15	5-10	2	-	-
Talc	%	-	-	-	-	-	40	75-80	-
Wollastonite	%	-	-	-	-	-	-	-	50-75
Non-plastic components total	%	55-65	55-65	55-60	60-65	50-60	55-60	75-80	50-75
out of that fired grog	%	40-45	20-30	0-10	0-10	5-10	0-5	0-5	0-5
Firing temperature of bisque	°C	1280	1250	1230	1050	1150	1200	1180	1060-1120
of glaze	°C	1120	1100	1080	960	1040	1040	1040	1020

The types of bodies are shown in terms of the mineralogical composition. The conversion from a chemical composition into a mineralogical one is shown in Table 2.

Table No. 2      Conversion of the Chemical Composition of  
Unfired Body into the Mineralogical Composition

A. Principle of the calculation:

1. Content of  $\text{Na}_2\text{O}$  is to be converted to sodium feldspar (albite)  $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ; the content of  $\text{K}_2\text{O}$  is to be converted to potassium feldspar (orthoclase)  $\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ .
2. The content of  $\text{Al}_2\text{O}_3$  in the both feldspars is to be deducted from the total content of  $\text{Al}_2\text{O}_3$  and the clay substance (kaolinite) is to be calculated from the remaining  $\text{Al}_2\text{O}_3$ .
3. The content of  $\text{SiO}_2$  in the feldspars and in the clay substance is to be deducted from the total content of  $\text{SiO}_2$  the rest being free silica.
4.  $\text{CaO}$  and  $\text{MgO}$  should be converted to carbonates.

B. Calculation

1. Content of  $\text{Na}_2\text{O}$  in terms of % multiplied by 8.450  
= content of sodium feldspar in terms of %

Content of sodium feldspar in terms of % multiplied  
by 0.1944 = content of  $\text{Al}_2\text{O}_3$  in sodium feldspar

Content of potassium feldspar in terms of %  
multiplied by 0.6873 = content of  $\text{SiO}_2$  in  
sodium feldspar

Content of  $K_2O$  in terms of % multiplied by 5.907  
= content of potassium feldspar in terms of %

Content of potassium feldspar in terms of %  
multiplied by 0.1332 = content of  $Al_2O_3$  in  
potassium feldspar

Content of potassium feldspar in terms of %  
multiplied by 0.6475 = content of  $SiO_2$  in  
potassium feldspar

2. Total content of  $Al_2O_3$   
- total of  $Al_2O_3$  from the feldspars  
   $Al_2O_3$  from kaolinite multiplied by 2.531  
= content of clay substance in terms of %

Clay substance multiplied by 0.4651 = content of  
 $SiO_2$  in kaolinite

3. Total content of  $SiO_2$   
-  $SiO_2$  from the feldspars  
-  $SiO_2$  from the clay substance  
 $SiO_2$  - free silica

4. Content of  $CaO$  in terms of % multiplied by 1.765  
= content of  $CaCO_3$  in terms of %  
+ Content of  $MgO$  in terms of % multiplied by 2.091  
= content of  $MgCO_3$  in terms of % = total amount of  
carbonates

C. Checking

Content of sodium feldspar	%
+ Content of potassium feldspar	%
+ Content of clay substance (kaolinite)	%
+ Content of silica	%
+ <u>Content of carbonates</u>	%
	-----
	100 %

The two below mentioned basic criteria are decisive for the choice of any of the types of the wall tile body:

- a) Raw material available
- b) Energy requirements being of ever increasing importance nowadays due to the world energy situation

No problem should occur in case of the first three types of bodies in view of the raw material availability because a clay raw material and a silica component are generally the prerequisites for starting a ceramic production and these materials in some form can be found everywhere in the world. Calcareous component (limestone, chalk, shells) need not be always available for lime-siliceous and combined siliceous types of body but it is added in small amount only. Hence, limestone or chalk may be worth importing in view of the energy savings in the firing process. The main raw materials required for the latter three types of the body, i. e. talc and wollastonite are comparatively rare and occur in some countries only but, on the other hand, they represent very valuable raw materials for any ceramic manufacture.

In view of energy requirements the first two types are the least suitable ones due to the high firing temperatures of both body and glaze on one

hand and that fired grog (fired kaolin or clay) is to be added into the body on the other hand that results in an increased consumption of energy.

Fired grog added to the other bodies is the exploitable breakage from the manufacturing process that would otherwise constitute an inconvenient waste.

As it ensues from the above mentioned facts the lime-siliceous type of a body is the cheapest and the most suitable one for the manufacture of porous wall tiles; it also has a higher coefficient of thermal expansion enabling thus the use of low-temperature glazes the coefficient of thermal expansion of which is always high, too.



## 2. Lime-siliceous Wall Tile Bodies Composition

The main components of these bodies are:

- plastic raw materials - clays or kaolin
- siliceous raw materials - silica sand, lumps of quartz or flint, raw kaolin or very sandy or little plastic clays
- calcareous or even calcareous-magnesium raw materials - limestone, chalk, shells, spongillite (along with its siliceous component), marls (along with the plastic component), marl slate or dolomite

### a) Choice of suitable plastic raw materials

The plastic component of a body gives the mechanical strength to the semiproducts to retain their shape after pressing, drying and partly also after firing.

Both ball and stoneware clays are suitable for the manufacture of porous wall tiles made of lime-siliceous body since the stoneware clays do not get vitrified yet at the 1050 - 1060°C temperature under which this type of body is fired. Hence, the clays are required to yield a porous or slightly densified body after firing at the above mentioned temperature. The body should be of a light colour as far as

possible. In case the products may not require a light-coloured body even darker-firing clays may be used, as many producers in Italy and Spain do so.

In general, we can say that wall tile body can be composed of any clay which shows

- a) requested colour after firing,
- b) continuous curve of thermal expansion,
- c) low moisture expansion after firing,
- d) pressability.

Evaluation of plastic raw materials according to the chemical composition

Chemical composition of the plastic raw materials proper is of little importance for the manufacture of wall tiles because the chemical composition of the entire wall tile body can be adapted by the amount of addition of the other components being the carriers of silica and calcium oxide.

Loss on ignition may range from 5 to 15%. Light-coloured clays without organic matter with higher content of silica or feldspars show lower loss on ignition. Dark-coloured plastic clays with higher content of organic matter show generally higher loss on ignition. These clays may cause so called "scalding" while firing the wall tile bodies stacked in columns. Calcium or magnesium

carbonates contained in clays may also increase the loss on ignition. Loess-clays and marls have a higher content of calcium carbonate. The content of carbonates is harmless in the wall tile body composition; these carbonates can partly replace the calcareous component in the wall tile body.

The content of silica in plastic raw materials usable for the manufacture of wall tiles ranges from 50% up to almost 75%. Washed kaolins or pure kaolinitic clays have a lower content of  $\text{SiO}_2$ . Unless it is indispensable to use clays with a low content of  $\text{SiO}_2$  (e.g. when other clays are not available) it is useful to apply such clays in the manufacture of fire-clays and to use clays with a higher content of  $\text{SiO}_2$  for the manufacture of wall tiles. When clays with a low content of  $\text{SiO}_2$  are used difficulties may also be encountered while composing the wall tile body with respect to achieve high enough thermal expansion in the wall tile body as it will be dealt with in the respective chapter.

Content of alumina in plastic raw materials for wall tile body may range from 17 to 35%. Washed kaolins and purely kaolinitic clays without silica or feldspatic minerals admixtures have high content of  $\text{Al}_2\text{O}_3$ . A small amount of washed kaolins only is to be added into the wall tile bodies because washed kaolins usually

do not give enough strength to the pressed tiles. The opposite principle is valid here against that one said of the  $\text{SiO}_2$  content namely, that clays with high  $\text{Al}_2\text{O}_3$  content are suitable for the manufacture of refractory products whereas clays with a lower content of  $\text{Al}_2\text{O}_3$ , if available, should be used for the manufacture of wall tiles.

Contents of calcium oxide and magnesium oxide are harmless in the wall tile bodies. To the contrary, clays of loessal or, mainly, marlous types are suitable because they supply the body with a part or even with all the calcium or magnesium-calcium components. The total of the two oxides may amount to as much as 25%.

Content of ferric oxide is of significance in such cases when the wall tile body colour is required to be light. The content of  $\text{Fe}_2\text{O}_3$  in such a case should not exceed 2 - 2.5%. As far as wall tiles of coloured bodies are envisaged to be manufactured the clays may contain a higher amount of  $\text{Fe}_2\text{O}_3$  - even 5 to 7%.

Content of titanium dioxide amounts about 1% in the majority of clays. Its higher content along with a higher content of  $\text{Fe}_2\text{O}_3$ , results in a dark colour of a body after firing; therefore, clays with the content of  $\text{TiO}_2$  higher than 1% cannot be used for the manufacture of

wall tiles with a light-coloured body.

Content of alkaline oxides  $K_2O$  and  $Na_2O$  results in a partial densification of a body though the alkalis effect is rather indistinct at  $1050 - 1060^{\circ}C$  bisque firing temperature. The total amount of alkaline oxides in most clays ranges between 1 and 2%. When the content of the alkaline oxides is higher such a clay may be suitable for the manufacture of floor tiles or stoneware products.

It is obvious from the aforesaid that the chemical composition of plastic raw materials suitable for the manufacture of porous wall tiles lies usually within the following limits:

Loss on ignition	5 to 15%
Silicon dioxide $SiO_2$	50 to 75%
Aluminum oxide $Al_2O_3$	17 to 35%
Ferric oxide $Fe_2O_3$	up to 2% for a light-coloured body up to 6% for a coloured body
Titanium dioxide $TiO_2$	up to 1% for a light-coloured body
Calcium oxide $CaO$	up to 25%
Magnesium oxide $MgO$	
Potassium oxide	up to 2%
Sodium oxide	

Evaluation of plastic raw materials  
suitability according to the physical  
properties

As aforesaid the plastic components impart coherence, shaping ability (in case of wall tiles manufacture by pressing), strength after pressing and drying, and to some extent, after firing, to the body, and plastic component of the body influences decisively the resistance of semiproducts and products to cracking. These are the aspects of evaluating the suitability of plastic raw materials for the manufacture of wall tiles.

The wall tile bending strength is to be at least 0.5 MPa after pressing and 1.0 MPa after drying. Hence, it is a prerequisite that the clays to be used should have at least 2.0 MPa bending strength after drying.

As far as washed kaolin is to be used in the blend of the wall tile body the washed kaolin can be dressed by activation and delamination whereby both its plasticity and mechanical strength are increased.

In case several types of clays are available, all of them having a suitable chemical composition and sufficient strength, such a clay or clays should be preferred which have the least overall shrinkage when fired at 1050°C.

Clays showing the least shrinkage render higher degree of surety that the products size tolerances will be adhered to and they show generally lower sensitivity to cracking during the firing process.

The tendency to cracking during the drying and firing processes is also an important physical-technological feature of plastic raw materials.

The sensitivity to drying may be determined by plotting the Bigot's curve using the values of moisture reduction and shrinkage of the respective raw material. Moisture content percentage of the test brick is to be plotted on the vertical axis while the test brick shrinkage percentage on the horizontal one. Critical point can be found by extending that part of the curve for water of shrinkage with the final shrinkage according to the Picture 1.

Clays of more than 7% critical moisture content are suitable for the manufacture of wall tiles the bodies of which are to be composed of plastic raw materials (clays) and pressed at 6 to 7% moisture content or even less in case of bodies prepared by dust-spray driers.

The sensitivity or tendency to cracking during drying can also be judged by a practical test of pressings made of the clay alone. The

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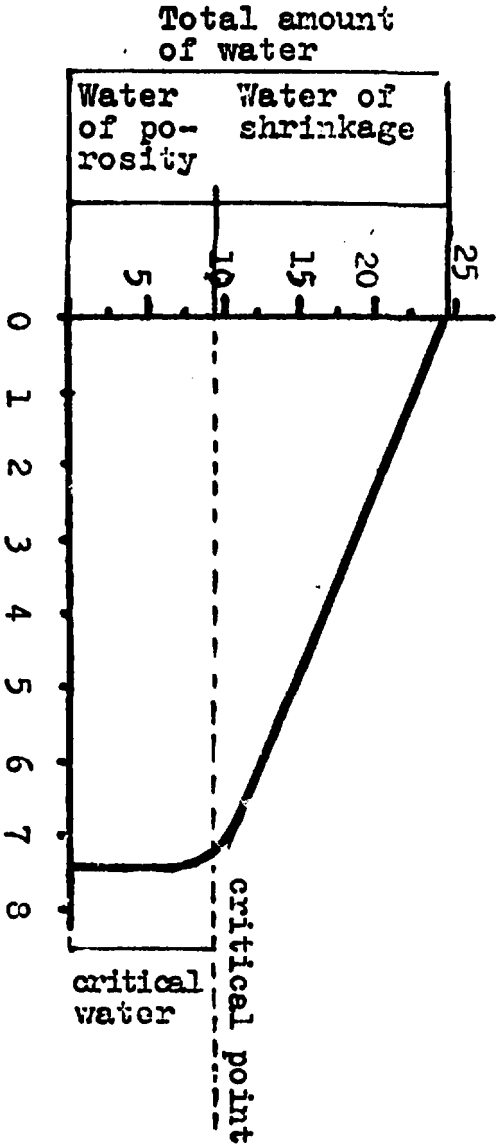
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Picture No. 1

Bigot's Curve



clay being tested is to be wet ground to about 5% rest on 10,000 mesh/sq.cm sieve (0.063 mm mesh), the slurry should be dewatered and dried subsequently to 6 - 8% moisture content. The body for pressing should then be screened by 1.5 to 2 mm mesh sieve. The wall tiles having been pressed are to be dried and checked for cracks occurrence. The wall tile edges should be painted with kerosene to make any potential cracks more distinct. The wall tiles made of the clays are then to be fired at 1060°C temperature and checked for cracks occurrence again. Such a clay or several clays showing the least tendency to cracking during drying and firing are to be selected for the wall tile body composition.

As regards the judgement of suitability of plastic raw materials for the manufacture of wall tiles for interior tiling it may be stated that the most suitable clays for this purpose are those of sufficient strength after pressing and drying, low shrinkage by drying and firing, if possible, and good resistance against cracking during drying and firing.

b) Choice of suitable non-plastic raw materials

Non-plastic raw materials for the manufacture of wall tiles are those supplying the silica component, calcareous or calcareous-magnesium component to the body and breakage of wall tiles from bisque and glost firing to utilize this breakage.

Mainly raw kaolins, pure silica sands (e.g. glass sands, dune silica sands or sands from kaolin washing plants) and even vein quartz or flint, if necessary, are used as a silica component for the manufacture of wall tiles. The latter two raw materials should be calcined at about 900 - 1000°C temperature and quickly cooled before processing so as they could be easily crushed and ground.

As far as chemical composition of the non-plastic components is concerned chiefly the content of colouring oxides, i.e. ferric oxide and titanium dioxide should not exceed 1% if wall tiles of light coloured body are required. In case of raw kaolin it should contain at least 80% of silica so that the required content of  $\text{SiO}_2$  in the blend with the plastic component could be achieved to provide for a sufficient dilatibility as it will be described in the next chapter.

The content of the colouring oxides ( $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$ ) is also important in the calcareous or calcareous-magnesium component in which these oxides,

too, should not exceed 1% for wall tiles with light-coloured body. The contents of silicon dioxide and aluminium oxide is not decisive as long as their content is constant because the required addition of lime or calcareous-magnesium component can be calculated according to the contents of these oxides.

Mostly limestone or chalk and, in maritime areas, even shells may be used as calcareous components. Since the reaction of the calcareous or calcareous-magnesium component with the clay substance is decisive for the formation of minerals which lend the required properties to the wall tile body of lime-siliceous type the calcareous or calcareous-magnesium component should be dispersed in the body as finely as possible. Therefore, it is desirable to use micro-ground limestone and, in case of the calcareous-magnesium component, ground dolomite should be added.

If clays of loessal character or marls containing enough CaO are available to reach the required content of CaO in the body it is advantageous to use these clays or marls because CaO is usually finely dispersed in them.

Other calcareous minerals may also be used e.g. spongillite or wollastonite the latter being the most suitable raw material for this type of a wall tile body.

c) Calculation of lime-siliceous body composition for porous wall tiles

The composition of a suitable body for the manufacture of wall tiles must correspond with both physical-technological requirements and chemical composition the latter being a prerequisite for the formation of minerals which are decisive for the physical-technological properties of wall tile bodies - mainly strength, shrinkage and thermal expansion.

The physical-technological properties, particularly drying and firing strength, drying and firing shrinkage and resistance against cracking are chiefly resulting from the ratio of the plastic component to the non-plastic one; it ranges usually between 40 : 60 and 50 : 50 and is mainly determined by the quality of the plastic body component. Higher proportion of the plastic component results in an increased bending strength after pressing and drying but causes usually shrinkage of a body and an increased tendency to cracking. The correct ratio of the plastic and non-plastic components cannot be theoretically specified in advance but it can only be found out by several experiments as it will be described below.

The chemical composition of the wall tile body is to be chosen in such a way so that it should enable the formation of an important

component of the lime-siliceous body, i. e. anortite (calcium feldspar) -  $\text{CaO}, \text{Al}_2\text{O}_3, 2\text{SiO}_2$  and the body should still contain enough free silica which increases the coefficient of thermal expansion whereby the resistance against the crazing of a glaze is guaranteed. When silica content is too high it makes the body sensitive to cracking both during bisque firing and glaze firing, particularly in the cooling zone due to the modification transformation of  $\alpha \rightleftharpoons \beta$  silica.

The content of the main oxides of the lime-siliceous wall tile body should range within the following limits (after firing):

66 to 70% of silica  
17 to 20% of alumina  
9 to 11% of calcium oxide

The content of these oxides can also be specified by the following modulus:

Modulus A for the judgement of the ratio of silicon dioxide and aluminum oxide:

$$\frac{0.25 \text{ SiO}_2}{\text{Al}_2\text{O}_3} \text{ should be } 0.98 \text{ to } 1.02$$

Modulus C for the judgement of the ratio of aluminum and calcium oxides to form anortite:

$$\frac{1.823 \text{ CaO}}{\text{Al}_2\text{O}_3} \text{ should be } 0.95 \text{ to } 0.98$$

Modulus K for the judgement of the content of silica uncombined in anortite, i. e. "free silica":

$$\text{SiO}_2 - 2.142 \text{ CaO} \text{ should be } 48.0 \text{ to } 50.0$$

Modulus S for the judgement of excess  $\text{SiO}_2$  against the calcareous phase:

$$\frac{\text{SiO}_2}{(0.591\text{Al}_2\text{O}_3) + (1.07\text{CaO})} \text{ should be } 3.30 \text{ to } 3.40$$

The both values of the content of the main oxides are in conformity.

According to the rational composition the wall tile body should contain:

- 35.0 to 37.0% of clay (kaolinite)
- 36.0 to 41.0% of silica
- 13.0 to 15.0% of limestone
- up to 2% of  $\text{Fe}_2\text{O}_3 + \text{TiO}_2$  (for a light-coloured body, if otherwise, even more)
- 6.0 to 8.0% of alkaline feldspars
- 3.0 to 5.0% of breakage - reject (after the deduction of silica)

It means practically what the general composition of the wall tile body while using raw kaolin as a carrier of the silica component in the body, is as follows:

plastic clay (or a mixture of clays)	35 to 45%
raw kaolin with high content of quartz	50 to 40%
limestone (chalk, shells)	10 to 16%
breakage (reject)	to 10%

While using silica sand or silica as a silica component the general composition of a body for porous wall tiles with a lime-siliceous body is as follows:

plastic clay (or a mixture of clays)	40 to 50%
silica or silica sand	10 to 35%
limestone (chalk, shells)	3 to 16%
breakage (reject)	to 10%

When clay containing CaO is used the addition of limestone is to be reduced in due proportion or when loess or marl containing appropriate amount of CaO are used in such a case limestone may be substituted by them completely.



The body so prepared or left for ageing till the next day is to be used for the pressing of the test wall tile under the pressure of 20 to 25 MPa, its size being 5 mm ± 0.5 mm thickness, 100 to 150 mm length and 35 to 50 mm width. In case a laboratory press with a suitable die is not available the test wall tile may be moulded in a plaster mould. The wall tile should be measured and dried after having been pressed. It should be measured after drying again and fired in the laboratory kiln at 1060°C temperature, the maturing time on this temperature being 30 minutes.

The wall tile is to be measured again and its water absorption determined after firing. Its changes in length by drying and firing as well as the total change in length are to be calculated. As far as the total change in length of the pressed wall tiles is less than 0.5% against the dimension of the mould or has a positive value against the dimension of the mould such a body can be regarded as being dimensionally stable. When a test wall tile is moulded from a plastic blend this figure is to be related to changes in length by firing only because the drying shrinkage in such a case is substantially higher.

In case the negative change in length (shrinkage) exceeds 0.5% the following change in the body composition is to be made:

- 1) The proportion of the plastic clay is to be reduced or a part of the plastic clay is to be substituted by a less plastic one, containing silica.

- 2) The addition of the silica component is to be increased slightly.
- 3) As far as the admixture of limestone is at the upper limit its addition is to be reduced by 1 to 2%.

In case the water absorption of the fired wall tile is found higher than 22% and the change in length by firing is positive opposite changes in the body composition are to be made, i. e.:

- 1) The addition of plastic clay is to be increased.
- 2) The addition of the silica component is to be reduced.
- 3) Or, the addition of limestone should be increased slightly, as the case may be.

The blend having been so adapted is to be prepared in the same way as stated above. The process should be repeated, if necessary, until the test wall tile bisque with absorption capacity up to 22% and change in length within  $\pm 0.5\%$  range are obtained.

If dilatometer is available a test piece is to be cut out of the test wall tile and its thermal expansion is to be determined within the temperature range from 20 up to 1000°C. A mean coefficient of thermal expansion -  $\alpha$  is to be calculated from the results and it should be within the following range:

$\alpha$ 20 - 500°C	75 to 83 $\cdot 10^{-7}$
$\alpha$ 20 - 600°C	80 to 85 $\cdot 10^{-7}$

If no dilatometer is available the test wall tile bisque is to be glazed with that glaze which is envisaged for the use in the expected production or, if such a glaze is not available, any other glaze for wall tiles for the firing temperature of 960 to 1020°C may be applied. The glazed wall tile is to be fired in the laboratory kiln at the respective temperature the maturing time at this temperature being 15 minutes.

The surface of the glaze and arching of the wall tile are to be examined after firing. If the arching is concave or even the glaze shows crazing it indicates that the body coefficient of thermal expansion is lower than that of the glaze.

When glaze with a lower coefficient of thermal expansion is not available the body thermal expansion should be increased by higher content of free silica by adding a silica component on the account of the breakage (reject) or clay.

As far as the wall tile after firing is flat or slightly convex only, it is to be subjected to Harkort's test of resistance against crazing of the glaze. The result, however, is for orientation only because the Harkort's test of the test wall tile

being of smaller size shows better results than on full size wall tiles, as far as the glaze withstands the cooling from the temperature at least 175°C without crazing this body may be subjected to a verification test. When the Markort's test value is lower the above mentioned steps towards increasing the thermal expansion are to be made but to smaller extent only than in the case of a wall tile becoming concave or crazing immediately after firing.

Pictures No. 2 through 5 show the practical evaluation of the relationship between coefficient of thermal expansion of the body and glaze.

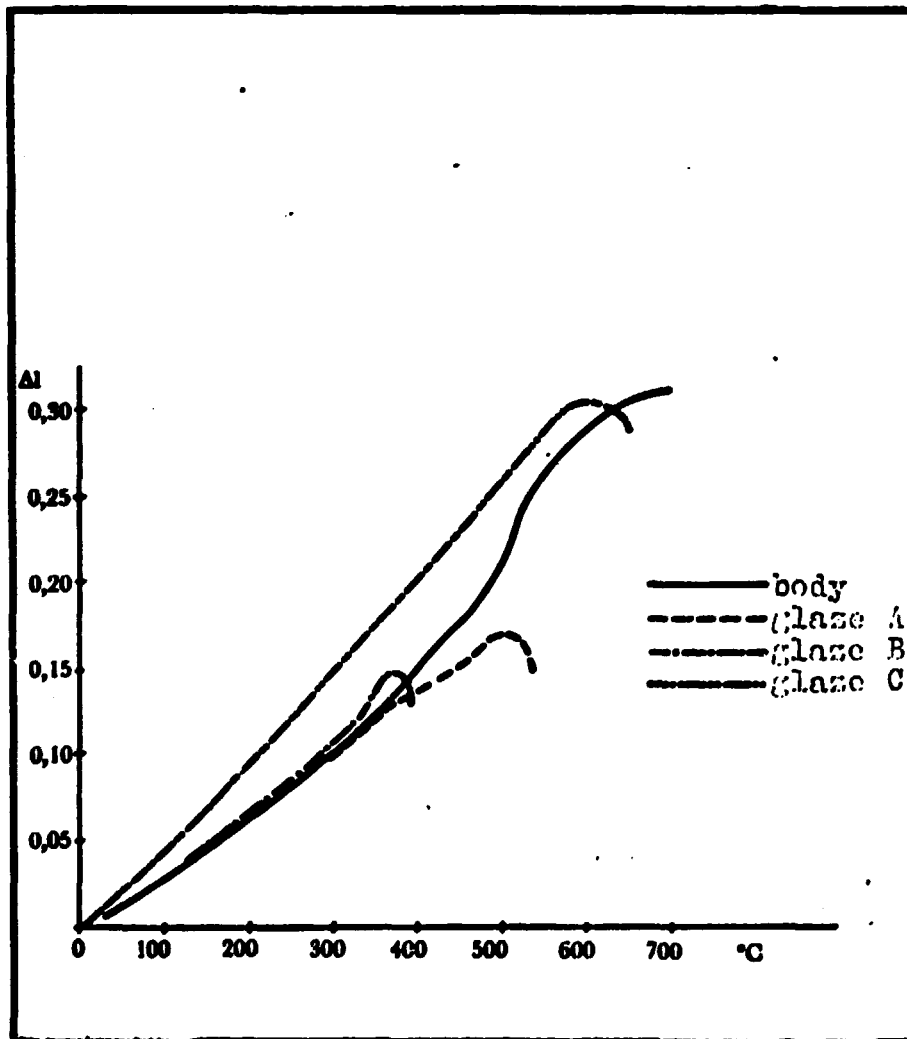
e) Preliminary tests

The body which has met the aforesaid requirements can be subjected to a verification test for which at least 3 to 5 kg of the blend is required.

The blend prepared according to the adapted recipe is to be weighed, in the amount of at least 3 to 5 kg, into the small porcelain drum mill depending on its capacity. The ratio of solids : milling nebbles : water is usually chosen to be 1 : 1 : 1. The blend is to be wet ground to the grain size of 2 to 3% rest on 0,063 mm mesh sieve (10,000 openings per sq.cm). The slurry then is to be poured from the small drum mill over a checking sieve with mesh sizes of 0.2 to 0.315 mm; the slurry is then to be dewatered in a suitable way (in a bag made of filter cloth, in

Picture No. 2

Thermal Expansion of a Lime-siliceous Wall Tile Body and Three Glazes with Different Coefficients of Thermal Expansion



Picture No. 3

Thermal Expansion Coefficients  
of the Body and Glaze Are Equal



Picture No. 4

Thermal Expansion Coefficient of  
the Glaze is Higher than that  
of the Body.  
The Tile is Concave.



Picture No. 5

Thermal Expansion Coefficient of  
the Body is Higher than that  
of the Glaze.  
The Tile is Convex.



a dish made of plaster, etc.) and dried in a drier to the residual moisture content of 6 to 7% or dried completely to 0.5 - 1% moisture content. In the latter case the dried-up blend is to be wetted with 6% of water. The blend is to be crushed e. g. by means of a wooden or metal roller and screened through 1.5 to 2 mm mesh sieve to prepare the blend for pressing which is advantageous to be kept in a closed polyethylene bag for ageing till the next day. The blends having so been matured are to be pressed in a laboratory or industrial press under the pressure of 20 to 25 MPa into 150 x 150 x 5.7 mm wall tiles. It is advisable to keep to the wall tiles thickness within  $\pm 0.2$  mm tolerance since any different thickness of the wall tiles may affect the test of the glaze resistance to crazing. Bending strength after pressing is to be determined by testing one or two wall tiles.

Wall tiles after having been pressed are to be measured, stacked on a refractory plate and left dried in the open air till the next day; then they are to be put into a cool drier in which the temperature is to be increased gradually within 6 hours to 110 to 120°C. The dwelling time on this temperature should be at least 4 hours.

The wall tiles having been dried are to be charged either into the industrial kiln or into the small laboratory one so that the stack should be heated uniformly from all sides. The temperature is to be raised at the rate of 80 to 100°C per hour up to the temperature of 1060°C at which the dwelling time

is to be 1 hour. The kiln is to be then switched off and left to cool down freely. When the temperature drops below  $500^{\circ}\text{C}$  the ventilating openings may be gradually opened (if they are provided at the kiln) or the kiln door gradually opened by parts. After the wall tiles have cooled down they are to be taken out from the kiln, measured and checked for crazing. Hair-line cooling cracks can be made more distinct by painting them with a water coloured by methylene blue or by sound when wall tiles are knocked on. The curvature of the face area is to be determined on five marked wall tiles to judge the curvature due to the glaze afterwards.

The wall tiles free from any defects are to be glazed with the glaze envisaged for porous wall tiles manufacture. The glazed wall tiles to be fired are either charged into an industrial kiln or put into a sagger with which they are put into a laboratory kiln. The temperature in the kiln is to be increased at the rate of 100 to  $150^{\circ}\text{C}$  per hour till the glaze firing temperature is reached. The maturing time of 15 minutes on the maximum temperature is to be chosen.

The wall tiles having been fired are to be checked for dimensions, curvature of face areas, rectangularity and curvature of face edges. The wall tiles then are to be divided into three groups:

- the wall tiles of the first group are to be tested for bending strength and after that the halves are to be tested for water absorption;



- the wall tiles of the second group are to be tested for resistance of glaze against crazing according to Harkort;
- the wall tiles of the third group are to be tested for resistance against crazing in an autoclave or for changes of face area curvature by boiling. The coefficient of thermal expansion can be determined, too.

f) Evaluation of tested blends

The properties of the fired wall tiles found out are to be evaluated as follows:

The dimensional stability can be found out from the dimensions of the fired wall tiles, from the curvature of the face edges and from the rectangularity. The difference between the dimension of the edges and the dimension of the mould should not exceed minus 0.5%. Dimensional deviations and curvature of face edges must not exceed the respective Quality Standards.

The harmony between the thermal expansion of the glaze and of the bisque can be judged from the difference between the curvature of the face areas of the fired glazed wall tiles and the curvature of the bisque face areas and also from the resistance of the glaze to crazing when subjected to the Harkort's test.

The change in the curvature of the face area of the glazed wall tiles against the curvature of the bisque should be at least + 0.15%, i. e. towards convexity. The resistance of the glaze against crazing according to Harkort is required to be at least 150°C for white glazes and at least 120°C for coloured glazes according to the most Standards.

By the testing in the autoclave the resistance of the wall tile bisque against the expansion due to moisture expansion is to be found out. The product of a pressure and exposure time is usually given different, the mean value is 0.9, i. e. 3 hrs at 0.3 MPa pressure or 4.5 hrs at 0.2 MPa pressure. If no cracks occur on the wall tiles after the aforesaid exposure in the autoclave the bisque moisture expansion is all right.

If no autoclave is available the moisture expansion may be judged from the change in curvature of the face area of the glazed wall tiles after firing and after the absorption capacity has been determined the latter must not exceed 0.20% value.

In case the resistance of the glaze against crazing on the developed bisque is high, the wall tiles are convex, the bisque shows no shrinkage but rather expansion as against the mould dimension and higher number of the wall tiles (more than 10%) crack during the firing, it indicates that there is too much free silica in the bisque. Therefore, the addition of silica component should be made smaller

and the decrease is to be substituted by breakage or by a slight increase of the plastic and calcareous components.

g) Adaptation of blend composition to achieve the required properties

The following corrections in the blend composition are to be made according to the values found out during the manufacture of the test wall tiles.

If the strength of the pressings and dried-up pressings is found low (below 0.5 MPa) the plastic component proportion is to be increased or the less plastic clay or kaolin should be substituted with a more plastic clay while keeping the ratio of the main oxides in the blend unchanged.

If wall tile shrinkage after drying exceeds 0.3% or cracks occur during the drying the proportion of the plastic component should be decreased or the too much plastic clay substituted with a less plastic one. Hence, the requirement for a sufficient strength after pressing and drying is contradictory to the requirement for the low or no shrinkage by drying and resistance against cracking. It is, therefore, necessary to choose a suitable compromise between these requirements.

Also contradictory is the requirement for a sufficiently high coefficient of thermal expansion of bisque and a resistance against cracking of the bisque during firing. Sufficiently high coefficient of thermal expansion of bisque and, thereby, sufficient resistance of the glaze against crazing require an increased content of silica component in the bisque which, however, makes the bisque more prone to cracking during firing especially during the cooling phase.

Unless a suitable compromise can be found from the raw materials being available to meet the both requirements, the glaze resistance against crazing being just at the required limit or slightly above it and despite that the wall tiles crack during firing it may be useful to choose a calcareous-magnesium type body instead of the lime-siliceous type body. The calcareous-magnesium type body having sufficiently high thermal expansion is enough resistant against cracking but on the account of slightly higher shrinkage. In such a case a part of the calcareous component is to be substituted with the calcareous-magnesium one (dolomite) so as the ratio of  $\text{CaO} : \text{Al}_2\text{O}_3$  should remain unchanged for the formation of anortite (according to chemical modulus C) while the content of free silica is to be decreased by the amount of the MgO content. In such a case, however, slightly higher firing shrinkage - about 1% - should be accepted. Nevertheless, the bisque will reach sufficiently high thermal expansion and resistance against cracking during firing and the shrinkage should be compensated by a larger size of the pressing mould.

Calcareous-magnesium siliceous body shows also a lower moisture expansion than the lime-siliceous one so that it guaranties a resistance against crazing while tested in the autoclave whereby it ensures that the glaze will not craze on the finished tiling during its entire lifetime.

h) Verification tests

When the wall tile body has been adapted according to the above mentioned principles so that it has the required properties in green state, during firing and after firing final verification tests may be commenced.

Following the final adapted recipe at least 200 to 300 kg of the blend is to be weighed and charged into the drum mill or in several batches if the drum mill capacity is lower, as the case may be. If no laboratory spray drier is available the ratio of solids : water : mill balls 1 : 1 : 1 is to be chosen. In case the pressing clay is dried and prepared in the spray drier liquefying tests are to be made first to achieve good fluidity of the slurry while the solids content in the slurry being at least 60%.

The body is to be milled to 2 - 3% rest on 0.063 mm mesh sieve (10,000 openings/sq.cm), discharged over a checking sieve with 0.1 mm aperture

size and dewatered in the spray drier to 3.5 - 5% moisture content whereby the pressing blend is obtained directly or in a filter press and subsequent drying and preparation of the pressing blend in a pan mill with a screening path with openings of 2 to 2.5 mm diameter. The pressing blend from the pan mill should have a uniform 5 to 6% moisture content and its granulometric composition should not contain any larger proportion of particles below 0.2 mm. A too fine blend cannot be well deaerated during pressing that may result in a formation of a laminated structure of the pressing caused by pressed-in air which could not have been released in time from the mould during the deaerating stroke of the press.

Wall tile samples are continuously taken during the pressing process to determine bending strength. The pressing force under which the wall tile has been pressed is to be recorded at the same time to find out the optimum pressure under which the wall tiles possess the highest strength, no signs of laminar structure and no sticking of the mass to the press punch occurs.

The pressed wall tiles are stacked into columns on fireclay plates and left for drying in open air till the next day. They are charged into a drier the next day in which they are dried up slowly to final moisture content below 0.5%. The dried-up wall tiles are also tested for bending strength and the wall tiles and their dimensions

are to be checked for cracks occurrence during drying and, if necessary, they are inspected by means of painting them with kerosene.

The wall tiles then are to be charged for firing after which their dimensions and bending strength are to be found out. The water absorption is to be determined on the about halves of the wall tiles. The same samples are used to determine their bending strength. The other wall tiles are to be sorted and the number of the cracked ones found out after firing should not exceed 10%. Twenty wall tiles are to be marked and the curvature of their face areas measured. All the undamaged wall tiles including the marked ones are then glazed and fired at the temperature for which the glaze has been composed (960 to 1020°C). At least a 100 wall tiles are to be measured after firing and deviations from the straightness of the face edges, from the rectangularity and curvature of the face areas are to be determined. These values are to be evaluated by mathematic-statistical methods. The dimension of the pressing mould for the future production is to be chosen according to the ratio of the pressing mould dimension to the mean dimension of products so as the products should correspond to the required dimensional accuracy.

At least twenty wall tiles each are to be used for the determination of bending strength, resistance against glaze crazing by Harkort's test and resistance against moisture expansion by an autoclave test.

The water absorption is to be determined on the same wall tiles used for the bending test.

If all the test results correspond with the Quality Standards the developed wall tile body is considered to be satisfactory and may be recommended for the start of the trial production. In case some of the wall tile parameters are unsatisfactory an adaptation is to be made in compliance with the above mentioned principles and the verification test is to be repeated till the wall tiles produced conform with the quality standard in all aspects.



### 3. Kaolinitic Wall Tile Bodies Composition

The plastic component of bodies of this type is usually formed by ball clays which yield usually a porous light-coloured bisque after having been fired at 1200°C temperature. These clays are characterized by the following chemical composition:

loss on ignition		6 to 15%
silicon dioxide	SiO <sub>2</sub>	50 to 75%
aluminum oxide	Al <sub>2</sub> O <sub>3</sub>	20 to 38%
ferric oxide	Fe <sub>2</sub> O <sub>3</sub>	1 to 2%
titanium dioxide	TiO <sub>2</sub>	0.5 to 2%
calcium oxide	CaO	up to 3%
magnesium oxide	MgO	up to 2%
potassium oxide	K <sub>2</sub> O	up to 2%
sodium oxide	Na <sub>2</sub> O	up to 2%

From the physico-technological point of view the requirements on the plastic component (clays) are similar to those for bodies of lime-siliceous type, i. e. sufficient strength after pressing and drying and resistance against cracking during drying and firing.

Fired kaolin or ball clay and fired breakage are used as a non-plastic component for this type of wall tile body since the content of silica (as an unburnt grog) in the body is limited. Kaolin or clay grog must be fired at the bisque firing temperature at

least and its average water absorption after firing should range between about 10 and 15%. The heat consumption of this type of body is still increased by the firing of grog.

The wall tile body composition proper of the kaolinitic type is as follows:

ball clays	35 to 55%
burned kaolin	30 to 50%
fired breakage (reject)	5 to 15%

Informative body composition testing is to be carried out in a similar way as in the case of the lime-siliceous wall tile body while only the bisque firing temperature is about 1200°C.

The kaolinitic type wall tile body has a lower coefficient of thermal expansion than the lime-siliceous one because of the lower contents of silica and fluxes. Therefore, to secure the glaze resistance to crazing, glazes with a lower coefficient of thermal expansion are required having always a higher firing temperature, i. e. 1030 to 1120°C.

If the test results of wall tiles made of the experimental blends do not meet the requirements the following correction in the body composition is to be made:

If the wall tile glaze has a low resistance against crazing (Harkort's test) the content of silica in the body should be increased either by using clay with a higher content of  $\text{SiO}_2$  or by adding raw kaolin or silica sand.

If the bisque has a low strength or high water absorption after firing a more vitrifying clay is to be used instead of purely porous clay after firing or fluxes are to be added into the blend such as a mixture of feldspar and dolomite milled beforehand.

Bodies of this type show rather high shrinkage and, therefore, the products made of these bodies are to be either calibrated after bisque firing or they are to be measured after glaze firing and sorted according to their dimensions.

#### 4. Semi-siliceous Wall Tile Bodies Composition

Wall tile body of this type has a slightly higher coefficient of thermal expansion than the kaolinitic type body and, therefore, glazes with a lower glaze firing temperature and a higher coefficient of thermal expansion than those of the kaolinitic bodies can be applied in the production of wall tiles.

It is advantageous to use clays with a higher content of silica and feldspars as a plastic component for this type of a body. Otherwise, raw kaolin, silica sand or other siliceous raw material such as pegmatite are usually added.

Practical composition of this body ranges as follows:

plastic clays (or siliceous ones)	35 to 50%
fired kaolin or clay	10 to 30%
raw kaolin	15 to 25%
pegmatite	0 to 15%
fired breakage (reject)	5 to 15%

Testing of the body composition is carried out similarly as of the lime-siliceous body composition. Same principles as for the kaolinitic body are applicable in the adaptation to achieve the required properties, i. e. when glaze resistance to crazing is low the  $\text{SiO}_2$  content should be increased by adding a silica

component or to substitute a clay of lower silica content with a clay of higher silica content. Unless the required strength or water absorption are obtained more fluxes are to be added or the bisque firing temperature increased.

This type of a body shows firing shrinkage, too and, therefore, it is necessary to take the calibration of the bisque into account to obtain constant sizes of the products or to sort the finished products dimension-wise to get groups of products of nominal sizes. This type of a body is also more sensitive to moisture expansion whereby the glaze resistance to crazing after wall tiling is decreased.

## 5. Feldspathic Wall Tile Bodies Composition

Fired grog such as fired kaolin or clay are not applied for this type of a wall tile body but only bisque and glazed reject are used as fired grog in order to be utilized economically. Plastic body component suitable for this purpose is similar to that for the semi-siliceous type of a body, i. e. a plastic clay with a higher content of silica or feldspars slightly vitrifying at the bisque firing temperature. About 10% of feldspar or a proportionally higher amount of pegmatite are added to obtain the required strength and an admissible absorption capacity. Feldspathic body can also be applied for crockery making.

The typical body composition of this type according to the use of feldspar or pegmatite is as follows:

	a)	b)
plastic siliceous clay	40 to 50%	45 to 55%
raw kaolin	25 to 35%	15 to 25%
feldspar	5 to 15%	-
pegmatite	-	10 to 35%
breakage (reject)	5 to 10%	5 to 10%

This type of a body shows usually a very low drying and firing shrinkage and, hence, no calibration of bisque or dimensionwise sorting must be necessary. However, the body is prone to moisture expansion and there can be a risk of glaze crazing on a finished wall tiling particularly if it is not in a perfectly

dry environment and if the body composition is not perfectly balanced to the firing temperature. Another disadvantage of this type of a body is its rather high tendency towards cracking during firing both in the heating as well as in the cooling zones within the temperature range of the reversing modification transformation  $\alpha \rightleftharpoons \beta$  silica at about  $573^{\circ}\text{C}$ . therefore, it is essential to pay attention to the firing curve, lest sudden temperature changes should take place in these zones.

## 6. Mixed Siliceous Wall Tile Bodies Composition

This type of a wall tile body shows a considerable dimensional stability and neither calibration nor dimensionwise sorting are generally required. Plastic clays containing silica and/or even feldspars are used as raw materials. Raw kaolin, silica sand or other siliceous raw material are the other components used for its preparation. Unless the clays used contain enough feldspars about 2 to 3% of feldspar or 5 to 10% of pegmatite are to be added. About 8% of limestone or identical quantity of dolomite form another component of the body. This type of a body does not generally show any tendency towards an excessive moisture expansion. Mixed siliceous body can also be applied for crockery making.

Testing of raw materials suitability and body composition is to be carried out similarly as in the case of lime-siliceous body. Unless the required properties are obtained the adaptations to be made are analogous to the other types of bodies. When the glaze resistance to crazing is low the silica content should be increased either by using clay with higher silica content or by increasing the addition of a silica component. In case of a low strength or high water absorption the proportion of feldspar or pegmatite should be slightly increased on the account of the silica component and the addition of limestone or dolomite is to be little increased, too.



This type of a body, similarly as the foregoing one, may also be prone to cracking in the zone of 573<sup>o</sup> temperature due to the modification transformation and, hence, attention is to be paid to the firing curve.

The typical body compositions of mixed siliceous wall tile bodies depending on the use either of silica sand or raw kaolin is specified as follows:

	a)	b)
plastic clay	40 to 50%	35 to 50%
silica sand	30 to 35%	-
raw kaolin	-	30 to 40%
feldspar	2 to 5%	2 to 5%
limestone or dolomite	5 to 10%	5 to 10%
breakage (reject)	5 to 10%	5 to 10%

## 7. Talcum Wall Tile Bodies Composition

This type of a wall tile body may come into consideration only where enough main raw material - talcum of sufficient purity is available.

The composition of this body is very simple. About 20 to 25% of plastic clay and 75 to 80% of talcum are used. It is necessary to find a suitable glaze for this body. Such a glaze should show rather low coefficient of thermal expansion with a sufficient resistance against crazing. This type of a body is not prone to moisture expansion.

Unless suitable glaze could be found for this body it may be necessary to switch over partly or completely to that type of talcum body, shown under I in the Table No. 1 which has a higher coefficient of thermal expansion. This can be achieved by substituting a part of the talcum by a clay of higher silica content or by adding raw kaolin or another siliceous component with an addition of small amount of a feldspatic raw material and limestone.

The typical compositions of talcum wall tile body is shown below:

	I.	II.
Plastic clay (ball clay)	-	20 to 25%
Plastic siliceous clay	45 to 60%	-
Feldspar	up to 3%	-
Limestone (or dolomite)	up to 2%	-
Talcum	40 to 45%	75 to 80%
Breakage (reject)	0 to 10%	0 to 10%

### 8. Wollastonite Wall Tile Bodies Composition

The occurrence of a natural  $\beta$  - wollastonite is a prerequisite for the manufacture of this type of wall tiles. In some countries even synthetic wollastonite is used but that would be rather uneconomical for the manufacture of double-fired wall tiles. Synthetic wollastonite as raw material for a wall tile body is chiefly used in the manufacture of one-fired wall tiles.

The basic composition of the wollastonite wall tile body is very simple - 25 to 40% of plastic clay and 60 to 75% of wollastonite. Unless a suitable glaze with corresponding thermal expansion would be available for this type of a body the coefficient of thermal expansion of the body should be enhanced by the addition of a siliceous component or by using a clay with higher content of silica.

Typical composition of wollastonite wall tile body is as follows:

plastic clay (siliceous)	25 to 40%
wollastonite	60 to 75%

#### IV. CONCLUSIONS

The presented paper gives a description of practical composing of bodies for porous wall tiles so as their properties should correspond with the respective quality Standards and, owing to their appearance and utility value, such wall tiles should enhance culture of living.

It is not the aim of this publication to give any theoretical justification of various types of bodies since it would become too extensive. Besides, the theoretical principles of composing bodies for wall tiles can be found in technical literature, articles, publications, etc.

The publication does not cover glazes for various types of porous wall tile bodies either because when wall tile manufacture is to be implemented in such places where they have not yet been made it would be very difficult to start the manufacture of frits and glazes for wall tiles, too. Hence, it is more advisable to approach some world manufacturers of frits and glazes for supplying a suitable type of a glaze for the particular type of a body which is envisaged to be made in compliance with the raw materials available.

On the other hand, the publication gives, in a popular way, an instruction how to verify local

non-metallic raw materials in view of their possible exploitation for the manufacture of wall tiles in developing and the least developed countries. It shows main types of body compositions while transiting types of compositions can be created in accordance with local raw materials.

It is an aid for all technical schools, laboratories, institutes, consulting companies, mining firms of non-metallic raw materials and ceramics manufacturers intending to utilize local raw materials for an industrial development.

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Thermal Expansion Coefficient of the Body  
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