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(54) **Title:** PRODUCT

(57) **Abstract:** A method of making a product including mixing together slate particles, clay particles, and water to a generally formable homogenous mixture; the slate particles and the clay particles of the mixture, substantially all being sized so as to pass through a sieve of 1 mm, and such that not more than 25% of the slate particles have a size less than 63 microns, and the ratio by weight of the slate to clay in the mixture being between 40% and 80% of the particles, the method including forming the wet mixture to a desired cross sectional shape by extruding the mixture, at least partially drying the formed mixture, cutting the extruded formed mixture into required lengths, and firing the cut lengths at a temperature of at least 950°C and for a time sufficient for at least some of the clay to convert into a binder to bind together the slate particles

Product

5 Description of Invention

This invention relates to a method of making a building product, and to a product made by the method. By way of example, the product may be a tile such as a roofing, flooring, or wall tile, or panel, or a brick or brick slip.

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European patent EP 0958258B discloses a method of making a product based on slate particles. In that method, a wet generally homogenous mixture of slate particles and clay is made, formed to a desired configuration, at least partially dried and then fired at a temperature sufficiently high to cause at least some of the clay to convert into a binder to bind together the slate particles. The process by which the mixture is formed to the required configuration for, e.g. a roofing tile, includes the placing of a ball of the wet mixture in a mould wherein it is subject to pressure to cause the mixture to fill the mould and thereby acquire the required configuration.

20

Whilst such a forming process is satisfactory, it would be desirable if other forming processes, such as extrusion directly to a required configuration, without any subsequent pressing process, could be utilised, but with the mixture disclosed in our patent aforesaid such a technique has not been usable; it has not been possible to obtain a suitable finish on the products.

25

With the aim of addressing this problem, according to one aspect of the present invention we provide a method of making a product including mixing together slate particles, clay particles, and water to a generally formable homogenous mixture, the slate particles and the clay particles of the mixture, substantially all being sized so as to pass through a sieve of 1mm, and such that not more than 25% of the slate particles have a size less than 63 microns,

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and the ratio by weight of the slate to clay in the mixture being between 40% and 60% of the particles, the method including forming the wet mixture to a desired cross sectional shape by extruding the mixture, at least partially drying the formed mixture, cutting the extruded formed mixture into required lengths, and firing the cut lengths at a temperature of at least 950°C and for a time sufficient for at least some of the clay to convert into a binder to bind together the slate particles.

As is the case in EP 0958258B, when the dried formed mixture is fired at the appropriate temperature, the clay binds together the slate particles to form a vitreous product of low, preferably substantially no, porosity having the required mechanical strength. The smaller particle size provided in accordance with the present invention, compared to the particle sizes referred to in the aforesaid patent, has the effect that the mixture, with a suitable water content, is able to be extruded with the extruded material having a satisfactory finish and dimensional accuracy without requiring a pressing process in addition to the extrusion. The extruded material may simply be cut to appropriate lengths to form the products, ready for drying and firing.

The slate particles may comprise fragments, slivers, powder and dust, able to pass through the 1mm-sized sieve.

The provision that not more than 25% of the slate particles have a size less than 63 microns is helpful in avoiding lamination problems in the finished product. When we refer to percentages in relation to the composition of mixtures used in the invention, we mean the percentages by weight thereof.

The solid constituents of the mixture may comprise substantially only slate particles and clay, although it will be appreciated that a small quantity (e.g. up to 5% or so of the solid content of the mixture) of a colouring agent or agents may be included in the mixture.

Preferably the clay content is approximately 50% i.e. the mixture comprises substantially equal quantities by weight of slate particles and clay particles.

If the finished product, after firing, is required to be of "red brick" colour, approximately 3% iron oxide may be added as a colouring agent. If a "Staffordshire blue brick" colour is required, approximately 3% iron oxide and 2% manganese dioxide may be used as a colouring agent.

In producing such coloured products, or when producing a "natural" (i.e. slate) coloured body, approximately 0.02% precipitated barium carbonate may be included in the mixture. This can be beneficial in preventing or reducing efflorescence in the final product.

For satisfactory extrusion of the mixture, sufficient water is required in the mixture to enable it to flow satisfactorily for extrusion to form it to the cross-sectional shape required in the finished products, the extrudate being cut into lengths to form the individual products.

Because clay powder mixes with water more readily than particles of slate mix with water, the method preferably includes mixing the slate particles with water prior to the introduction of clay to the mixture.

According to another aspect of the invention, we provide a method of making a product utilising at least slate particles, clay particles and water, comprising firstly mixing a quantity of the slate particles with sufficient water to wet the particles over substantially their entire surface area; and subsequently mixing a quantity of the clay particles with the slate particles and optionally with additional water, to produce a generally homogenous mixture formable to a desired configuration; forming the mixture to the shape of the product; at least partially drying the formed mixture, and subjecting the formed mixture to firing

under conditions to cause at least some of the claim to convert into a binder to bind together the slate particles.

5 In accordance with the second aspect of the invention, the sizes of the slate and clay particles and relative proportions thereof, additional constituent materials, and the forming of the product may be as above set forth in relation to the first aspect of the invention.

10 Preferably the slate particles are initially mixed with at least sufficient water to ensure thorough wetting thereof over substantially their entire surface area, before the addition of clay. This water may be about 10%-15%, preferably about 12%-14%, of the weight of the slate particles. Further water may be added to the mixture when the clay is added, to achieve a water content enabling the mixture to be formed into products. Different types of slate, such
15 as those obtained from different quarries, may require different quantities of water to achieve the surface area wetting.

The initial mixing of the slate particles with water may be carried out in a first mixer, e.g. an Eirich or Eirich-type mixer with a mixing paddle rotating about an
20 upright axis in a mixing receptacle. In a "batch" process, a required quantity of slate particles may be weighed and introduced into such a mixer, followed by the weighed required quantity of wetting water, and the two mixed together in the mixer prior to the addition of the weighed required quantity of clay thereto and further mixing.

25

The mixture from the first mixer may be supplied to a further mixer in which any required further additions, e.g. a colouring agent or agents and/or barium carbonate, are added, and further mixing carried out with the possible addition of further water to bring the mixture to an extrudable condition.

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Such a further mixer may be a Winkworth or Winkworth-type mixer.

As an alternative to the addition of a predetermined quantity of water to the slate particles, it would be possible for the slate particles to be treated by immersion and agitation in a greater quantity of water, with excess water subsequently being drained off the wet slate particles to leave the required water content in the slate before the clay is added thereto.

The firing temperature may be up to 1200 °C or possibly even higher. Such temperatures provide for products of sufficient mechanical strength and water resistance.

Preferably the firing temperature is between 980°C and 1040°C and in a particular example of the invention, the firing temperature may be in the range 1020°C to 1040°C, the lower temperature being more appropriate for firing "blue" products and the higher temperature more appropriate for firing "red" products.

It will be appreciated that the required temperatures and times for firing products will depend on the dimensions, particularly the cross-sectional sizes, of the products. In general, lower firing temperatures will require longer firing, while higher firing temperatures will allow shorter firing times as long the cross-sectional size of the products is not too great.

When firing to produce a "blue" product the firing may be carried out under reducing conditions, while to produce a "red" product oxidation conditions may be applied in the firing.

European patent EP0958258B refers to the possibility that feldspar particles may be included in the mixture. In accordance with the present invention, preferably the mixture includes no or substantially no feldspar particles. Such particles cause difficulty in the extrusion process, and their presence may make the finished products unduly brittle.

In one example of making products in accordance with the invention, a quantity of slate particles of the required size (as described hereafter), able to pass through a 1mm-size sieve, is weighed out and mixed with the respective quantity of water. To this end, the slate is delivered, e.g. by conveyor, to a weighing/loading hopper from which a required quantity of it is supplied to a mixer, and sufficient water added to it in the mixer, to enable mixing to cause all the slate particles to be coated with water. The required quantity of clay is then added to the wet slate and thoroughly mixed therewith. Adding the water and clay to the slate in this order obviates potential problems with the clay "balling-up" in consequence of its preferential, in comparison with slate particles, mixing with water.

The above-described mixing stages may be carried out using an Eirich mixer.

The whole of the required water content of the mixture may not be added in the above process stages. By way of example, approximately 12% (of the weight of the slate) of water may be added, with any necessary additional water, to bring the mixture to an extrudable consistency being added at a later stage accompanied by further mixing.

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The mixture resulting from the above stages is passed through a 1mm sized sieve, and then delivered to a further mixer at which the required quantity of colouring/staining agent/barium carbonate is added while mixing is continued. The further mixer may be a Winkworth mixer having mixing paddles rotating in a horizontal drum. From here the mixture is delivered to an extruder which may include a two-shaft mixing device wherein further water is added to the mixture, which is mixed and de-aerated to bring it to an extrudable consistency. The amount of such additional water which might be necessary depends largely on the exact proportions and particle sizes of the constituents of the mixture, and will be a matter of the judgement of a skilled operator of the process.

Alternatively, if the mixture were not required to be extruded but instead pressed to its required configuration, it could be taken directly from the Winkworth mixer with no further water being added .

- 5 By carrying out the addition of colouring agent and/or additional water in the further mixer, the effect is achieved that if it is required to change the colour of products being manufactured, or to change from extruded products to pressed products, cleaning out of the entire apparatus being used including the first mentioned (Eirich) mixer in which the initial mixing of slate with water, and then
10 with the clay, is carried out, is not required. Cleaning out of only the further mixer and extruder may be necessitated.

A further possible pre-extrusion treatment to which the mixture may be subject is a sieving stage.

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- When the mixture, extruded to the appropriate cross-sectional shape, has left the extruder it is cut into appropriate lengths and sizes to provide finished (ready for drying and firing) products of the required dimensions. It may, after extrusion, be subjected to a surface treatment, e.g. it may have a sand/stain
20 agent or mixture applied on one or more surface portions, but no other operation to form or shape the products may be necessary prior to their drying and firing.

- The extruded products may be generally flat or slightly curved, or may
25 incorporate surfaces inclined to one another. They may incorporate one or more surfaces or surface areas which are grooved. If a product of, e.g. L-shaped cross-section is required, it would be possible for the mixture to be extruded to a cross-sectional shape having an interior cavity, the extruded material being cut longitudinally into two or more elements each having a
30 cross-sectional shape part of that of the extruded material, as well as being cut transversely of the direction of extrusion to form the individual extruded

products. For example, the material may be extruded into a hollow rectangular cross-sectional shape, which is cut longitudinally to produce products with one or more "corners".

- 5 Such cutting of an extruded section is preferably carried out after it has been subjected to at least initial drying, so that it is able to retain its shape. The cutting may be done after firing, when the product is fully stable.

The products thus obtained are subsequently stacked onto kiln trucks,
10 whereon they are subjected to a drying process e.g. four, 10-24 hours at a temperature of around 90°C. Possibly microwave heating may assist the drying process. When drying to 1% or less moisture content has been completed, the products are transferred to a kiln at which firing takes place e.g. at the temperatures indicated above. The firing process may, in a
15 conventional kiln, take place over a period of up to around 24 hours.

Products resulting from performance of the method of the invention may use a high proportion of recycled material. Thus, the slate particles may comprise mill fines, dust, or powder recovered from slate processing. The clay may
20 comprise unfired clay dust recovered from clay processing, e.g. in brick manufacture, or recycled clay. It will thus be appreciated that the invention enables effective use to be made of what would otherwise be waste products. If waste slate or clay is in the form of particles too large for use in the process of the present invention, it may be processed to reduce its particle size, e.g. by
25 roller or ball milling.

As far as the slate is concerned, it has been found that a stronger product may result if the slate is obtained by milling larger particles of slate to the size described above. In particular, products made with slate may better be able to
30 be provided with holes or slots for fastenings, providing an increased

resistance to pulling-out of fastenings, compared with products wherein the slate is the poor-quality material created as waste in slate processing.

5 To provide slate of which substantially all the particles are able to pass through
a sieve of 1mm size, while only a small proportion of the clay particles have a
size less than 63 microns, the raw stock of slate material, possibly milled as
above referred to, may be subject to a washing out process using water,
based on a sieve size of 63 microns. Since recovered slate may have a very
large proportion of very fine dust, such a washing out process may remove up
10 to above 20% of the raw, possibly milled, slate stock, but leaves a very small
quantity of slate with too small a particle size.

As alternatives to such a washing-out process, dry sieving may be utilised, or
possibly a washing-out process not involving sieving.

15

The above examples are given with reference to using slate particles of "blue"
Penrhyn slate. Slate from a different source, or "green" slate, may require a
different quantity of wetting water.

20 When used in this specification and claims, the terms "comprises" and
"comprising" and variations thereof mean that the specified features, steps or
integers are included. The terms are not to be interpreted to exclude the
presence of other features, steps or components.

25 The features disclosed in the foregoing description, or the following claims, or
the accompanying drawings, expressed in their specific forms or in terms of a
means for performing the disclosed function, or a method or process for
attaining the disclosed result, as appropriate, may, separately, or in any
combination of such features, be utilised for realising the invention in diverse
30 forms thereof.

Claims

1. A method of making a product including mixing together slate particles, clay particles, and water to a generally formable homogenous mixture, the slate particles and the clay particles of the mixture, substantially all being sized so as to pass through a sieve of 1mm, and such that not more than 25% of the slate particles have a size less than 63 microns, and the ratio by weight of the slate to clay in the mixture being between 40% and 60% of the particles, the method including forming the wet mixture to a desired cross sectional shape by extruding the mixture, at least partially drying the formed mixture, cutting the extruded formed mixture into required lengths, and firing the cut lengths at a temperature of at least 950°C and for a time sufficient for at least some of the clay to convert into a binder to bind together the slate particles.
2. A method according to claim 1 wherein the cutting of the formed mixture into required lengths is carried out before the at least partial drying thereof.
3. A method according to claim 1 wherein the cutting of the formed mixture into required length is carried out after the at least partial drying thereof.
4. A method according to any one of the preceding claims wherein the slate particles and clay particles comprise substantially 100% of the solid constituents of the mixture.
5. A method according to any one of the preceding claims wherein the mixture comprises substantially equal quantities of slate particles and clay particles.
6. A method according to any one of the preceding claims wherein the mixture further comprises up to about 5% of the weight of slate and clay particles in the mixture, of at least one colouring agent.

7. A method according to claim 6 wherein a colouring agent comprises iron oxide.
8. A method according to claim 6 or claim 7 wherein a colouring agent
5 comprises manganese dioxide.
9. A method according to any one of the preceding claims wherein the mixture includes barium carbonate.
- 10 10. A method according to claim 9 wherein the mixture further includes about 0.02%, of the weight of clay and slate particles in the mixture, of barium carbonate.
11. A method according to any one of the preceding claims comprising
15 initially mixing slate particles with water and subsequently mixing in the clay particles.
12. A method according to claim 11 wherein the slate particles are initially mixed with at least sufficient water to wet substantially all their surface area.
- 20 13. A method according to claim 12 wherein the slate particles are initially mixed with about 10% to 15%, of the weight of the slate particles, of water.
14. A method according to claim 13 wherein the slate particles are initially
25 mixed with about 12% to 14%, of the weight of slate particles, of water.
15. A method according to any one of claims 11 to 14 wherein initial mixing of the slate particles with water and subsequently with the clay particles is carried out in a first mixer.

16. A method according to any one of claims 11 to 14 wherein initial mixing of the slate particles with water is carried out prior to introducing the slate particles to a first mixer.
- 5 17. A method according to claim 11 or any claim appendent thereto, wherein additional water is added to the mixture prior to its extrusion.
18. A method according to claims 15 or 16 as appendent to any one of claims 6 to 10 or to claim 11 or 12, wherein the colouring agent(s), and/or
10 barium carbonate is added in a further mixer.
19. A method according to claim 15 or 16 wherein the first mixer is an Eirich or Eirich-type mixer.
- 15 20. A method according to claim 18 or claim 19 wherein the further mixer is a Winkworth or Winkworth-type mixer.
21. A method according to any one of the preceding claims wherein the mixture is passed through a sieve prior to extrusion,
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22. A method according to any one of the preceding claims wherein the firing temperature is up to about 1200°C.
23. A method according to any one of the preceding claims wherein the
25 firing temperature is in the range 980°C to 1040°C.
24. A method according to any one of the preceding claims wherein the firing temperature is in the range 1020°C to 1040°C .

25. A method according to any one of the preceding claims, wherein the extruded and cut products are subjected to surface treatment only prior to drying/firing.
- 5 26. A method according to any one of the preceding claims wherein the product is of generally flat configuration.
27. A method according to any one of claims 1 to 24, or claims 27 or 28
10 appendent thereto, wherein the mixture is extruded to a cross-sectional shape having an interior cavity.
28. A method according to claim 27 wherein the extruded material is cut longitudinally into two or more elements each having a cross-sectional shape part of that of the extruded material.
- 15 29. A method according to claim 28 wherein the cross-sectional shape of the extruded material is generally rectangular and that of each element includes a corner.
- 20 30. A method according to any one of the preceding claims wherein the product is a building product.
31. A method according to claim 30 wherein the product is a tile, panel, brick, or brick slip.
- 25 32. A method of making a product utilising at least slate particles, clay particles and water, comprising firstly mixing a quantity of the slate particles with sufficient water to wet the particles over substantially their entire surface area; and subsequently mixing a required quantity of the clay powder with the
30 slate particles and optionally with additional water, to produce a generally homogenous mixture formable to a desired configuration; forming the mixture

to the shape of the product; at least partially drying the formed mixture, and subjecting the dried formed mixture to firing under conditions to cause at least some of the clay to convert into a binder to bind together the slate particles.

- 5 33. A method according to claim 32 wherein the first mixing of slate particles with water is carried out by mixing predetermined measured quantities of slate particles and water.
34. A method according to claim 33 wherein the water is about 10% to 15%
10 by weight of the slate particles.
35. A method according to claim 33 wherein the water is about 12% to 14% by weight of the slate particles.
- 15 36. A method according to claim 32 wherein the first mixing of slate particles with water is carried out by mixing the slate particles with excess water and subsequent removal of water not adhering to slate particles.
37. A method of making a product, substantially as hereinbefore described.
20
38. A product made by a method according to any one of the preceding claims.
39. Any novel feature or novel combination of features described herein.

INTERNATIONAL SEARCH REPORT

International application No PCT/GB2013/051555

A. CLASSIFICATION OF SUBJECT MATTER INV. C04B33/02 C04B33/13 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) C04B		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, INSPEC, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 958 258 B1 (DIGIVE LTD [GB]) 24 November 1999 (1999-11-24) the whole document -----	1-39
X	GB 2 394 471 A (DIGIVE LTD [GB]) 28 April 2004 (2004-04-28) the whole document -----	1-39
X	BE 899 022 A2 (BCC ENGINEERING NV) 18 June 1984 (1984-06-18) the whole document -----	1-39
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents :		
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family	
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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