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Eljárás cement alapú adalékanyag előállítására

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(54) **METHOD FOR MANUFACTURING OF SUPPLEMENTARY CEMENTITIOUS MATERIALS (SCMS)**
VERFAHREN ZUR HERSTELLUNG VON ERGÄNZENDEN ZEMENTMATERIALIEN
PROCÉDÉ DE FABRICATION DE MATÉRIAUX CIMENTAIRES SUPPLÉMENTAIRES (MCS)

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Description

[0001] The present invention relates to a method for manufacturing of supplementary cementitious materials (SCMs), i.e. pozzolans.

[0002] Fly ash is a supplementary cementitious material, which is useful for production of concretes, mortars and other mixtures comprising cement. Fly ash is a by-product of coal burning power plants and is produced worldwide in large quantities each year.

[0003] In the Swedish patent No 532790 a method is described which eliminates fluctuations in the fly ash quality due to variations in the coal chemical compositions and parameters of coal burning processes. Processing of pozzolans, i.e. fly ashes according to the patented method significantly improves performance of the concrete and provides higher level of replacement of Standard Portland cement, which leads to significant economical and environmental benefits.

[0004] Fly ash usually contains about 85% glassy, amorphous components in the form of cenosphere particles. According to ASTM C 618 fly ash is classified in two classes, Class C and Class F. The Class F fly ash typically contains more than 70% by weight of silica, alumina, and ferric oxides, while Class C typically contains between 70% and 50%. Class F is produced as a by-product of the combustion of bituminous coal. Class C fly ash has a higher calcium content and is produced as a by-product of the combustion of sub-bituminous coal.

[0005] Comprehensive research has demonstrated that a high volume fly ash concretes, where Portland cement has been replaced by fly ash to a level over 50% showed a higher long term strength development, a lower water and a gas permeability, a high chloride ion resistance, etc. in comparison with Portland cement concretes without fly ash.

[0006] At the same time a high volume fly ash concrete has significant drawbacks. One drawback is long setting times and unsatisfactory slow strength development during the period 0 to 28 days. These negative effects significantly reduce the level of fly ash used for replacement of Portland cement, e.g. about 15% in the US.

[0007] Serious problems are also related to the stability of fly ash performance. Usually variations in the chemical composition of coal and frequent changes in operating parameters of power plants cause, among others, formation of crystalline and quasi-crystalline phases, so called scoria, which leads to reduction in fly ash reactivity, so called pozzolanic activity.

[0008] Historically limestone-pozzolan mixes have been used for 2000 years ago by ancient Romans and a lot of ancient buildings as e. g. Coliseum are still in a good shape. At the same time despite the fact that according to the US Geological Survey billions of tons of natural pozzolan can be found in Western USA and in most other regions of the world. The usage of natural pozzolans in concrete in the modern building industry is very limited.

[0009] Natural pozzolans belong to materials of volcanic origin and sedimentary origin, such as diatomaceous earth. According to ASTM C 618 they are designated Class N pozzolans. The natural pozzolans are described in ACI 232.1R-00 as "Raw or calcinated natural pozzolans that comply with the applicable requirements for the class herein, such as some diatomaceous earth; opaline chert and shales; tuffs and volcanic ashes or pumicites, any of which may or may not be processed calcinations; and various materials requiring calcination to induce satisfactory properties, such as some clays or shales."

[0010] The reason why the use of natural pozzolans is very limited can be explained by the following.

[0011] The microstructure of natural pozzolan particles is characterized by high porosity, which significantly increases water demand of concrete mixes containing natural pozzolans to achieve required flowability/workability of the concrete. However, increases in water demand lead to unsatisfactory compressive strength development.

[0012] Additionally, uneven distribution of active minerals i.e. amorphous materials, e.g. due to weathering has a further and unacceptable negative impact on strength development of concrete with natural pozzolan content.

[0013] Optimized natural pozzolan particle size distribution is required in order to achieve consistent performance of high pozzolan containing concrete. An optimized particle distribution is such that there are fine and coarse fractions present.

[0014] According to the Report of American Concrete Institute ACI 232.1R-00 "Use of raw or processed natural pozzolans in concrete", properties of natural pozzolans can vary considerably, depending on their origin and therefore variable proportions of chemically active minerals. Chemically active minerals usually contain amorphous materials (e.g. amorphous silicon dioxide), which react with calcium hydroxide released during Portland cement hydration (pozzolanic reaction) and form calcium-silicate-hydrate gel [C-H-S gel] - a product similar to the resulting product of Portland cement hydration.

[0015] The present invention solves the problem of using natural pozzolans in concrete.

[0016] The present invention thus refers to a method for manufacturing of supplementary cementitious materials for replacement of Portland cement in production of mortars and concretes, where the supplementary cementitious materials comprise natural pozzolans in the form of rocks and ashes and sedimentary origin, such as diatomaceous earth.

[0017] The invention is characterized in, that the said pozzolans in crushed state are subjected to a high energetic mechanical processing by means of grinding in a grinding equipment, whereby the pozzolan particles receive mechanical impulses, and in that the grinding is carried out for a predetermined time resulting in a compressive strength of a 5.08 cm (2 inch) side cube of mortar

comprising 80 % Portland cement and 20 % natural pozzolan in a ratio of 1:2.75 to standard sand and in addition water required to obtain a flow of the mortar according to American standard ASTM C 109, which has been properly compacted under vibration and hardened at + 20 °C in sealed condition, which after 28 days is ≥ 75 % of the compressive strength of a 5.08 cm (2 inch) side cube, treated as said cube, of a mortar comprising a ratio of Portland cement:sand of 1:2.75 and in addition water corresponding to 48.5 % of the weight of Portland cement.

[0018] This test corresponds to the American standard ASTM C 109.

[0019] The present invention will be described in more detail below.

[0020] The invention refers to a method for manufacturing of supplementary cementitious materials for replacement of Portland cement in production of mortars and concretes, where the cementitious materials comprises natural pozzolans in the form of rocks and ashes of volcanic origin and sedimentary origin, such as diatomaceous earth.

[0021] According to the invention the said pozzolans in a crushed state are subjected to a high energetic mechanical processing by means of grinding in a grinding equipment, whereby the pozzolan particles receive mechanical impulses.

[0022] The grinding leads to modification of its surface properties in the form of surface porosity reduction and improvement of its chemical reactivity with alkaline medium produced by hydrating cement. It has been observed that the treatment of the pozzolan particles gives that there occurs local melting of the surface of the particles, whereby zones with a high degree of amorphization are created. By this treatment it has shown that natural pozzolans show a surprisingly good effect when the so treated natural pozzolans replace some of the amount of Portland cement in a cement paste, mortar and concrete comprising Portland cement, pozzolans, sand and water.

[0023] Further according to the invention, the grinding is carried out for a predetermined time resulting in a compressive strength of a 5.08 cm (2 inch) side cube of mortar comprising 80 % Portland cement and 20 % natural pozzolan in a ratio of 1:2.75 to standard sand and in addition water required to obtain a flow of the mortar according to American standard ASTM C 109, which has been properly compacted under vibration and hardened at + 20 °C in sealed condition, which after 28 days is ≥ 75 % of the compressive strength of a 2 inch side cube, treated as said cube, of a mortar comprising a ratio of Portland cement:sand of 1:2.75 and in addition water corresponding to 48.5 % of the weight of Portland cement.

[0024] The amount of water required to obtain a flow of the mortar according to ASTM C 109 may vary, but is approximately 40% - 50% of the weight of Portland cement and the pozzolan.

[0025] The compressive strength when using natural pozzolan corresponds to the strength which is obtained

when fly ash is used together with Portland cement.

[0026] The present invention can be realized with the use of different types of grinding equipment such as media milling equipment, e.g. stirred, centrifugal, tumbling ball or non-media milling equipment, e.g. jet, impact, roller with dominating shear mechanical impulses applied to the particles subjected to processing and combined with air classification.

[0027] The pozzolan can be subjected to grinding in a grinding equipment, which is adopted to the open or closed circuit for material being grinded.

[0028] A preferred particle size distribution is:

< 5 microns 15-50%,

≤ 10 microns 30-65%,

≤ 30 microns 90-95 %.

[0029] According to a preferred embodiment Portland cement, fly ash Class F or C, fine quartz, granite quarry fines, fine fractions of recycled concrete or blast furnace slag or its blends are added to the said pozzolan during or after said processing of the pozzolan.

[0030] According to another preferred embodiment Portland cement, fly ash Class F or C, fine quartz, granite quarry fines, fine fractions of recycled concrete or blast furnace slag or its blends are subjected to pregrinding to achieve fineness with a retention on the sieve of 45 microns which is less than 5%.

[0031] According to still another preferred embodiment water reducing agents, set time regulators, and strength accelerating admixtures in powder form are added to the said pozzolan during or after said grinding of the pozzolan.

[0032] It is preferred that replacement of Portland cement by the said pozzolan in concrete or mortar is from 15 to about 70%.

[0033] It is also preferred that the pozzolans is subjected to grinding in said grinding equipment to a fineness of the final product with a retention on a sieve of 30 microns being less than 5%.

Claims

1. Method for manufacturing of supplementary cementitious materials for replacement of Portland cement in production of mortars and concretes, where the supplementary cementitious materials comprise natural pozzolans in the form of rocks and ashes, **characterized in, that** the said pozzolans in crushed state are subjected to a high energetic mechanical processing by means of grinding in a grinding equipment, whereby the pozzolan particles receive mechanical impulses, and **in that** the grinding is carried out for a predetermined time, **in, that** the final product of the pozzolan after grinding has the following par-

particle size distribution:

- ≤ 5 microns 15-50%,
- ≤ 10 microns 30-65%,
- ≤ 30 microns 90-95 %

in, that a mixture of ground pozzolans, portland cement and water is resulting in a compressive strength of a 2 inch side cube of mortar comprising 80 % Portland cement and 20 % natural pozzolan in a ratio of 1:2.75 to standard sand and in addition water required to obtain a flow of the mortar according to American standard ASTM C 109, which has been properly compacted under vibration and hardened at + 20 °C in sealed condition, which after 28 days is ≥ 75 % of the compressive strength of a 5.08 cm (2 inch) side cube, treated as said cube, of a mortar comprising a ratio of Portland cement:sand of 1:2.75 and in addition water corresponding to 48.5 % of the weight of Portland cement.

2. Method according to claim 1, **characterized in, that** Portland cement, fly ash Class F or C, fine quartz, granite quarry fines, fine fractions of recycled concrete or blast furnace slag or its blends are added to the said pozzolan during or after said processing of the pozzolan.
3. Method according to claim 1 or 3, **characterized in, that** Portland cement, fly ash Class F or C, fine quartz, granite quarry fines, fine fractions of recycled concrete or blast furnace slag or its blends are subjected to pregrinding to achieve fineness with a retention on the sieve of 45 microns which is less than 5%.
4. Method according to claims any of the claims 1 - 3, **characterized in, that** water reducing agents, set time regulators, and strength accelerating admixtures in powder form are added to the said pozzolan during or after said grinding of the pozzolan.
5. Method according to any of claims 1-4, **characterized in, that** replacement of Portland cement by the said pozzolan in concrete or mortar is from 15 to about 70%.
6. Method according to any of the preceding claims, **characterized in, that** the pozzolan is subjected to grinding in said grinding equipment to a fineness of the final product with a retention on a sieve of 30 microns being less than 5%.
7. Method according to any of the preceding claims, **characterized in, that** pozzolan is subjected to grinding in a grinding equipment, which is adopted to open or closed circuit for material being grinded.

Patentansprüche

1. Verfahren zur Herstellung von Zusatzzementmaterialien für den Ersatz von Portlandzement bei der Herstellung von Mörtel und Beton, worin die zusätzlichen Zementmaterialien natürliche Puzzolane in Form von Steinen und Asche umfassen, **dadurch gekennzeichnet, dass** die genannten Pozzuolane in zerkleinertem Zustand in einer hochenergetisch mechanischen Bearbeitung durch Zerkleinerung in einem Zerkleinerungsgerät ausgesetzt wird, wobei die Puzzolan-Teilchen mechanische Impulse erhalten, und in der die Zerkleinerung für eine festgelegte Zeit durchgeführt wird, indem das Endprodukt des Puzzolans nach der Zerkleinerung folgende Partikelgrößenverteilung aufweist:

- ≤ 5 Mikrometer 15-50 %
- ≤ 10 Mikrometer 30-65 %
- ≤ 30 Mikrometer 90-95 %

eine Mischung aus zerkleinerten Puzzolane, Portlandzement und Wasser resultiert zu einer Druckfestigkeit eines 2 Zoll-Würfels aus Mörtel, umfasst 80 % Portlandzement und 20 % natürliches Puzzolan, im Verhältnis zum Standardsand von 1:2,75. Zusätzlich wird Wasser benötigt, um gemäß der amerikanischen Norm ASTM C 109 eine Strömung des Mörtels zu erhalten, die unter Vibration fest verdichtet und bei + 20 °C in versiegeltem Zustand ausgehärtet wird. Nach 28 Tagen beträgt die Unter-Druckfestigkeit der Würfel ≥ 75 % und umfasst einen Durchmesser von 5,08 cm (2 Zoll), als Würfel behandelt, von einem Mörtel, mit Portlandzement und Sand bei einem Verhältnis von 1:2,75 und zusätzlich Wasser auf 48,5 % des Gewichts von Portlandzement umfasst.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** Portland-Zement, Flugasche Klasse F oder C, feiner Quarz, Granit-Steinbruch, Feinanteile aus recyceltem Beton oder Hochofenschlacke, oder deren Mischungen in das Puzzolan, während oder nach der Verarbeitung des Puzzolans zugegeben werden.
3. Verfahren nach Anspruch 1 oder 3, **dadurch gekennzeichnet, dass** Portland-Zement, Flugasche Klasse F oder C, feiner Quarz, Granit-Steinbruch, Feinanteile aus recyceltem Beton oder Hochofenschlacke, oder deren Mischungen werden vorvermahlen, um Sieb-Retention und einem Feinheitsgrad von 45 Mikrometern zu erlangen, was weniger als 5 % beträgt.
4. Verfahren nach einen der vorangegangenen Ansprüchen 1 - 3, **dadurch gekennzeichnet, dass** Wasserreduktionsmittel, eingestellte Zeitregler und

Stärke-Erstarrungsbeschleuniger in Pulverform, während oder nach der Zerkleinerung des Puzzolans in das Puzzolan zugegeben werden.

5. Verfahren nach einem der vorangegangenen Ansprüche 1 - 4, **dadurch gekennzeichnet, dass** der Ersatz von Portlandzement durch das Puzzolan in Beton oder Mörtel zwischen 15 und etwa 70 % beträgt.
6. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Puzzolan in einem Zerkleinerungsgerät zerkleinert wird, auf einen Feingehalt des Endprodukts mit einer Sieb-Retention von 30 Mikrometer, weniger als 5 % beträgt.
7. Verfahren nach einem der vorangegangenen Ansprüche, **dadurch gekennzeichnet, dass** Puzzolan durch ein Zerkleinerungsgerät zerkleinert wird, die einen geöffneten oder geschlossenen Kreislauf annimmt, damit das Material zerkleinert werden kann.

Revendications

1. - Procédé de fabrication d'ajouts cimentaires pour le remplacement du ciment Portland dans la production de mortiers et bétons, suivant lequel les ajouts cimentaires comprennent des pouzzolanes naturelles sous la forme de roches et de cendres, **caractérisé par le fait que** lesdites pouzzolanes à l'état broyé sont soumises à un traitement mécanique hautement énergétique au moyen d'un broyage dans un équipement de broyage, ce par quoi les particules de pouzzolane reçoivent des impulsions mécaniques, et **par le fait que** le broyage est effectué pendant une durée prédéterminée, **par le fait que** le produit final de la pouzzolane après broyage a la distribution granulométrique suivante :

≤ 5 microns 15-50 %,
 ≤ 10 microns 30-65 %,
 ≤ 30 microns 90-95 %

par le fait qu'un mélange de pouzzolanes broyées, de ciment Portland et d'eau conduit à une résistance à la compression d'un cube de mortier de 2 pouces de côté comprenant 80 % de ciment Portland et 20 % de pouzzolane naturelle dans un rapport de 1 : 2,75 à du sable normalisé et en plus de l'eau requise pour obtenir un écoulement du mortier selon la norme américaine ASTM C 109, qui a été convenablement compacté sous vibration et durci à +20°C dans un état scellé, qui, après 28 jours, est ≥ 75 % de la résistance à la compression d'un cube de 5,08 cm (2 pouces) de côté, traité comme le cube précité, d'un mortier comprenant un rap-

port de ciment Portland : sable de 1 : 2,75 et en plus de l'eau correspondant à 48,5 % du poids du ciment Portland.

2. - Procédé selon la revendication 1, **caractérisé par le fait que** du ciment Portland, des cendres volantes de Classe F ou C, du quartz fin, des fines de carrière de granit, des fractions fines de béton recyclé ou de laitier de haut fourneau ou leurs mélanges sont ajoutés à ladite pouzzolane pendant ou après ledit traitement de la pouzzolane.
3. - Procédé selon l'une des revendications 1 ou 3, **caractérisé par le fait que** du ciment Portland, des cendres volantes de Classe F ou C, du quartz fin, des fines de carrière de granit, des fractions fines de béton recyclé ou de laitier de haut fourneau ou leurs mélanges sont soumis à un pré-broyage pour parvenir à une finesse avec une rétention sur le tamis de 45 microns qui est inférieure à 5 %.
4. - Procédé selon l'une quelconque des revendications 1 à 3, **caractérisé par le fait que** des agents réduisant l'eau, des régulateurs du temps de prise et des mélanges accélérateurs de résistance sous forme pulvérulente sont ajoutés à ladite pouzzolane pendant ou après ledit broyage de la pouzzolane.
5. - Procédé selon l'une quelconque des revendications 1 à 4, **caractérisé par le fait que** le remplacement du ciment Portland par ladite pouzzolane dans le béton ou mortier est de 15 à environ 70 %.
6. - Procédé selon l'une quelconque des revendications précédentes, **caractérisé par le fait que** la pouzzolane est soumise à un broyage dans ledit équipement de broyage jusqu'à une finesse du produit final avec une rétention sur un tamis de 30 microns qui est inférieure à 5 %.
7. - Procédé selon l'une quelconque des revendications précédentes, **caractérisé par le fait que** la pouzzolane est soumise à un broyage dans un équipement de broyage, qui est adapté à un circuit ouvert ou fermé pour une matière soumise au broyage.

REFERENCES CITED IN THE DESCRIPTION

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SZABADALMI IGÉNYPONTOK

1. Eljárás beton és habarcs előállításánál Portland cement helyettesítésére alkalmas, cement alapú adalékanyagok előállítására, ahol a cement alapú adalékanyagok közet és hamu állapotú természetes puccolánokat tartalmaznak, *azzal jellemezve*, hogy az említett puccolánokat aprított állapotban nagy energiájú mechanikai eljárás során őrlőberendezésben őröljük, ahol a puccolán szemcsék mechanikai impulzusokat kapnak, továbbá *azzal*, hogy az őrlés meghatározott ideig tart, továbbá *azzal*, hogy az őrlést követően a puccolán végtermékek a következő szemcseméret eloszlása van:

≤ 5 mikron 15 – 50 %,

≤ 10 mikron 30 – 65 %,

≤ 30 mikron 90 – 95 %.

továbbá *azzal*, hogy az őrölt puccolánok, Portland cement és víz keverékével olyan nyomószilárdságot érünk el, amely standard homokhoz 1 : 2,75 arányban kevert, 80 % Portland cementet, 20 % természetes puccolánt tartalmazó elegy, és a habarcsnak az ASTM C 109 szabvány szerinti folyékonyága eléréséhez szükséges mennyiségű víz keverékéből készített, vibrációval megfelelően tömörített, és 20 °C-on légmentesen zárt körülmények között megszilárdult habarcsból előállított, 5,08 cm (2 hüvelyk) oldalélű kocka próbatesten 28 nap elteltével mérve nagyobb, mint az említett kockával azonosan kezelt, standard homokhoz 1 : 2,75 arányban kevert Portland cementből, és a Portland cement tömegének 48,5 %-át kitevő mennyiségű vízből készített habarcsból előállított 5,08 cm (2 hüvelyk) oldalélű kocka nyomószilárdságának 75 %-a.

2. Az 1. igénypont szerinti eljárás, *azzal jellemezve*, hogy a puccolán feldolgozása során vagy azt követően a puccolánhoz Portland cementet, F vagy C típusú szállópernyét, finom kvarcot, gránit terméskőport, visszajáratott betont vagy kohósalak finom frakcióit, vagy ezek keverékét adagoljuk.

3. Az 1. vagy 3. igénypontok szerinti eljárás, *azzal jellemezve*, hogy a Portland cementet, F vagy C típusú szállópernyét, finom kvarcot, gránit terméskőport, visszajáratott betont vagy kohósalak finom frakcióit, vagy ezek keverékét előőrlésnek vetjük alá mindaddig, amíg szemcsefinomságát mérve 45 mikronos szitán a szitamaradék kevesebb, mint 5 %.

4. Az 1. – 3. igénypontok bármelyike szerinti eljárás, *azzal jellemezve*, hogy a puccolán őrlése során vagy azt követően a puccolánhoz por alakú vízmegkötő szereket, kötési idő szabályzókat és szilárdságnövelő adalékokat adagolunk.

5. Az 1. – 4. igénypontok bármelyike szerinti eljárás, *azzal jellemezve*, hogy a Portland cementet az említett puccolánnal betonban vagy habarcsban 15 – 70 %-ig terjedő mértékben helyettesítjük.

6. Az előző igénypontok bármelyike szerinti eljárás, *azzal jellemezve*, hogy a puccolánt az említett őrlőberendezésben őrlésnek vetjük alá mindaddig, amíg szemcsefinomságát mérve a szitamaradék 30 mikronos szitán kevesebb, mint 5 %.

7. Az előző igénypontok bármelyike szerinti eljárás, *azzal jellemezve*, hogy a puccolánt az őrlendő anyagot nyitott vagy zárt körben őrölni képes őrlőberendezésben őröljük.