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# PATENT SPECIFICATION

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## (54) HIGH-ALUMINA REFRACTORY CEMENT AND A PROCESS FOR PRODUCING THE SAME

(71) We, LAFARGE FONDU INTERNATIONAL, a French Company, of 157 Avenue Charles de Gaulle, 92200 Neuilly/Seine, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention concerns a high-alumina refractory cement and a process for producing the same.

One important purpose of the invention is to provide a cement with good refractory properties, but which can be used under conditions similar to those applicable to conventional cements. This new refractory cement also offers the general advantage of allowing simple starting materials to be used.

This invention relates to a high-alumina refractory cement which contains, in addition to ingredients adapted to form a hydraulic bond, one or more ingredients adapted to form a phosphatic chemical bond.

This new high-alumina refractory cement offers an unexpected solution to the problem of deficiencies in performance that can occur at intermediate temperatures with concretes obtained using only aluminous binders. Previously, this problem was solved by mixing the ingredient designed to produce a hydraulic bond with a filler; this produces ceramic bonds at intermediate temperatures, at which hydraulic bonds deteriorate. The ceramic bonds result from sintering of the filler with the other ingredients, at intermediate temperatures. This premature ceramization ensures the strength of the concrete when the hydraulic bond deteriorates at these intermediate temperatures.

The high alumina refractory cement according to the invention offers high performances at intermediate temperatures.

According to this invention we provide a high alumina refractory cement comprising alumina, an aluminous clinker containing calcium aluminate (CA) and having a Blaine specific surface-area of 3000 to 5000 cm<sup>2</sup>/g for forming a hydraulic bond and 0.5 to 5% by weight in relation to the weight of the whole composition of at least one water soluble alkali metal phosphate expressed as P<sub>2</sub>O<sub>5</sub>, for forming a phosphatic chemical bond.

This phosphatic chemical bond offers an unexpected cheap, simple solution to the problem of the zone of weakness found at intermediate temperatures in concretes made from conventional high-alumina cements.

The secondary ingredients consists of compounds normally used in this field, for example carboxylic acids and their salts, such as monosodium citrate.

In one embodiment of the invention, the ingredients designed to promote the phosphatic chemical bond consist of 0.5 to 5% by weight (expressed as phosphorus pentoxide P<sub>2</sub>O<sub>5</sub>), in relation to the weight of high-alumina cement, of at least one water soluble alkali metal phosphate. The content of water soluble alkali metal phosphate, expressed as P<sub>2</sub>O<sub>5</sub>, is preferably between 1.5 and 5%. Moreover, in another embodiment of the invention, the water soluble alkali metal phosphate is sodium hexametaphosphate, tripolyphosphate or pentapolyphosphate. It is preferably sodium hexametaphosphate.

In one embodiment of the invention, the ingredients adapted to form a hydraulic bond consist of a ground clinker obtained from aluminous materials on the one hand, and calcarious or dolomitic materials on the other.

According to another feature of the invention, the ingredients adapted to form a

hydraulic bond essentially comprise a ground clinker containing at least 35% by weight of monocalcic aluminate (CA) in relation to the weight of clinker.

According to yet another feature of the invention, aluminous material (calculated as  $\text{Al}_2\text{O}_3$ ) forms 65 to 95% of the weight of high-alumina refractory cement, and comprises calcined alumina and monocalcic aluminate present in the ground clinker.

According to one feature of the invention, the high-alumina refractory cement has a minimum specific surface area measured by gas flow (Blaine) of  $4300 \text{ cm}^2/\text{g}$ .

This invention also relates to a process for manufacturing this new high-alumina refractory cement, wherein the cement clinker and calcined alumina are ground together, and the alkali metal phosphate and any other ingredients are mixed in after grinding.

The invention also relates to a process for manufacturing this new high-alumina refractory cement, by mixing together a ground aluminous clinker, containing calcium aluminate (CA) and having a Blaine specific surface of 3000 to  $5000 \text{ cm}^2/\text{gm}$ , previously ground alumina, the alkali metal phosphate and any other ingredients.

The clinker is ground to ensure a Blaine specific surface area of 3000 to  $5000 \text{ cm}^2/\text{g}$ , and preferably 3600 to  $4000 \text{ cm}^2/\text{g}$ .

According to one feature of the invention, the high-alumina refractory cement is obtained by mixing:

- 20 to 90% by weight of ground clinker containing at least 35% by weight CA
- 2 to 79% by weight of calcined alumina with a specific-area of between 0.3 and  $50 \text{ m}^2/\text{g}$ , measured by gas adsorption, (BET)
- 0.5 to 5% by weight of water soluble alkali metal phosphate, expressed as  $\text{P}_2\text{O}_5$ .

The clinker used is obtained by the fusing or sintering at between  $1400$  and  $1700^\circ$  of aluminous materials and calcitic and/or dolomitic materials. Limestone, magnesian limestone, dolomitic limestone, each possibly decarbonated, and similar substances can be used for this purpose.

The aluminous material used to make the clinker is selected from bauxites, laterites or other aluminous ores and particularly red bauxites, white bauxites with low iron oxide content, or hydrated or calcined aluminas. The BET specific surface-area of the calcined alumina added to the clinker is preferably between 0.5 and  $10 \text{ m}^2/\text{g}$ .

The invention is illustrated by, without being confined to, the following examples, which will reveal other benefits thereof. All percentages are by weight.

#### EXAMPLES 1 to 10.

520 kg high-alumina cement clinker containing approximately 70% CA (Blaine surface area  $3700 \text{ cm}^2/\text{g}$ ) was placed in a Lodige batch mixer, together with 480 kg of a commercial alumina reground to  $6000 \text{ cm}^2/\text{g}$  and containing at least 90%  $\alpha$ -alumina; additives, namely sodium hexametaphosphate and monosodium citrate, were also placed in the mixer.

Table I below shows the quantities of sodium hexametaphosphate (also expressed as  $\text{P}_2\text{O}_5$ ) and monosodium citrate added.

The effect of the soluble alkali metal phosphate content on the hydraulic and refractory properties of fireclay concretes made from this cement is also shown in Table I.

This table shows that performances in flexion (F) and in compression (C) after heating and cooling again to room temperature are little affected by possible variations in additives.

TABLE I

| Phosphate containing complex, % |                               | W/C*** | Fireclay concrete 42-44% (0-5 mm) a) — content 500 kg/m <sup>3</sup> b) — |     |     |     |  |                  |      |        |   |     |
|---------------------------------|-------------------------------|--------|---------------------------------------------------------------------------|-----|-----|-----|--|------------------|------|--------|---|-----|
| Example                         | HMP*                          |        | Storage at 20°C                                                           |     |     |     |  | After heating to |      |        |   |     |
|                                 | CM**                          |        | 6h                                                                        |     | 24h |     |  | 800°C            |      | 1100°C |   |     |
|                                 | P <sub>2</sub> O <sub>5</sub> |        | F                                                                         | C   | F   | C   |  | F                | C    | F      | C |     |
| 1                               | 3                             | 0,3    | 19                                                                        | 125 | 49  | 500 |  | 105              | 1005 | 85     |   | 600 |
| 2                               | 1,86                          |        | 19                                                                        | 130 | 55  | 530 |  | 100              | 955  | 84     |   | 630 |
| 3                               | 3                             | 0,5    | 27                                                                        | 170 | 45  | 550 |  | 95               | 975  | 88     |   | 610 |
| 4                               | 3,5                           | 0,3    | 21                                                                        | 155 | 43  | 495 |  | 98               | 990  | 90     |   | 595 |
| 5                               | 2,17                          |        | 20                                                                        | 110 | 42  | 480 |  | 110              | 990  | 84     |   | 585 |
| 6                               | 3,5                           | 0,5    | 35                                                                        | 160 | 46  | 505 |  | 87               | 965  | 82     |   | 600 |
| 7                               | 4                             | 0,3    | 25                                                                        | 160 | 52  | 570 |  | 110              | 965  | 90     |   | 600 |
| 8                               | 2,48                          |        | 24                                                                        | 210 | 55  | 585 |  | 94               | 1010 | 86     |   | 585 |
| 9                               | 4                             | 9,5    | 36                                                                        | 190 | 50  | 495 |  | 84               | 935  | 80     |   | 615 |
| 10                              | 8                             | 0,5    | 8                                                                         | 30  | 38  | 285 |  | 128              | 1315 | 99     |   | 615 |

\* HMP = Sodium hexametaphosphate      \*\* CM = Monosodium citrate      \*\*\* W/C = water/cement weight ratio

a) with 42 to 44% Al<sub>2</sub>O<sub>3</sub> and a grain-size of 0-5 mm (Fuller curve)  
b) 500 kg cement per m<sup>3</sup> of concrete

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Chemical analysis of the high-alumina fire resistant cement from examples 8 and 9 revealed the following composition:

|                                         |       |
|-----------------------------------------|-------|
| * SiO <sub>2</sub>                      | 0,15  |
| * Al <sub>2</sub> O <sub>3</sub>        | 79,80 |
| * CaO                                   | 14,90 |
| * Al <sub>2</sub> O <sub>3</sub> (free) | 47,45 |
| * Fe <sub>2</sub> O <sub>3</sub>        | 0,05  |
| * K <sub>2</sub> O                      | 0,01  |
| * Na <sub>2</sub> O                     | 1,50  |
| * SO <sub>3</sub>                       | 0,10  |
| * P <sub>2</sub> O <sub>5</sub>         | 2,48  |
| * Fire losses                           | 0,7   |

Grain-sizes, measured with a laser technique, were as follows:

| Diameter (microns) | Percentage |
|--------------------|------------|
| < 2                | 14         |
| < 4                | 35         |
| < 8                | 60         |
| < 16               | 74         |
| < 32               | 85         |
| < 64               | 97         |

Finally, the following physical properties were measured:

|                                                     |      |
|-----------------------------------------------------|------|
| * Density                                           | 3,29 |
| * Blaine specific surface-area (cm <sup>2</sup> /g) | 4370 |
| * BET surface-area (m <sup>2</sup> /g)              | 1    |
| * Amount of water required for pure paste           | 24%  |

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This high-alumina refractory cement does not necessarily need to be prepared in a Lodge batch mixer; mixing can be done in other types of mixers, operating continuously or intermittently.

#### EXAMPLE 11.

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520 kg of aluminous clinker and 480 kg of alumina were placed in a mill, and ground together until a Blaine specific surface-area of  $4900 \text{ g/cm}^2$  was obtained. 40 kg of sodium hexametaphosphate and 3 kg of monosodium citrate were then added and mixed in a mixer, using the same procedure as in Examples 1 to 10.

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Fireclay concretes made from the cement from Examples 7 and 8 had approximately the same properties shown in Table I as those obtained during tests for these examples. The advantage of grinding materials together is to avoid the need for a special alumina grinder in the installation. This saves equipment costs.

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#### EXAMPLES 12 and 13.

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High-alumina refractory cements were prepared from 930 kg of clinker containing 70% of CA, 70 kg of alumina and 40 kg of HMP (Example 12) or 50 kg of HMP (Example 13), following the procedure described for Examples 1 to 10. The resulting cements contained 63% CA and 2.48% (Example 12) and 3.1% (Example 13) by weight of  $\text{P}_2\text{O}_5$ , that is to say 4% and 5% of HMP respectively. Fireclay concretes made from these cements had the properties shown in Table II below.

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TABLE II

Fireclay (42-44%  $Al_2O_3$ ) concrete - 500 kg/m<sup>3</sup> cement content

| Example | % $P_2O_5$ | W/C  | Storage at 20°C |     |    | After heating |     |      |       |      |     |
|---------|------------|------|-----------------|-----|----|---------------|-----|------|-------|------|-----|
|         |            |      | 6h              |     |    | 24h           |     |      | 110°C |      |     |
|         |            |      |                 |     |    |               |     |      |       |      |     |
|         |            |      | F               | C   |    | F             | C   |      | F     | C    |     |
| 12      | 2,48       | 0,38 | 47              | 435 | 87 | 740           | 116 | 1035 | 108   | 1155 | 95  |
| 13      | 3,10       | 0,38 | 42              | 390 | 79 | 625           | 117 | 1030 | 79    | 965  | 705 |

TABLE III

Fireclay concrete \*

| Example | W/C  | Storage at 20°C |    |    | After heating |    |     |       |     |      |
|---------|------|-----------------|----|----|---------------|----|-----|-------|-----|------|
|         |      | 6h              |    |    | 24h           |    |     | 800°C |     |      |
|         |      |                 |    |    |               |    |     |       |     |      |
|         |      | F               | C  |    | F             | C  |     | F     | C   |      |
| 14      | 0,36 | 18              | 95 | 29 | 105           | 61 | 630 | 45    | 310 | 156  |
|         |      |                 |    |    |               |    |     |       |     | 1070 |

\* As defined in Table I.

TABLE IV

Fireclay (42-44%  $\text{Al}_2\text{O}_3$ ) concrete - 500 kg/m<sup>3</sup> cement content

Effect of polymer phosphates

|    | Phosphates added<br>Type %           | W/C  | Storage at 20°C |     |     |     | After heating |      |        |     |
|----|--------------------------------------|------|-----------------|-----|-----|-----|---------------|------|--------|-----|
|    |                                      |      | 6h              |     | 24h |     | 800°C         |      | 1100°C |     |
|    |                                      |      | F               | C   | F   | C   | F             | C    | F      | C   |
|    |                                      |      |                 |     |     |     |               |      |        |     |
| 15 | Sodium tri-<br>polyphosphate<br>2%   | 0,39 | 30              | 210 | 62  | 395 | 59            | 640  | 52     | 440 |
| 16 | Sodium tri-<br>polyphosphate<br>4%   | 0,39 | 0               | 25  | 50  | 300 | 104           | 950  | 54     | 480 |
| 17 | Sodium penta-<br>polyphosphate<br>2% | 0,39 | 65              | 590 | 87  | 675 | 110           | 1060 | 73     | 560 |
| 18 | Sodium penta-<br>polyphosphate<br>4% | 0,39 | 72              | 625 | 92  | 780 | 120           | 1100 | 84     | 590 |

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**EXAMPLE 14.**

A high-alumina refractory cement was made, using the methods described in Examples 1 to 10, from 550 kg of clinker containing approximately 17% of dolomitic materials (expressed in the form of spinel:  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ) and 35% of CA, 450 kg of alumina reground to 6000  $\text{cm}^2/\text{g}$ , 40 kg of HMP and 3 kg of monosodium citrate.

Fireclay concretes made from this cement had the properties shown in Table III above.

**EXAMPLES 15 to 18.**

High-alumina refractory cement was made using the methods described in Example 12, except that different proportions of sodium tripolyphosphate (Examples 15 and 16) and sodium pentapolyphosphate (Examples 17 and 18) were used.

Table IV above shows the properties of fireclay concretes made from these cements.

**EXAMPLES 19 and 20.**

A high-alumina refractory cement was prepared by the methods described in Examples 7 and 8, except that a reground calcined alumina with a Blaine specific surface-area of 22000  $\text{cm}^2/\text{g}$  ( $\text{BET} = 10 \text{ m}^2/\text{g}$ ) was used.

Fireclay concretes (42—44%  $\text{Al}_2\text{O}_3$ ) and tabular alumina concretes (0—7 mm); containing 500 kg of cement per cubic metre of concrete poured, had the properties shown in Table V below.

**TABLE V**

| Aggregate         | W/C  | Concrete containing 500 $\text{kg}/\text{m}^3$ cement |     |               |      |        |      |
|-------------------|------|-------------------------------------------------------|-----|---------------|------|--------|------|
|                   |      | 20°C                                                  |     | After heating |      |        |      |
|                   |      | 4h                                                    |     | 1100°C        |      | 1400°C |      |
|                   |      | F                                                     | C   | F             | C    | F      | C    |
| * Fireclay        | 0,34 | 25                                                    | 160 | 123           | 870  | 225    | 1320 |
| * Tabular alumina | 0,36 | 32                                                    | 180 | 162           | 1060 | 236    | 1190 |

This cement in fact also contains alumina, which acts as a filler and also produces a ceramic bond.

Cement prepared in this way involves three bonding principles: chemical, hydraulic and ceramic; this is what distinguishes it from other cements. It can be used as a basis for refractory concretes which have to withstand particularly severe conditions.

**EXAMPLE 21.**

High-alumina refractory cement was made by the methods described in Examples 7 and 8, using 210 kg of aluminous clinker containing approximately 70% of CA and 790 kg of alumina reground to 6000  $\text{cm}^2/\text{g}$ .

Table VI below shows the properties of fireclay concretes made from this cement. Because of the low CA content of the cement (~16%), concrete made from it shows better resistance to corrosion from certain molten metals.

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TABLE VI

| Example | W/C  | Fireclay (42-44% $Al_2O_3$ ) concrete |     |               |     |        |     |
|---------|------|---------------------------------------|-----|---------------|-----|--------|-----|
|         |      | 20°C                                  |     | After heating |     |        |     |
|         |      | 24h                                   |     | 110°C         |     | 1100°C |     |
|         |      | F                                     | C   | F             | C   | F      | C   |
| 21      | 0.34 | 20                                    | 165 | 55            | 470 | 40     | 290 |

## EXAMPLE 22.

High-alumina refractory cement was made by the method described in Examples 7 and 8, using 520 kg of aluminous clinker containing approximately 70% of CA, and 480 of under-calcined alumina ( $BET=47 \text{ m}^2/\text{g}$ ) reground to approximately 22000  $\text{cm}^2/\text{g}$ , 40 kg of HMP and 3 kg of MC.

Table VII below shows the properties of fireclay concretes made from these cements.

TABLE VII

| Example | W/C  | Fireclay (42-44% $Al_2O_3$ ) concrete |    |     |     |               |     |        |     |
|---------|------|---------------------------------------|----|-----|-----|---------------|-----|--------|-----|
|         |      | Storage at 20°C                       |    |     |     | After heating |     |        |     |
|         |      | 6h                                    |    | 24h |     | 800°C         |     | 1100°C |     |
|         |      | F                                     | C  | F   | C   | F             | C   | F      | C   |
| 22      | 0.38 | 12                                    | 85 | 20  | 180 | 63            | 658 | 64     | 450 |

## WHAT WE CLAIM IS:—

1. A high alumina refractory cement comprising alumina, an aluminous clinker containing calcium aluminate (CA) and having a Blaine specific surface-area of 3000 to 5000  $\text{cm}^2/\text{g}$  for forming a hydraulic bond and 0.5 to 5% by weight in relation to the weight of the whole composition of at least one water soluble alkali metal phosphate expressed as  $P_2O_5$ , for forming a phosphatic chemical bond.

2. A cement as defined in claim 1, in which the content of water soluble alkali metal phosphate, expressed as  $P_2O_5$ , is between 1.5 and 5%.

3. A cement as defined in claim 1, in which the water alkali metal phosphate is a sodium polyphosphate.

4. A cement as defined in claim 3, in which the water soluble alkali metal phosphate is sodium hexametaphosphate.

5. A cement as defined in claim 1 further containing calcitic or dolomitic materials for forming a hydraulic bond.

6. A cement as defined in claim 1, in which the aluminous clinker contains at least 35% by weight of monocalcium aluminate (CA).

7. A cement as defined in claim 1 comprising from 65 to 95% of aluminous material calculated as  $Al_2O_3$ , said aluminous material comprising calcined alumina and calcium aluminates present in the ground clinker.

8. A cement as defined in claim 1, which has a minimum Blaine specific surface area of 4300  $\text{g}/\text{cm}^2$ .

9. A cement as defined in claim 1, further containing carboxylic acids or their salts.



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TABLE VI

| Example | W/C  | Fireclay (42-44% $\text{Al}_2\text{O}_3$ ) concrete |     |               |     |        |     |
|---------|------|-----------------------------------------------------|-----|---------------|-----|--------|-----|
|         |      | 20°C                                                |     | After heating |     |        |     |
|         |      | 24h                                                 |     | 110°C         |     | 1100°C |     |
|         |      | F                                                   | C   | F             | C   | F      | C   |
| 21      | 0.34 | 20                                                  | 165 | 55            | 470 | 40     | 290 |

## EXAMPLE 22.

High-alumina refractory cement was made by the method described in Examples 7 and 8, using 520 kg of aluminous clinker containing approximately 70% of CA, and 480 of under-calcined alumina (BET=47 m<sup>2</sup>/g) reground to approximately 22000 cm<sup>2</sup>/g, 40 kg of HMP and 3 kg of MC.

Table VII below shows the properties of fireclay concretes made from these cements.

TABLE VII

| Example | W/C  | Fireclay (42-44% $\text{Al}_2\text{O}_3$ ) concrete |    |     |     |               |     |        |     |
|---------|------|-----------------------------------------------------|----|-----|-----|---------------|-----|--------|-----|
|         |      | Storage at 20°C                                     |    |     |     | After heating |     |        |     |
|         |      | 6h                                                  |    | 24h |     | 800°C         |     | 1100°C |     |
|         |      | F                                                   | C  | F   | C   | F             | C   | F      | C   |
| 22      | 0.38 | 12                                                  | 85 | 20  | 180 | 63            | 658 | 64     | 450 |

## WHAT WE CLAIM IS:—

1. A high alumina refractory cement comprising alumina, an aluminous clinker containing calcium aluminate (CA) and having a Blaine specific surface-area of 3000 to 5000 cm<sup>2</sup>/g for forming a hydraulic bond and 0.5 to 5% by weight in relation to the weight of the whole composition of at least one water soluble alkali metal phosphate expressed as P<sub>2</sub>O<sub>5</sub>, for forming a phosphatic chemical bond.

2. A cement as defined in claim 1, in which the content of water soluble alkali metal phosphate, expressed as P<sub>2</sub>O<sub>5</sub>, is between 1.5 and 5%.

3. A cement as defined in claim 1, in which the water alkali metal phosphate is a sodium polyphosphate.

4. A cement as defined in claim 3, in which the water soluble alkali metal phosphate is sodium hexametaphosphate.

5. A cement as defined in claim 1 further containing calcitic or dolomitic materials for forming a hydraulic bond.

6. A cement as defined in claim 1, in which the aluminous clinker contains at least 35% by weight of monocalcium aluminate (CA).

7. A cement as defined in claim 1 comprising from 65 to 95% of aluminous material calculated as Al<sub>2</sub>O<sub>3</sub>, said aluminous material comprising calcined alumina and calcium aluminates present in the ground clinker.

8. A cement as defined in claim 1, which has a minimum Blaine specific surface area of 4300 g/cm<sup>2</sup>.

9. A cement as defined in claim 1, further containing carboxylic acids or their salts.

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10. A cement as defined in claim 9 further containing monosodium citrate.

11. A process of making a high-alumina refractory cement according to claim 1 wherein the cement clinker and calcined alumina are ground together, and the alkali metal phosphate and any other ingredients are mixed in after grinding.

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12. A process of making high alumina fire resistant cement according to claim 1 by mixing together a ground aluminous clinker containing calcium aluminate (CA) and having a Blaine specific surface of 3000 to 5000 cm<sup>2</sup>/gm, previously ground alumina, the alkali metal phosphate and any other ingredients.

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13. A process as defined in claim 12, in which the aluminous clinker is ground to a Blaine specific surface area of 3600 to 4000 cm<sup>2</sup>/g.

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14. A process as defined in claim 11 or claim 12 in which 20 to 90% of clinker containing at least 35% of monocalcium aluminate, 2 to 79% of calcined alumina with a BET specific area of between 0.3 and 50 m<sup>2</sup>/g, and 0.5 to 5% of water soluble alkali metal phosphate expressed as a percentage of the weight of P<sub>2</sub>O<sub>5</sub>, are mixed together.

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15. A process as defined in claim 11 or claim 12 in which the clinker is obtained by fusing or sintering aluminous materials and calcitic and/or dolomitic materials at between 1400 and 1700°C.

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16. A process as defined in claim 15 in which the aluminous material is selected from red bauxites, white bauxites with low iron oxide content and hydrated or calcined aluminas.

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17. A process as defined in claim 16 in which the BAT specific surface area of the added calcined alumina is between 0.5 and 10 m<sup>2</sup>/g.

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