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1 European Patent Number

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3 European Patent Bulletin Date:

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Signed this 22 day of September 1990

A. Pajaczkowski

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The present invention relates to a process for sealing soils and materials such as mortars, binders or concretes and the like, which have already been put into use and which are found to contain a porosity such that they are not impervious.

There are already known processes for sealing such materials, which consist in injecting into the said materials at ground level a silica liquor such as that described in Luxembourgian Patent No. 84,196, in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio may be, for example, lower than 2, preferably between 1.5 and 1.8.

In the case where the injection is performed into soils or materials which are poor in lime or in calcium, an injection of calcium salts consisting, for example, of a water-soluble calcium chloride must be performed in practice before the injection of the silica liquor.

It has also been proposed to mix these soluble calcium salts with the silica liquor, but the reaction between these soluble calcium salts and the silica liquor generally takes place in less than 10 minutes, with the result that mixing before the injection is hardly practicable, and in practice this makes it necessary to work in two stages and complicates the use of the process, while creating an uncertainty concerning the quality of the sealing obtained, because of the fact that the sealing runs the risk of being imperfect if the silica liquor and the calcium salt do not come into contact at each point of the soil to be sealed.

Document NL-A-291,298 describes a mixture of soluble glass and of finely ground calcium oxide, which does not exhibit the characteristics of the composition according to the invention and which does not enable the process according to the invention to be applied because of the fact that this document does not indicate the SiO_2/Na_2 ratio or the solubility in water of the compound containing calcium, which are the characteristics of the invention.

The present invention relates to a process and to materials for putting it into practice, which make it

possible to seal a soil or other porous materials by virtue of the injection of a single product which sets in more than one hour after the injection, enabling a reliable and economic sealing to be obtained.

5 The subject of the present invention is a process for sealing a soil or a material such as a mortar, a binder or a concrete, with the aid of the injection of a silica liquor, characterized in that before the injection
10 a finely ground powder of a compound containing calcium which has a solubility in water of between approximately 0.01 per litre and approximately 2 g per litre is mixed with the silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is between 1 and 1.3.

15 In accordance with the invention, the powdered compound containing calcium which may be employed is a finely divided powder of calcium carbonate (CaCO_3) consisting, for example, of a marble, a limestone or finely ground calcite. Lime ($\text{Ca}(\text{OH})_2$) powder may also be
20 employed. The particle size of the powder containing calcium is a function of the size of the interstices which exist in the material which is to be made impervious.

25 In accordance with the invention, the powdered compound containing calcium can be obtained in the desired particle size by grinding marble, calcite or chalk. In the case where this grinding does not enable particles smaller than a given size to be obtained, this powder is suspended in water with the addition thereto of
30 a deflocculating agent consisting, for example, of a compound of naphthalene type, and by allowing this suspension to settle in a settling vessel for a suitable time, the supernatant part forming the suspension of particles of calcium-based compounds which can be employed in accordance with the invention.

35 The deflocculation may also be obtained by a physical route: ultrasonics, vibration and the like.

 In accordance with the invention, a silica liquor is employed in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is between approximately 1 and 1.3, the SiO_2 content being preferably

between 10 and 20% and the Na_2O content being preferably between 10 and 15%.

5 When the mixture of silica liquor and of powdered compounds containing calcium is injected into the soil the Ca^{++} ions are progressively released by hydrolysis and react with the silica liquor to form hydrated lime silicates. Owing to a shift in the chemical equilibrium, the collection of these Ca^{++} ions permits a new release of new ions by hydrolysis, and this enables an abrupt and fast reaction to be replaced by a progressive reaction which is spread over several hours and which enables the process according to the invention to be put into use.

10 Another subject of the present invention is a composition intended to be injected into soils or materials such as mortars, binders or concretes with the objective of sealing them, this composition being characterized in that it contains as a combination a silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is between 1 and 1.3 and a finely divided powder of a material containing calcium, whose solubility in water is approximately between 0.01 and 2 g per litre.

In accordance with the invention, setting times which permit an easy injection of the mixture and which are generally between 1 hour and 12 hours can be obtained.

25 The setting time can be adjusted by modifying the solubility of the Ca-based compound which is chosen, it being understood that the setting time varies inversely with the solubility of the calcium-based compound.

30 The setting time is also a function of the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio and it varies in the same direction as this ratio. However, if the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is higher than approximately 1.3, poor silica crystallization is produced and if this ratio is lower than 1 the mechanical strength is insufficient and the result which is sought is not obtained.

35 In order to make the invention better understood, a number of embodiments thereof, which are given in the examples below, will be described by way of illustration and without any limitation being implied:

EXAMPLE I

In order to seal a fine sand by injection, a suspension is first of all prepared of CaCO_3 powder consisting of marble, limestone or finely ground calcite. This powder has a solubility in water of approximately 0.01 g per litre.

To do this, from 200 to 500 kg of calcium carbonate powder which has an initial particle size smaller than 50 microns is mixed with 1000 litres of water.

A deflocculating agent consisting, for example, of a compound of naphthalene type is added to the suspension.

Settling is allowed to take place for three quarters of an hour to one hour and after this settling time the powder which is in suspension in the supernatant liquid is smaller than 10 microns in particle size.

A silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is approximately equal to 1 is then prepared in a conventional manner.

The calcium carbonate suspension is mixed with the silica liquor in a proportion of one volume of calcium carbonate suspension to 0.5 to 1.5 volumes of silica liquor.

A composition which remains very fluid is obtained.

This composition is injected under pressure using a conventional technique into the fine sand, which becomes impervious and strong after a few hours.

The setting time is approximately 1 to 3 hours.

Analysis enables lime silicates to be detected in the pores of the sand.

EXAMPLE II

The procedure followed is as indicated in Example I, but with a silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is 1.3.

In this case the setting time is between 2 and 10 hours.

EXAMPLE III

5 The procedure followed is as indicated in Example I, but using lime ($\text{Ca}(\text{OH}_2)$) powder instead of marble, limestone or calcite, in a proportion of approximately 100 to 300 kg per 1000 litres of water.

In this case the setting time is between 30 minutes and 2 hours.

10 It is obvious that the method of application which has been described above is not limiting in any manner and that it would be capable of being subjected to any desirable modification without departing thereby from the scope of the invention.

In particular, various types of silica liquor may be employed.

15 Similarly, the agents containing calcium may be other than calcium carbonate powders, on condition that these compounds release calcium ions progressively by hydrolysis in the silica liquor so as to obtain a progressive reaction.



CLAIMS

1. Process for sealing a soil or a material such as a mortar, a binder or a concrete, with the aid of the injection of silica liquor, characterized in that before the injection a finely ground powder of a compound containing calcium which has a solubility in water of approximately between 0.01 and 2 g per litre is mixed with the silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is between 1 and 1.3.
2. Process according to Claim 1, characterized in that the compound containing calcium is a calcium carbonate (CaCO_3) powder consisting, for example, of a marble, a limestone or calcite.
3. Process according to Claim 1, characterized in that the compound containing calcium is lime ($\text{Ca}(\text{OH})_2$) powder.
4. Process according to any one of the preceding claims, characterized in that to obtain the powder of the compound containing calcium and intended to be mixed with the silica liquor, the ground particles are suspended in water while a deflocculating agent such as a compound of naphthalene type is added thereto and that this suspension is allowed to settle in a settling vessel for a sufficient time for the supernatant part to contain only particles of a size smaller than the desired maximum size.
5. Composition intended to be injected into soils or materials such as mortars, binders, or concretes with the objective of sealing them, characterized in that it contains as a combination a silica liquor in which the $\text{SiO}_2/\text{Na}_2\text{O}$ ratio is between 1 and 1.3 and a finely ground powder of a material containing calcium, whose solubility in water is approximately between 0.01 and 2 g per litre.