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(54) Title: RADIATION-IMPERMEABLE AND RADON GAS-TIGHT FIBER-REINFORCED CONCRETES

(57) Abstract: The present invention relates to radiation-attenuating and radon gas-tight fiber-reinforced concretes having improved physico-mechanical properties and containing at least one aggregate and at least one superplasticizer, and to methods for producing the same. The present invention also relates to a fiber-reinforced concrete containing at least one superplasticizer along with an aggregate composition comprising at least one aggregate selected from the group consisting of barite, colemanite, magnetite, and mixtures thereof.



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## **RADIATION-IMPERMEABLE AND RADON GAS-TIGHT FIBER-REINFORCED CONCRETES**

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### **TECHNICAL FIELD**

The present invention relates to radiation-attenuating and radon gas-tight fiber-reinforced  
concrettes having improved physico-mechanical properties and containing at least one aggregate  
and at least one superplasticizer, and to methods for producing the same.

### **PRIOR ART**

Radiation is a form of energy emitted from both natural and man-made sources. Radiation  
emitting sources include cosmic rays, radon gas, radioactive materials in the earth's crust, and the  
field of nuclear energy, as well as the devices employed in medicinal, agricultural, medical,  
industrial, and military applications. Most of the radiation dose we receive from natural sources  
is due to radon gas. Radon gas is a radioactive gas with a tendency to accumulate in closed  
spaces and is the second most common cause of lung cancer. It tends to accumulate by  
infiltrating from the soil and leaking through cracks and junctions in buildings. The building  
industry and the manufacture of innovative materials are the most prominent fields for measures  
to be taken for protection against exposure to radiation in buildings.

Concrettes with radiation attenuation properties in literature are manufactured at high densities by  
using natural minerals such as heavyweight aggregate, barite, hematite, limonite, and magnetite.  
In addition to these minerals, steel, iron, and lead scraps and wastes were also tested as artificial  
aggregates. Physico-mechanical properties of the concrete vary depending on the selected natural  
or man-made aggregate, and the effect of such variation on radiation attenuation/shielding  
character of the concrete has been studied. In literature, radiation attenuation properties of  
concrettes have been evaluated by calculating half-value thickness (HVT), tenth-value thickness  
(TVT), and Linear Attenuation Coefficient, and the effect of material thickness on radiation  
permeability is studied. R. K. Chauhan et al., "Development and Design Mix of Radiation  
Shielding Concrete for gamma-ray Shielding" (2017), found similar attenuation factors for

values after 8 cm in samples with a thickness of 2-10 cm among samples with a size of 10 cm x 10 cm x 10 cm.

5 In concretes in the technical field, cracks, as well as collapse, delamination, and contraction are observed due to the high water absorption. The aggregates in use exhibit a dispersion problem in the cement matrix, leading to a failure to ensure homogenization, and thus a failure to achieve physicochemical compatibility between concrete components. On the other hand, there are a very low number of studies with an aim to achieve a concrete having radon gas impermeability.

10 The patent application CN103224369 discloses an anti-radiation concrete with a structure containing polypropylene, steel or lead fiber and cinder aggregate, and a method for producing the said concrete. The anti-radiation performance of this concrete is improved thanks to its contents of silica ash and fly ash minerals. Similarly, the patent application CN101921093 also discloses a concrete structure containing silica ash and fly ash. The aim of this disclosure was  
15 also to improve radiation impermeability of concrete by using various aggregates.

US6630683B2 discloses a concrete improved in terms of anti-radiation by using two types of aggregates one of which is metallic.

20 There is, however, still a need in the art for the use of low-level radioactive materials in building construction and the development of radiation shielding, innovative concrete products.

## **OBJECT OF THE INVENTION**

25 The present invention relates to a fiber-reinforced concrete product that meets the above-mentioned needs, eliminates the disadvantages of the prior art, and provides a number of additional advantages.

The primary object of the present invention is to provide a fiber-reinforced concrete product  
30 having radon gas-tightness properties in addition to its radiation attenuating (shielding) properties. Such concretes with radon gas-tightness properties will not only provide protection from radiation sources (nuclear energy, medical, medicinal, military, industrial), but also against naturally occurring radiation.

An object of the present invention is to provide a fiber-reinforced concrete product with a low level of thermal conductivity and improved fire resistance and non-combustibility properties.

5 Another object of the present invention is to provide a fiber-reinforced concrete product with a minimal amount of crack formation, delamination, and collapse.

Another object of the present invention is to provide a fiber-reinforced concrete product which is thinner and lighter than the anti-radiation concretes in the technical field.

10 Another object of the present invention is to provide a fiber-reinforced concrete product as a construction material alternative to the lead sheets, which were developed especially for use in hospitals.

#### **DETAILED DESCRIPTION OF THE INVENTION**

15 The present invention relates to radiation-attenuating and radon gas-tight fiber-reinforced concrete having improved physico-mechanical properties and containing at least one aggregate and at least one superplasticizer.

20 In one aspect of the present invention, the fiber in the fiber-reinforced concrete according to the present application is selected from the group comprising zirconium materials, carbon, fiberglass, polypropylene, nylon, polyethylene, polyester, and steel, and mixtures thereof, and is preferably fiberglass.

25 In one aspect of the present invention, the fiber-reinforced concrete according to the present application contains at least one cement material. Here, such cement materials are selected from the group comprising white Portland cement, gray Portland cement, and mixtures thereof.

30 In another aspect of the present invention, the fiber-reinforced concrete according to the present application contains at least one aggregate. Here, such aggregate is selected among hematite, goethite, barite, limonite, serpentine, colemanite, and magnetite.

The present invention relates to a fiber-reinforced concrete containing at least one superplasticizer along with an aggregate composition comprising at least one aggregate selected from the group consisting of barite, colemanite, magnetite, and mixtures thereof.

- 5 On the other hand, the present invention relates to a fiber-reinforced concrete at least one superplasticizer, and an aggregate composition comprising a mixture of barite, colemanite, and magnetite.

- 10 In one aspect of the present invention, a fiber-reinforced concrete according to the present application contains an aggregate composition in the range of 40% to 70, and preferably of 50% to 60%.

- 15 The aggregate composition according to the present application contains 1-60% barite, 1-60% colemanite and 1-60% magnetite relative to the aggregate composition. Owing to an aggregate composition with such rates of components, the physicochemical compatibility between cement and aggregates in the fiber-reinforced concrete appears to be optimized. In this way, the capacity of concrete to absorb water was decreased, and crack formation, collapse and delamination were minimized.

- 20 In one aspect of the present invention, the particle size of aggregates according to the present application varies between 1-3000  $\mu\text{m}$ , and preferably between 1-1000  $\mu\text{m}$ .

- 25 Another aspect of the present invention relates to a fiber-reinforced concrete containing at least one superplasticizer along with an aggregate composition comprising colemanite with a d50 of 1-3000  $\mu\text{m}$ , barite with a d50 of 300-750  $\mu\text{m}$ , and magnetite with a d50 of 1-400  $\mu\text{m}$ . Here, the particle size is measured by known methods from the prior art, and preferably with a Malvern particle size analyzer. This measurement is carried out with Malvern Mastersizer 2000 according to ISO 787/7 standard.

- 30 By using aggregates with such particle sizes, radiation impermeability and radon gas tightness of the fiber-reinforced concrete according to the present application are increased. More particularly, the physico-mechanical properties, and thus the thickness of the fiber-reinforced concrete are optimized.

In one aspect of the present invention, the fiber-reinforced concrete according to the present invention contains at least one aggregate having two different particle sizes. Here, particle sizes vary between 1-4000  $\mu\text{m}$ .

5 In one aspect of the present invention, the fiber-reinforced concrete according to the present application has a thickness (wall thickness) of 4-7 cm, and preferably of 3-6 cm. A number of concrete forms with different wall thicknesses obtained through a precise measurement with a ruler are filled with the optimized concrete mortars to give those mortars the shape of such forms. Following setting, the microstructure of the produced concrete mortar is measured with  
10 known methods (such as scanning electron microscope, SEM) in the technical field.

In another aspect of the present invention, the fiber-reinforced concrete according to the present application contains at least one superplasticizer. Here, superplasticizers are selected among acrylate, methallylsulfonate, lignosulfonate, polynaphthalene, sulfonic acid alkali metal salt,  
15 polycarboxylate polymer (polycarboxylic acid salt), and mixtures thereof, and are preferably polycarboxylate polymer.

A superplasticizer according to the present application is included in the fiber-reinforced concrete in the range of 0.1-5%, and preferably of 0.25-60%.

20 In another embodiment of the present invention, the superplasticizer is polycarboxylate polymer, and it is included in the fiber-reinforced concrete in the range of 0.1-5%, and preferably of 0.25-60%. By means of such a rate of superplasticizer, the rate of water in the fiber-reinforced concrete was decreased, and the diffusion of radon gas into the pores of concrete was minimized.  
25 The diffusion of radon gas into the pores appears to be directly proportionate to the rate of water in the fiber-reinforced concrete. Particularly, radon gas tightness of fiber-reinforced concretes of which the ratio of water content to the amount of cement material varies from 0.1 to 0.5 and preferably from 0.2 to 0.35 was found to be approximately 100%.

30 In one aspect of the present invention, the water:cement material ratio of the fiber-reinforced concrete according to the present application is preferably between 0.2-0.35, and more preferably between 0.23-0.30.

In one aspect of the present invention, the fiber-reinforced concrete may additionally contain cement materials, aggregates, surfactants, superplasticizers, and curing materials.

5 In one aspect of the present invention, the fiber-reinforced concrete contains at least one curing material. Herein, the said curing material is selected among para toluene sulfonic acid (PTSA), phthalic acid anhydride, aromatic polyamines, polymercaptan polymers, acrylic copolymer, vinyl acetate and ethylene polymer dispersion, and mixtures thereof, and is preferably acrylic copolymer, and more preferably vinyl acetate and ethylene polymer dispersion.

10 A curing material according the present application is included in the fiber-reinforced concrete in the range of 0.1-5%, and preferably of 1-1.16%.

In another aspect of the present invention, the fiber-reinforced concrete contains at least one surfactant. Here, the surfactant is selected among polyvinyl alcohol,  
15 polyoxyethylenemonomethyl ether, dodecylbenzene sodium sulfonate, polyethylene lauryl ether, non-ionic isopropyl alcohol fatty acid ester and derivates, non-ionic glycol fatty acid ester and derivates, polyethylenepropylene glycol, and mixtures thereof, and is preferably polyethylenepropylene glycol.

20 A surfactant according to the present application is included in the fiber-reinforced concrete in the range of 0.001-2%, and preferably of 0.001-0.004%.

In an embodiment of the present invention, the fiber-reinforced concrete according to the present application contains the following components:

25

- a) a cement material in the range of 30-60%,
- b) at least one aggregate in the range of 30-80%,
- c) at least one superplasticizer in the range of 0.1-5%, and/or
- d) at least one fiber in the range of 1-5%.

30

In another aspect of the present invention, the fiber-reinforced concrete according to the present application contains the following components:

- a) a cement material in the range of 35-50%,

- b) at least one aggregate in the range of 40-70%,
- c) at least one superplasticizer in the range of 0.25-60%, and/or
- d) at least one fiber in the range of 1.5-3.5%.

5 In one aspect of the present invention, the fiber-reinforced concrete according to the present application contains the following components:

- a) a cement material in the range of 30-60%,
- b) at least one aggregate in the range of 30-80%,
- 10 c) at least one curing material in the range of 0.1-5%,
- d) at least one superplasticizer in the range of 0.1-5%, and/or
- e) at least one surfactant in the range of 0.001-2%.

15 In another aspect of the present invention, the fiber-reinforced concrete according to the present application contains the following components:

- a) a cement material in the range of 35-50%,
- b) at least one aggregate in the range of 40-70%,
- c) at least one curing material in the range of 1-1.6%,
- 20 d) at least one superplasticizer in the range of 0.25-0.60%, and/or
- e) at least one surfactant in the range of 0.001-0.004%.

25 In one aspect of the present invention, the fiber-reinforced concrete according to the present application contains 0.25-0.6% polycarboxylate polymer as a superplasticizer, and 0.1-5% vinyl acetate and ethylene polymer dispersion as a curing material.

In one aspect of the present invention, the fiber-reinforced concrete according to the present application contains 0.25-0.6% polycarboxylate polymer as a superplasticizer, 0.1-5% vinyl acetate and ethylene polymer dispersion as a curing material, and 0.001-2%  
30 polyethylenepropylene glycol as a surfactant.

In one aspect of the present invention, the fiber-reinforced concrete according to the present application contains an aqueous chemical mixture comprising at least one surfactant, at least one

superplasticizer and at least one curing material. Here, such aqueous chemical mixture preferably comprises polyethylenepropylene glycol, polycarboxylate polymer, and acrylic copolymer.

5 In one aspect of the present invention, the process for preparing the fiber-reinforced concrete according to the present application comprises the following steps:

- a) at least one aggregate and at least one cement material are put into a mixer,
- b) at least one surfactant, at least one superplasticizer, and at least one curing material are mixed with water to form an aqueous chemical mixture,
- 10 c) this aqueous chemical mixture is mixed with the mixture obtained in step a) under appropriate conditions and speed, and
- d) the final mixture is applied to the concrete form together with fiberglass.

15 In another aspect of the present invention, the process for preparing the fiber-reinforced concrete according to the present application comprises the following steps:

- a) at least one aggregate and at least one cement material are put into a mixer, and a solid mixture is obtained,
- b) at least one surfactant, at least one superplasticizer, and at least one curing material are mixed with water to form an aqueous chemical mixture,
- 20 c) the solid mixture is stirred at 20 rpm for 35 seconds,
- d) the aqueous chemical mixture and the solid mixture are stirred at 11 rpm for 55 seconds,
- e) the resulting mixture from step d) is stirred at high speed for 35 seconds, and
- f) the resulting mixture from step e) is applied to the concrete form together with fiberglass.

25 In another aspect of the present invention, the process for preparing the fiber-reinforced concrete according to the present application comprises the following steps:

- a) a number of aggregates, namely barite, colemanite, and magnetite, are put into a mixer together with at least one cement material,
- b) polyethylenepropylene glycol, polycarboxylate polymer, and acrylic copolymer are mixed with water to form an aqueous chemical mixture,
- 30 c) this aqueous chemical mixture is mixed with the mixture obtained in step a) under appropriate conditions and speed, and

d) the final mixture is applied to the concrete form together with fiberglass.

The present invention is further described by the following examples. Although these examples are not intended to limit the scope of the present invention, the details thereof should be considered and evaluated in the light of the foregoing description.

#### EXAMPLES:

##### Example 1:

10

Components	Type of component	Premix and Spray GRC mixture ratios (%)
Cement	CEM II B/L 42.5 White CEM II 42.5 R Gray	35-50
Aggregate composition	Barite - colemanite - magnetite	40-70
Water/cement		0.23-0.30
Chemical 1	Curing material	1-1.6
Chemical 2	Polycarboxylate superplasticizer	0.25-0.60
Chemical 3	Surfactant	0.001-0.004
Fiberglass		1.5-3.5

#### Preparation Process:

- 15 a) a number of aggregates, namely barite, colemanite, and magnetite, are mixed with a cement material,
- b) polyethylenepropylene glycol, polycarboxylate polymer, and acrylic copolymer are mixed with water to form an aqueous chemical mixture,
- c) this aqueous chemical mixture is mixed with the mixture obtained in step a) under appropriate conditions and speed,
- 20 wherein an automatic stirring system is employed:  
in the first stage, stirring liquid raw materials at 11 rpm for 55 seconds,

in the second stage, stirring solid raw materials at 20 rpm for 35 seconds, having a break, and proceeding at high speed after the break, and

in the third stage, stirring at high speed for 35 seconds, and

d) the final mixture is applied to the concrete form together with fiberglass.

5

The fiber-reinforced concretes according to the present application provide the following properties:

Flexural strength: 4-5 MPa

10 Water absorption:  $11 \pm 3\%$

Dry density: 1.9-2.2 g/cm<sup>3</sup>

Freeze-thaw effect:  $RL \geq 0.75$

Largest dimensional changes due to moisture:  $1.2 \pm 0.3$  mm/m

Modulus of elasticity, MOE: 10,000 - 25,000 (TS EN 11701 Standards 1-8)

15

Radiation Attenuation Property	Compared to the anti-radiation concretes in literature, at least 10% higher linear attenuation coefficients (cm <sup>-1</sup> ) for gamma and neutron radiation in FRC products.
Radon Gas Tightness Property	< 4 pCi/L (148 Bq/m <sup>3</sup> ) Source: US EPA < 2.7 pCi/L (100 Bq/m <sup>3</sup> ) Source: World Health Organization (WHO) (Required values within buildings)

The fiber-reinforced concretes according to the present application provide the following properties: (TS EN 196 Standards)

20

25

Compressive strength values: The early strength at 2 days is higher than 20 MPa. The early strength at 28 days is higher than 35.5 MPa.
Initial setting time: $\geq 60$ min.
Soundness: $\leq 10$ mm
Flow: 14.5 - 20.5 cm
Unit weight: $> 2200$ g/cm <sup>3</sup>
Loss on ignition and insoluble residual: $\leq 5\%$
Chloride content: $\leq 0.1$
Sulfate amount: $\leq 3.5\%$

**CLAIMS**

1. A fiber-reinforced concrete containing at least one aggregate composition and at least one superplasticizer, wherein the aggregate composition comprises barite, colemanite, and magnetite.  
5
2. The fiber-reinforced concrete according to claim 1, wherein the aggregate composition comprises 1-60% of barite, 1-60% of colemanite, and 1-60% of magnetite.
- 10 3. The fiber-reinforced concrete according to claim 1 or 2, wherein the aggregate composition comprises barite with a d50 of 1-3000  $\mu\text{m}$ , colemanite with a d50 of 300-750  $\mu\text{m}$ , and magnetite with a d50 of 1-400  $\mu\text{m}$ .
- 15 4. The fiber-reinforced concrete according to claim 1, wherein the superplasticizer is selected from the group comprising acrylate, methallylsulfonate, lignosulfonate, polynaphthalene, sulfonic acid alkali metal salt, polycarboxylate polymer, and mixtures thereof.
- 20 5. The fiber-reinforced concrete according to claim 4, wherein the superplasticizer is polycarboxylate polymer.
6. The fiber-reinforced concrete according to claim 4 or 5, wherein the superplasticizer is included in the fiber-reinforced concrete in the range of 0.25-0.6%.
- 25 7. The fiber-reinforced concrete according to any one of the preceding claims, wherein the concrete additionally contains at least one curing material.
8. The fiber-reinforced concrete according to claim 7, wherein the curing material is included in the fiber-reinforced concrete in the range of 0.1-5%.
- 30 9. The fiber-reinforced concrete according to claim 7 or 8, wherein the curing material is selected from the group comprising para toluene sulfonic acid (PTSA), phthalic acid anhydride, aromatic polyamines, polymercaptan polymers, acrylic copolymer, vinyl acetate and ethylene polymer dispersion, and mixtures thereof.

10. The fiber-reinforced concrete according to claim 9, wherein the curing material is vinyl acetate and ethylene polymer dispersion.
- 5 11. The fiber-reinforced concrete according to any one of the preceding claims, wherein the concrete contains at least one surfactant.
12. The fiber-reinforced concrete according to claim 11, wherein the surfactant is included in the concrete in the range of 0.001-2%.
- 10 13. The fiber-reinforced concrete according to claim 12, wherein the surfactant is selected from the group comprising polyvinyl alcohol, polyoxyethylenemonomethyl ether, dodecylbenzene sodium sulfonate, polyethylene lauryl ether, polyethylenepropylene glycol, and mixtures thereof.
- 15 14. The fiber-reinforced concrete according to claim 13, wherein the surfactant is polyethylenepropylene glycol.
- 20 15. The fiber-reinforced concrete according to any one of the preceding claims, wherein the concrete contains fiberglass as fiber.
- 25 16. The fiber-reinforced concrete according to any one of the preceding claims, wherein the concrete contains at least one cement material.
17. The fiber-reinforced concrete according to claim 16, wherein the cement material is selected from the group comprising white Portland cement, gray Portland cement, and mixtures thereof.
- 30 18. The fiber-reinforced concrete according to any one of the preceding claims, wherein the concrete additionally contains water and a cement material, and the water:cement material ratio is between 0.1-0.5.
19. A process for producing the fiber-reinforced concrete according to claim 1, wherein the process comprises the following steps:

- a) at least one aggregate and at least one cement material are put into a mixer,  
b) at least one surfactant, at least one superplasticizer, and at least one curing material are mixed with water to form an aqueous chemical mixture,  
5 c) this aqueous chemical mixture is mixed with the mixture obtained in step a) under appropriate conditions and speed, and  
d) the final mixture is applied to the concrete form together with fiberglass.

10 **20.** A process for producing the fiber-reinforced concrete according to claim 19, wherein the process comprises the following steps:

- a) at least one aggregate and at least one cement material are put into a mixer, and a solid mixture is obtained,  
b) at least one surfactant, at least one superplasticizer, and at least one curing material are mixed with water to form an aqueous chemical mixture,  
15 c) the solid mixture is stirred at 20 rpm for 35 seconds,  
d) the aqueous chemical mixture and the solid mixture are stirred at 11 rpm for 55 seconds,  
e) the resulting mixture from step d) is stirred at high speed for 35 seconds, and  
20 f) the resulting mixture from step e) is applied to the concrete form together with fiberglass.

25 **21.** A process for producing the fiber-reinforced concrete according to claim 1, wherein the process comprises the following steps:

- a) a number of aggregates, namely barite, colemanite, and magnetite, are put into a mixer together with at least one cement material,  
b) polyethylenepropylene glycol, polycarboxylate polymer, and acrylic copolymer are mixed with water to form an aqueous chemical mixture,  
30 c) this aqueous chemical mixture is mixed with the mixture obtained in step a) under appropriate conditions and speed, and  
d) the final mixture is applied to the concrete form together with fiberglass.