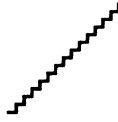


Buildings
Infrastructure
Environment



Investigation of the environmental quality of obsidian with regard to its re-use

Heel4.4
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1. INTRODUCTION

Witteveen+Bos Consulting Engineers b.v. was commissioned by Edelchemie B.V. in the period January - September 1993 to carry out a study on the leaching and radioactivity of obsidian released as a stabilized residual product of vitrification in their production processes.

The study was carried out by Witteveen + Bos in compliance with the Dutch draft standards NEN 7340, 7341, 7343 and 7345 of October 1992 (ref. 1,2,3,4) and the German DIN 38.414 Part 4 (ref. 7). The radioactivity investigation was carried out by the Radiologische Dienst TNO.

The objective of the study was to assess the environmental quality of obsidian with regard to its re-use.

This report comprises the following chapters:

- Chapter 2: description of the production of obsidian in the production process at Edelchemie
- Chapter 3: description of leaching tests and the choice of leaching tests
- Chapter 4: outline of the framework for leaching tests
- Chapter 5: description of leaching test carried out in 1986
- Chapter 6: discussion of the leaching tests carried out
- Chapter 7: comparison of environmental quality of obsidian with other residual materials that are re-used;
- Chapter 8: discussion of the results of the radioactivity investigation;
- Chapter 9: use of obsidian in concrete;
- Chapter 10: conclusions of the investigation.

2. PRODUCTION OF OBSIDIAN

Edelchemie processes highly problematic waste, such as photographic waste, according to the nil-option, by which the waste is pyrolysed. The residual ash is smelted to recover silver and other substances. Other metals and polluting substances are vitrified to obsidian.

Obsidian is stored on the Edelchemie site for about 6 months, where it disintegrates into a separatable product containing pyrite-like enclosures and hard stable lumps of obsidian. The separatable product is smelted again and the obsidian lumps washed. The potential for re-use of the washed obsidian lumps was investigated in this study.

3. SELECTION OF LEACHING TESTS

3.1. Types of leaching tests

In the Netherlands there are draft standards for investigating leaching of materials: these are NEN 7340, 7341, 7343, and 7345 (ref. 1,2,3,4,).

NEN 7340

NEN 7340 gives general guidelines for carrying out leaching tests and for the choice of suitable tests for a particular material. An overview diagram from NEN 7340 is given in Appendix I together with the possible choice of leaching tests based on various criteria.

NEN 7341

This standard sets out the determination method for the availability of inorganic components for leaching from building materials and solid waste materials under laboratory conditions (availability test). This test aims to assess how much of a certain component can be leached from a material under extreme conditions, such as over a long period of time, after disintegration of the material, complete oxidation and loss of neutralizing capacity.

The availability for leaching is determined from a sample of finely ground material extracted twice each for a period of three hours successively, in a ratio of liquid to solids (L/S) of 50 l/kg, with a pH of 7 and 4 respectively. A mixture of both component extracts (pH 7 and pH 4) is subsequently analyzed. On the basis of this test, the quantities of inorganic compounds present in the building material and waste material available for leaching are calculated.

From the data, the acid neutralizing capacity of the material is calculated. This indicates the extent to which a material is resistant to acid corrosion, for example, acid rain.

NEN 7343

This standard describes a determination method for leaching properties of inorganic components from powder and granule building materials and waste materials (column and cascade tests).

The column and cascade tests determine leaching behaviour as a function of time in powder and granular building materials and waste materials. These tests give the cumulative emission (mg/kg) as a function of the relationship between liquid and solid material (L/S), whereby the L/S relationship in practical application can be related to a time scale. The column test simulates the leaching behaviour in the short and medium term and the cascade test in the long-term. The cumulative leaching in the column test beyond L/S 10 (mg/kg ds) can be tested on the leaching limits U_1 and U_2 (see Chapter 4).

NEN 7345

This standard describes the determination method for the leaching properties of inorganic components of solid building materials and waste materials which have predominately inorganic properties.

The leaching of components from solid products, monolithic waste materials and stabilized waste materials is measured by placing a sample under water and analysing the concentration of the components in the water at set times. This test gives the cumulative emission (mg/m^2) as a function of time ($T=0$ to $T=64$ days). The cumulative emission after 64 days in mg/m^2 (if the release by diffusion is determined) can be tested on the U_1 and U_2 leaching limits (see Chapter 4).

3.2. Choice of leaching tests and parameters

Examination of the diagram in Appendix I shows that an availability test according to NEN 7341 and a diffusion test according to NEN 7345 are the methods of choice for the leaching of obsidian (rough waste material with a diameter of > 40 mm). Both tests were carried out and the results are presented in Section 6.3 of this report.

Column tests according to NEN 7343 were carried out in order to assess whether obsidian granules can be re-used (see choice diagram Appendix I). The results are discussed in Section 6.4.

The possibility of re-use in Germany was also checked. In discussions with the Ministerium für Umwelt, Raumordnung und Landwirtschaft des Landes Nordrhein-Westfalen in Düsseldorf, it was decided also to carry out a leaching test in compliance with DIN 38.414 Part 4 (ref. 7). The results are discussed in Section 6.5.

Leaching parameters

The leaching tests were carried out for chloride, sulphate, arsenic, and the 12 heavy metals, cadmium, chromium, copper, lead, nickel, zinc, silver, tin, antimony, selenium, mercury and molybdenum.

4. FRAMEWORK FOR TESTING LEACHING

4.1. Dutch standards 1991

The leaching limits (U_1 and U_2) and the composition limits (S1) for inorganic substances are set out in the Draft Building Materials Guideline (Ontwerp Bouwstoffenbesluit) published in the Government Gazette of June 1991. These guidelines are for both building products and granular building materials. For re-use, building materials must satisfy both the leaching and composition requirements.

The leaching limits U_1 for inorganic substances were set up on the basis that leaching from materials must not be higher than the marginal soil load. A marginal soil load means that as a consequence of leaching, the mean concentration increase in the soil solid phase in the top metre of a soil profile (homogenous soil) over a period of 100 years is 1% of the reference value. The U_2 leaching limits are set at ten times the U_1 limit.

Leaching of inorganic contaminants from building products is measured in the 64 days standard test according to NVN 5432. The cumulative leaching in mg/m^2 after 64 days can be tested against the U_1 and U_2 leaching limits.

Leaching of inorganic contaminants from granular building materials is measured in the column test, conforming to NVN 2508. The cumulative leaching is measured, and expressed in mg/kg building material with L/S (liquid/solid) = 10, that is after 10 ml water per gram building material has percolated and has been collected. The cumulative leaching after L/S = 10 can be tested against the U_1 and U_2 limits. A total of seven fractions are collected in a complete column test in order to follow the leaching process in time (L/S 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0).

If the cumulative leaching in the standard test or the column test is less than U_1 and the total level < S1, then the material is suitable for re-use. When leaching is between U_1 and U_2 and the total level is < S1, re-use is possible, provided soil protection measures are taken (IBC containment). If leaching is > U_2 and/or the total level is > S1, re-use is not permitted and the material must be stored on a waste disposal site.

4.2. Changes to Dutch standards

In 1994, changes to the Dutch standard (Bouwstoffenbesluit) are to be announced in the Government gazette (Staatscourant). On the basis of a policy memorandum from the Minister for Housing, Physical Planning and Environment and the Minister for Public Works and Transport to the Tweede Kamer (Lower House of Parliament; vergaderjaar 1991-1992, 22683 no.1), the standard of June 1991 will most likely be amended.

The U_1 and U_2 leaching standards will no longer be applied for re-use of materials, but instead absorption standards will be applied, which set out the acceptable soil load. Based on the acceptable soil load, different leaching limits (U_1 and U_2) are given for granular building materials for use at different layer depths. Materials with relatively heavy leaching can sometimes be used but then in restricted layer depths.

The acceptable soil load has been increased for different materials on the basis of the results of the recent RIVM study (RIVM report no. 771402005, June 1992). The new leaching limits are more liberal, partly because now, in addition to the acceptable marginal soil load, the natural background leaching has been included in the limits.

Since the above mentioned RIVM report was issued, a number of changes have been made to the leaching standards. These are only marginal changes. In 1994, a new RIVM report will be brought out with the adjusted figures (personal communication with RIVM, 22 June 1993).

For the parameters on which the leaching standards have been set up (heavy metals and a number of inorganic compounds), the composition limits S1 are no longer to be used. Thus

only leaching is important and not the total level.

There is still no leaching standards set for organic substances, such as mineral oil and PAH, because there are as yet no satisfactory leaching tests. For these substances, the total level is the determining factor for their possible re-use. The S1 limits are also more liberal than the S1 limit set in the Ontwerp Bouwstoffenbesluit of June 1991.

The recently published U_1 and U_2 leaching standards (for use in a layer depth of 0.7m) for various inorganic substances (ref 8) are given in Appendix II.

4.3. Standards in Europe

The European Community has not developed a building materials policy and testing framework for leaching. The Netherlands is ahead in this respect. Some other European countries have their own leaching tests and related standards.

In Germany, leaching is tested according to the DIN 38.414 Part 4 (Schlamm und Sedimente, Bestimmung der Eluierbarkeit mit Wasser). This test is applicable to solid, paste-like, and silty materials (< 10 mm). The test is carried out to determine the category of disposal site on which waste material can be dumped. The related leaching standards are relatively high and are not very suitable for testing whether re-use is possible.

In Belgium, a test has been set up to evaluate building materials. This S_2 test gives the total level and leaching.

According to a personal communication with a Belgian Government spokesman, the leaching test is to be changed this year.

5. PREVIOUS STUDIES

In 1986 a leaching study was carried out, in which 100 g ground obsidian was mixed with 900 ml demineralized water (L/S 9) for 24 hours at pH 4. The extract was analyzed for arsenic and 11 heavy metals: cadmium, chromium, copper, mercury, nickel, lead, zinc, tin, antimony, selenium, and silver. The results are given in Appendix III.

The test carried out in 1986 was more vigorous (24 hours, pH 4) than the present availability test in which the samples are mixed for only three hours at a pH of 4. This test only gives an indication of the leachable quantities under extreme conditions. The measured concentrations do not give direct indications of expected leaching behaviour under field conditions.

There is no testing framework for the results of this test. When the results were compared with the most recently published U_1 and U_2 leaching limits for the column test (see Appendix I), only zinc was above the U_1 limit but was below the U_2 limit (zinc leaching 11.25 mg/kg ds). Leaching in the column test will be considerably less.

6. LEACHING STUDY ON OBSIDIAN

6.1. Sampling

On 22 December 1992, a number of obsidian lumps from various parts of the obsidian deposit on the Edelchemie site were sampled.

6.2. Total analyses

A mixed sample of obsidian was analyzed in January 1993 in compliance with the relevant NEN or VPR standards (ref. 5) for the total levels of chloride, sulphate, arsenic, and 11 heavy metals: cadmium, chromium, copper, mercury, lead, nickel, zinc, antimony, tin, silver and selenium. In December 1993 an additional total analysis of molybdenum was carried out. The results of these analyses are set out in Appendix IV. The measured total levels in the obsidian sample were above the C value for soil (see ref.6) for copper, lead, zinc and silver.

6.3. Leaching investigation for re-use of obsidian lumps

6.3.1. Availability tests

Two availability tests were carried out, one in January 1993 and the other in December 1993. In January 1993, the acid /base properties and the acid neutralizing capacity were also reported.

For the availability tests, obsidian lumps were crushed and sieved to a fraction with a diameter of < 125 µm. The availability test was carried out at a laboratory temperature of 20 °C.

Acid/base property

pHA = 8.86

pHB = 9.06

Conclusion: the obsidian has a neutral reaction.

Acid neutralizing capacity (ANC)

Additional weight (M ₁)	:	15.618 g
Moisture content g (40°C)	:	0.00 g/g
Moisture content g (105°C)	:	0.00 g/g
V ₁ + V ₂	:	25.2 ml HNO ₃
C(HNO ₃)	:	1 mol/l

$$\text{ANC} = \frac{V_1 + V_2}{m_1 \times (1 - g_{105} + g_{40})} \times C(\text{HNO}_3)$$

$$\text{ANC} = \frac{25.2}{15.618} = 1.61 \text{ mol/kg}$$

Availability for leaching

In January 1993, a mixed extract of both part extracts (pH7 and pH4) of the availability test was analyzed on the parameters given in Section 3.2, except for molybdenum. The results of the analysis are given in Appendix Va. The mixed extract from the availability test of December 1993 was analyzed for molybdenum. The results are given in Appendix Vb.

In Table 6.1, the results are given of the various components converted to availability on the basis of dry material (mg/kg ds) and relative availability (%) from the total levels as given in Appendix IV.

Just as in 1986, leaching of zinc was verified and also of nickel, molybdenum and antimony. The zinc leaching measured was considerable. As stated in the introduction to NEN 7341, it should be noted that the availability test only gives an indication of the possible cumulative emission over a very long period and under extreme conditions. The measured concentrations do not reflect the expected leaching behaviour under field conditions. The diffusion test and the column test, for which the testing framework is set up, are more suitable for this purpose.

Table 6.1: Results of the availability tests

Parameters	Availability (mg/kg ds)	Relative availability (%)
January 1993		
Chloride	< 1	
Sulphate	0.41	0.5
Arsenic	< 0.52	
Cadmium	< 0.05	
Chromium	< 0.10	
Copper	< 0.52	
Lead	0.62	0.04
Nickel	9.19	2.4
Zinc	165	5.3
Silver	< 10	
Tin	< 0.52	
Antimony	1.24	24.8
Selenium	< 0.10	
Mercury	0.03	> 15
December 1993		
Molybdenum	5.3	12.3

6.3.2. Diffusion test

Test data

Temperature	: 20 ⁰ C
Volume obsidian lump	: 260 ml
Weight	: 790 gram
Volume water	: 1300 ml
Area (estimate)	: 220 cm ²
Periods of water replacement (days)	: 0.25
	: 1
	: 4
	: 9
	: 16
	: 36
	: 64

Results of analyses

The results of the analyses of the seven extracts are given in Appendix VI. Copper and lead were found in all extracts and zinc in one of them.

Data processing

Table 6.2 presents the results of the analyses of the seven extracts processed for emissions per time period and the cumulative emission to t = 64 days (mg/m²).

Table 6.2: Measured emissions from diffusion test (mg/m²)

Period (days)	0-0.25	0.25-1	1-4	4-9	9-16	16-36	36-64	Cumulative
Copper	1.36	0.59	0.95	2.25	1.95	4.20	2.01	13.3
Lead	0.83	0.77	0.83	2.90	1.83	0.71	<	7.9
Zinc	<	<	3.07	<	<	<	<	3.1

In draft NEN 7345, the steps in calculation methods are given to work out whether the release of the contaminated components is determined by diffusion. When these methods are applied to the results of the diffusion test, the following emerges:

- Release of copper and lead only in one part of the middle phase (2.25-16 days) was determined by diffusion. In another part of the middle phase, there was indication of solution rather than diffusion. In the end phase (9-64 days), the supply appeared to be depleted.
- Release of zinc was not determined by diffusion (zinc was only found in the fraction after four days in a concentration just above the detection level. This is possibly the consequence of a one-time surface wash.

In Table 6.3, the measured accumulative leaching after 64 days (mg/m²) is compared with the most recently published U₁ and U₂ leaching limits for the diffusion test in the RIVM report no. 771402005 (ref. 8).

Table 6.3: Evaluation of measured cumulative leaching in diffusion test after t = 64 days

	Measured (mg/m ²)	U ₁ (mg/m ²)	U ₂ (mg/m ²)
Copper	13.3	50	350
Lead	7.9	100	800

The measured cumulative leaching of copper is clearly lower than the U₁ and U₂ limits. On the basis of the results of the diffusion test, there is potential for the re-use of obsidian lumps. This is all the more the case because the release (in addition to diffusion) is determined partly by solution and because in the final phase of the diffusion test, the supply appeared to be depleted.

6.4. Leaching test on re-use of ground obsidian (column tests)

Three column tests were carried out to determine the potential for re-use of ground obsidian. These tests and the results are described below.

Column test 1

The first column test was carried out with ground obsidian (diameter < 4 mm) in January 1993, in conformity with the NEN 7343, in which one percolation fraction L/S 10 was collected and not the earlier in-between fractions. The L/S 10 fraction was analyzed on the relevant parameters. The results of the analyses are set out in Appendix VIIa. Only antimony was present in measurable quantities.

Column test 2

In June 1993 a column test conforming to NEN 7343 was carried out in which seven fractions (L/S 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, 10.0) were collected and analyzed for antimony. A total determination of antimony was also carried out on the obsidian brought into the column. The results are set out in Appendix IV (total level) and in VIIb (fractions in the column test).

In the first fraction (L/S 0.1) 50 ug/l antimony was measured. The level decreased gradually to 8 ug/l in the last fraction. Antimony leaching decreased with time.

Column test 3

In January 1994, an additional column test was carried out, in which the L/S 10 fraction was investigated for molybdenum. The results are given in Appendix VIIc.

In Table 6.4, the results of all three column tests are converted to cumulative leaching (mg/kg ds) and compared with the U₁ and U₂ leaching standards (valid for re-use in a layer depth of 0.7 m) as set out in RIVM report no. 771402005 of June 1992 (ref. 8).

Table 6.4: Evaluation of L/S 10 column test results

Parameter	Measured accumulative leaching L/S 10 (mg/kg ds)	U ₁ , layer depth 0.7 m in mg/kg ds (ref. 8,9)	U ₂ , layer depth 0.7 m in mg/kg ds (ref. 8,9)
1st Column test			
Arsenic	< 0.05	0.9	7.0
Cadmium	< 0.005	0.03	0.07
Chromium	< 0.01	1.0	15
Copper	< 0.05	0.7	4.0
Lead	< 0.05	2.0	9.5
Nickel	< 0.05	1.0	4.0
Zinc	< 0.5	3.5	15
Silver*	< 1.0	-	-
Tin	< 0.05	0.2	2.5
Antimony *	0.25	0.04	0.4
Selenium	< 0.01	0.04	0.1
Mercury	< 0.0005	0.02	0.07
2nd Column test			
Antimony	0.15	0.04	0.4
3rd Column test			
Molybdenum	0.68	0.3	1.0

* = U₁ and U₂ standards not known

In both tests, leaching of antimony was similar, 0.25 and 0.15 mg/kg ds respectively. Leaching was above the U₁ limit but below the U₂ limit. Molybdenum leaching was also between the U₁ and the U₂ limits.

On the basis of the results of the column test (U₁ < antimony and molybdenum leaching < U₂), ground obsidian is suitable for re-use. However, measures must be taken to prevent contact with rainwater and groundwater.

6.5. German leaching test DIN 38.414 Part 4

General

The German leaching test DIN 38.414 Part 4 (ref. 7) was set up for sludge and sediment (Gruppe S) and is the only leaching test applied in Germany, except for the special leaching tests for slag (Hüttenstoffen und Nebenprodukte).

Method

An amount of 100 g dry weight obsidian < 10 mm is mixed for a period of 24 hours with 1 litre demineralized water in a 2 litre bottle. (This test was carried out in the Witteveen + Bos laboratory with 50 g obsidian and 0.5 litre water in a one litre bottle).

Results

The results of the analyses are set out in Appendix VIII.

There are no standards in Germany for acceptable levels in materials and acceptable leaching in relation to re-use of materials. Standards are being prepared which will be applicable throughout Germany. The first section on standards for the re-use of 'Bodenaushub' will be ready in 1994. This will be followed by a section on the re-use of 'Bauschutt', 'Verbrennungsrückstände', 'Hochfenschlacken und Steinkohlflugasche' (personal communication with Dr. Leuchs of the Landesamt für Wasser und Abfall NRW, Düsseldorf).

There are leaching standards (Zuordnungskriterien) which waste materials must satisfy for disposal at certain waste dump sites. Three categories are defined:

category 1: Sonderabfälle: special waste dump sites for heavily contaminated waste (chemical)

category 2: an in-between category

category 3: normal household waste

The Zuordnungskriterien for categories 1 and 2 are set out in Appendix IX. The measured concentrations in the extract were all below the limit for category 1, as expected.

7. COMPARISON OF OBSIDIAN LEACHING BEHAVIOUR WITH OTHER RESIDUAL MATERIALS

On the basis of the leaching investigation, it can be concluded that there is good potential for the re-use of obsidian. Leaching in the diffusion test was well below the U_1 limit, and in the column test only leaching of antimony and molybdenum was between the U_1 and U_2 limits.

This is supported by the leaching behaviour of other residual materials which are re-used in large quantities. The RIVM report no. 771402005 of 20 June 1992 (ref. 9) gives data on leaching behaviour for many residual materials currently being re-used. Data for a number of residual materials are also given in Appendix X. These data are discussed below.

EC fly ash and AVI fly ash are often used as supplementary material in concrete and asphalt. In column tests, EC fly ash leaching is often over the U_2 standard for molybdenum and selenium. The same is the case for AVI fly ash, which also often shows leaching exceeding the standard for cadmium, lead, zinc, barium and chloride.

Oven ash which is also re-used as supplementary material shows less leaching than fly ash in column tests. EC oven ash sometimes exceeds the U_2 standards for selenium and regularly exceeds the U_1 standards for barium and selenium.

AVI slag is very frequently used for roads and water works structures, yet generally in half of the column tests the U_2 standards is exceeded for copper and molybdenum. In the column tests, solid blast furnace slag exceeds the U_1 standard for barium.

Porous blast furnace slag, LD steel slag and phosphorus slag and (non-red) mining material do not or only slightly exceed the U_1 standard.

8. RADIOACTIVITY STUDY OF OBSIDIAN

Two samples of < 2.0 mm of ground obsidian were investigated for low gamma activity by the Radiologische Dienst TNO. The results were interpreted and compared with data in the literature on gamma activity of EC fly ash. The report of the Radiologische Dienst TNO is included in Appendix XI.

The activity concentrations of Ra-226 and Th-232 of the obsidian samples were much lower than those for EC fly ash, while for K-40, activity was slightly higher. In Table 8.1, the mean measured activity concentrations (Bq.kg⁻¹) of Ra-226, Th-232 and K-40 in obsidian are compared with literature values for fly ash, concrete, river clay and dune sand (ref. 10, 11, 12). The Ra-226 and Th 232 activities of the obsidian were even lower than for the river clay and concrete.

Table 8.1: Activity concentrations (Bq.kg⁻¹) of obsidian, fly ash, concrete, river clay and dune sand

	Ra-226	Th-232	K-40
Obsidian	12	12	1.200
Fly ash	180	150	750
Concrete	20	20	150
River clay	20-50	20-80	300-700
Dune sand	5	10	180

9. APPLICATION OF OBSIDIAN IN CONCRETE

Edelchemie has experimented with the use of obsidian up to 4 cm diameter in concrete instead of gravel as supplementary material. The technical aspects have been studied and found to be satisfactory.

On the basis of the result of the leaching investigation, no problems are anticipated. In the diffusion test, the obsidian itself did not leach in excess of the U_1 limit. When obsidian is processed in concrete into a building product, for example concrete tiles, leaching per surface entity will be even less.

The radioactivity study gave no indication for objections to the use of obsidian in concrete. The absorbed doses of radiation in the air per time unit ($\text{Gy}\cdot\text{hr}^{-1}$) have been calculated for three situations on the basis of the values given in Table 8.1 for Ra-26, Th-232 and K-40 as prepared by the Radiologische Dienst TNO. The results are as follows:

- 30 cm concrete layer	26 $\text{nGy}\cdot\text{hr}^{-1}$
- 30 cm concrete layer with 10% fly ash	43 $\text{nGy}\cdot\text{hr}^{-1}$
- 30 cm concrete layer with 10% obsidian	30 $\text{nGy}\cdot\text{hr}^{-1}$

For comparison, the mean background absorbed dose of radiation at ground level distributed over the world is 35 $\text{nGy}\cdot\text{hr}^{-1}$.

10. CONCLUSIONS

On the basis of the performed investigation, the following conclusions can be drawn:

- Leaching of metals from obsidian in the diffusion test was well below the U_1 standards. This indicates potential for re-use of obsidian lumps and also obsidian as supplementary material in concrete.
- In the column test, antimony and molybdenum leaching was measured $> U_1$ and $< U_2$. On the basis of this, the re-use of ground obsidian (diameter < 40 mm) is possible, provided adequate soil protection measures are taken.
- Leaching of metals from obsidian was shown to be less than from than other materials such as fly ash, oven ash and AVI slag, which are re-used for many purposes.
- The measured radioactivity concentrations of Ra-226 and Th-232 from obsidian are much lower than from EC fly ash and even still lower than that from river clay and concrete. The K-40 activity of obsidian was higher than that of fly ash. Processing obsidian in concrete does not result in a relevant increase in radiation.

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