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# METHOD OF ASBESTOS SEPARATION IN SOIL SAMPLES AND DETERMINATION BY OPTICAL MICROSCOPE

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## ABSTRACT

A new method of asbestos determination for quantitative analysis in environmental samples, e.g., soil and water has been developed. The proposed procedure is inexpensive and relatively fast and easy. It can be used e.g. for quantitative determination of asbestos in the soil after the removal of asbestos containing materials for confirmation of the efficiency of work executed. A Polish Patent Application for the procedure of asbestos determination and an analytical set for asbestos determination have been prepared. The analytical set for asbestos determination is based on optical microscopy with optional phase contrast, and is equipped with a digital camera connected to a computer with the appropriate software. The procedure of determination involves leaching the asbestos fibers from the soil and subsequently investigation of leachate by optical microscope. A surface active agent is used for a better leaching of the asbestos fibers. It has been shown, that application of the surface active agent for analysis improves the accuracy of determination. We would like to briefly present the proposed procedure of asbestos determination and the results of studies on the optimization of this procedure, especially in soils.

Keywords: asbestos determination, airborne fibers, microscopic analysis

## INTRODUCTION

The harmful properties of asbestos fibers are well-known and widely described [1-3]. A health hazard is caused by asbestos fibers being brought into the human body through the respiratory system [4]. The size of the particles is important: danger is only brought about by airborne fibers, i.e., those longer than 5 μm, smaller than 3 μm in diameter and with the length-to-diameter ratios greater than 3:1.

The introduction and use of asbestos in the European Union has been totally banned since 2005 [5]. In Poland, the act banning the use of materials containing asbestos has been in force since 1997 [6]. According to the regulations of the Polish Health Minister asbestos, is included in the carcinogen Category 1 and its **boundary concentration is 0.1%**.

For all intents and purposes asbestos determination is restricted to samples of air, primarily in workplaces. Moreover, other components of the environment, including water and soil are neglected despite the fact that they can also be contaminated and can be a source of airborne fibers of asbestos in the air [7].

The identification of asbestos minerals is based on the appearance of minerals and fibers, the chemical composition of the minerals and their crystalline build. The following methods are applied for determination of asbestos [8]: TEM (Transmission Electron Microscopy), SEM (Scanning Electron Microscopy), PLM (Polarised Light Microscopy), PCM (Phase Contrast Optical Microscopy), XRD (X-Ray Diffractometry), IR (Infra-red Spectroscopy), XRF (X-Ray Fluorescence).

Nowadays, problems are caused by asbestos-containing waste [9]. According to careful estimates, the are about 15 mld tons of various waste containing different levels of asbestos in Poland alone. A kind of disposal other than landfill is possible, after the Waste Act has been amended. All these facts create the requirement to determine the content of asbestos, mainly quantitatively, for samples other than air. Unfortunately, in Poland, though not only here, the increasing industry demands are not followed by developments in determination methods. A majority of determination standards are obsolete, for example, the only Polish Standard PN-88 Z-04202/02 *Air purity protection. Tests for asbestos. Determination of the number concentration of respirable asbestos fibres in work places by optical microscopy* [10], is from 1988 and analytical techniques have developed observably since then. Another difficulty is that the existing asbestos determination methods are intended, first of all, for air samples, for qualitative analysis. Finally, sparse methods for quantitative asbestos analysis are very expensive and dedicated mainly for building materials rather than for environmental monitoring.

A new method of asbestos determination for quantitative analysis in environmental samples, e.g., soil and water has been described. The proposed procedure is not expensive and relatively fast and easy. It can be used, e.g., for the quantitative determination of asbestos in the soil after the removal of asbestos-containing materials, for confirmation of the efficiency of work executed. A **Polish Patent Application** [11] for the procedure of asbestos determination and an analytical set for asbestos determination have been prepared.

## EXPERIMENTAL

It was the main goal of our work to elaborate a method for determination of airborne fibers of asbestos in environmental samples, e.g., soils and water. The new procedure of determination consists of leaching the asbestos fibers from

soil with the use of surface active agents and subsequently investigation of leachate by means of an optical microscope which is equipped with a digital camera and the appropriate software. Then our work was focused on optimizing the procedure for asbestos determination in soils. Various kinds of surface active agents, at various concentrations have been investigated. The results of asbestos fiber counting by two different researchers have also been compared.

## Equipment

Sample preparation was performed using the following apparatus:

– BIOMIX BroT-10s rotator with speed regulation in the range 0-35 rpm and digital work time control in the range of 1-24 hours. The leaching of the asbestos fibers from soil samples was carried out in 1 L twist polypropylene bottles.
– Acetone Vaporiser P112041 (JS HOLDINGS, UK) using the hot block method for clearing the MCE filter [12, 13] (Mixed Cellulose Ester Membranes, no support pad, 0.8 μm, 25 mm). Additionally, the device is equipped with a heater pad to evaporate any excess of liquid.

Microscopic analysis was performed using the following instruments:

– Glass slide 26 x 76 x 1 mm and cover slip 24 x 36 mm.
– Optical Microscope OPTEK BINO ADVANCED with phase contrast option, magnification in the range 40x-1000x. A 400x magnification was used in the study.
– Digital microscope high-resolution camera MOTIC MOTICAM 2000 2.0 MP, connected to a computer using USB 2.0, imaging chip1/2", live resolution 1600 x 1200 pixels. Photos of the size 800 x 600 pixels were used in the study.
– Computer software MOTIC IMAGES PLUS 2.0 ML which enables the microscopic photos to be recorded (in bmp format) and archived. It has the important advantage of enabling a precise measurement of fibers sizes (length and width) after prior calibration, which means that the Walton-Beckett Graticule, which only enables the estimation of the fiber sizes, may be abandoned.

## Chemicals and reagents

Chrysotile asbestos fibers were purchased from POCh (Gliwice, Poland).

The following soil samples were used for the experiments: clayey soil from the valley of the River Warta in Poznań; podsolic soil from the village Barcinek (Pobiedziska District, Wielkopolskie Province, Poland), podsolic soil from the town of Skoki (Skoki District, Wielkopolskie Province, Poland), soil from the village Kurzelów (Włoszczowa District, Świętokrzyskie Province, Poland).

In our studies were used various kinds of surface active agents:

- **anionic**: soft soap, sodium stearate, Sulfapol E-20;
- **cationic**: didecyldimethylammonium chloride;
- **nonionic**: Triton X.

Water for the experiments was purified in the Milli-Q apparatus.

Acetone for acetone vaporiser was purchased from Chempure (Piekary Śląskie, Poland).

## Sample preparation

90.00 g dry weight of soil (fixed according to the standard PN-EN 12457-4:2006 [14]), the appropriate weighed amount (mass percentage in terms of dry weight of soil) of the surface active agent and water at the liquid-to-solid ratio of 10 L/kg was mixed in a 1 L twist polypropylene bottle. The bottles were put in the rotator, the time of leaching was set to 24 hours, with speed regulation at 10 rpm. The samples were collected after 30 minutes. A twentyfold dilution was used. For the microscopic analysis, a 0.25 ml sample was collected on a glass slide with MCE filter. Next, filter clearing through the medium of acetone vaporiser was performed. For each sample, 50 microscopic photos were taken. Each numbered fiber was measured (width and length) by means of an appropriate computer software.

## Procedures of calculation

The microscopic analysis of the leachate by Phase Contrast Optical Microscopy was based on the Polish Standard PN-88 Z-04202/02 [10] but the document concerns air samples only and adaptation of the formula for fibers counting in a liquid was required.

The primary formula from the Polish Standard is as follows:

$$X = (A \cdot N)/(a \cdot n \cdot r \cdot t) \quad (1)$$

X – asbestos concentration [number of fibers/ml of air],
A – effective collecting area of filter [$mm^2$],
N – total number of fibers counted,
a – microscope count field area [$mm^2$],
n – total number of fields counted on the filter,
r – volume of air flow through the filter [ml/min],
t – time of sample collection.

There are only liquid samples (leachate) in target procedure, therefore, flow parameters should be substituted by volume data. A modified formula is presented by the following equation:

$$C = (A \cdot N \cdot 1000)/(a \cdot n \cdot p) \quad (2)$$

C – asbestos concentration [number of fibers/L],
A – effective collecting area of filter [$mm^2$],
N – total number of fibers counted,
a – microscope count field area [$mm^2$],
n – total number of fields counted on filter,
p – volume of collected samples for microscopic analysis [ml].

In the next step the formula from the Environmental Protection Agency method [15] for recalculating the fiber size (width and long) to their mass was used. A precise measurement of the fibers by optical microscopy made it possible to use an appropriate computer software. After the operation, it is possible to express "N" from Formula (2) as a total of all fibers counted in mg units, then "X" is an asbestos concentration expressed in mg/L. Thus, we can obtain two different values.

In the last stage of the calculation of released component amount was counted according to the Polish Standard PN-EN 12457-4:2006 [14] by means of the following equation:

$$Z = C \cdot [(L/M_D) + (MC/100)] \quad (3)$$

Z – released component amount at a liquid-to-solid ratio of 10 L/kg [number of fibers/kg dry weight] or [mg/kg dry weight],
C – concentration of component in leachate [number of fibers/L] or [mg/L],
L – volume of leaching liquid [L],
$M_D$ – dry mass of analytical sample [kg],
MC – moisture ratio [%].

Depending on the kind of parameter C (in different units) used in the calculations, two different results may be obtained. What is important, thanks to the target method, the results expressed in mg/kg dry weight of soil may be compared with a boundary asbestos concentration of 0.1%.

## RESULTS AND DISCUSSION

### Effect of surface active agent by asbestos determination

The first stage of studies was focused on the increase in the asbestos fibers leaching from the soil into the leachate. Sulfapol E-20 was chosen for the purpose, being a surfactant which is popular and within easy reach. Preliminary experiments on reduced parameters confirmed the influence of Sulfapol E-20, improving the leaching of asbestos fibers into the leachate. The best result was observed for a surfactant mass concentration of 5%.

The next step was an investigation performed precisely with the target procedure. Tests were carried out for clayey soil and podsolic soil (from Barcinek). The first sample of each soil was "blank", the second and third samples were intentionally contaminated (in laboratory conditions) with 0.1% of asbestos per dry weight of soil. And with the third sample, 5% mass concentration of the surface active agent Sulfapol E-20 was added to the leachate. After 24 hours of mixing, the prepared leachate sample was investigated by means of optical microscopes. The results are presented in Table 1 for the clayey soil and in Table 2 for the podsolic soil.

Table 1. Concentration of fibers in clayey soil enabling the number of fibers to be counted (columns 2-3) and recalculated to their mass (columns 4-5).

| Kind of samples | Total number of fibers counted | Concentration of fibers in soil [· $10^9$ fib./kg dry matter] | Total mass of fibers counted [· $10^{-3}$ µg] | Concentration of fibers in soil [mg/kg dry matter] |
|---|---|---|---|---|
| Clayey soil | **15** | **3.60** | **0.04** | **8.74** |
| Clayey soil + 0,1% asbestos | **48** | **11.52** | **0.52** | **125.75** |
| Clayey soil + 0,1% asbestos + surfactant 5% | **88** | **21.11** | **3.54** | **848.26** |

Table 2. Concentration of fibers in podsolic soil enabling the number of fibers to be counted (columns 2-3) and recalculated to their mass (columns 4-5).

| Kind of samples | Total number of fibers counted | Concentration of fibers in soil [· $10^9$ fib./kg dry matter] | Total mass of fibers counted [· $10^{-3}$ µg] | Concentration of fibers in soil [mg/kg dry matter] |
|---|---|---|---|---|
| Clayey soil | **15** | **3.60** | **0.03** | **6.27** |
| Clayey soil + 0,1% asbestos | **57** | **13.68** | **0.57** | **135.77** |
| Clayey soil + 0,1% asbestos + surfactant 5% | **126** | **30.23** | **3.95** | **947.13** |

The presented results confirm the idea that the use of surface active agents increases the number of asbestos fibers being leached from the soil. The concentration of asbestos fibers in soils, expressed in the unit number of fibers/kg dry weight give us only information that the quantity of fibers in the soil, which is able penetrate into the air, is huge. The data cannot be compared with any standards or norms because no directives or other normative documents provide limiting values for soils in such units (number of fibers per kg). Thanks to recalculating the sizes of fibers to their mass, results can be expressed in mg/kg dry weight of soil and compared with referent values which, in this case, is the amount of asbestos used for soil contamination. In samples with the surfactant, results were near to the real asbestos concentration (0.1%) in the soil. On the grounds of such information it is possible to determine the eventual contravention of boundary concentration of a dangerous substance in the soil. Any differences in leaching asbestos fibers from various kind of soils result from their characteristics.

## Effect of type of surface active agent and its concentration

The next stage of our work was optimization of the procedure for asbestos determination in soils. Various kinds of surface active agents in various concentrations have been investigated.

The study was carried out for the following kinds of surfactants:

- **anionic**: soft soap, sodium stearate, Sulfapol E-20;
- **cationic**: didecyldimethylammonium chloride;
- **nonionic**: Triton X.

The mass concentrations of the surface active agent were: 0%; 1%; 3%; 5%; 7%; 10%.

A total of 8 investigations were carried out. The podsolic soil (from Skoki) was used. For the blank test, „pure" soil and soft soap and sodium stearate (1:1) as a surfactant mix were used. Other soil samples were intentionally contaminated with asbestos at the level of its boundary concentration – 0.1% per kg of dry weight of soil.

The leachates, prepared according to the target procedure were subjected to microscopic analysis. For each sample 50 microscopic photos were taken and airborne fibers were counted. Results are presented in Figure 1.

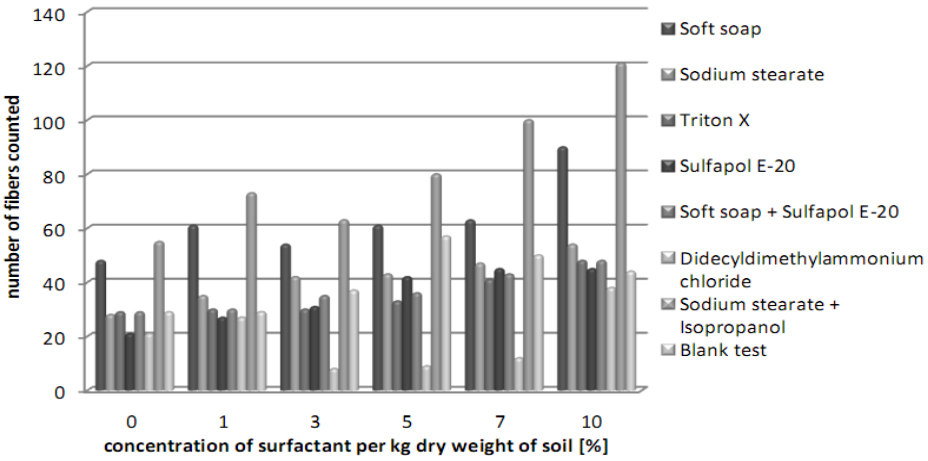


Fig. 1. Graph with the number of fibers counted for various kinds of surfactants, in various concentrations; for each surfactant concentration samples are presented in the order shown in the legend.

Each counted fiber was also measured (width and length) by means of the appropriate computer software. The fiber sizes were recalculated to their mass and the concentration of asbestos fibers in mg/kg dry weight of soil was counted. Results are presented in Figure 2.

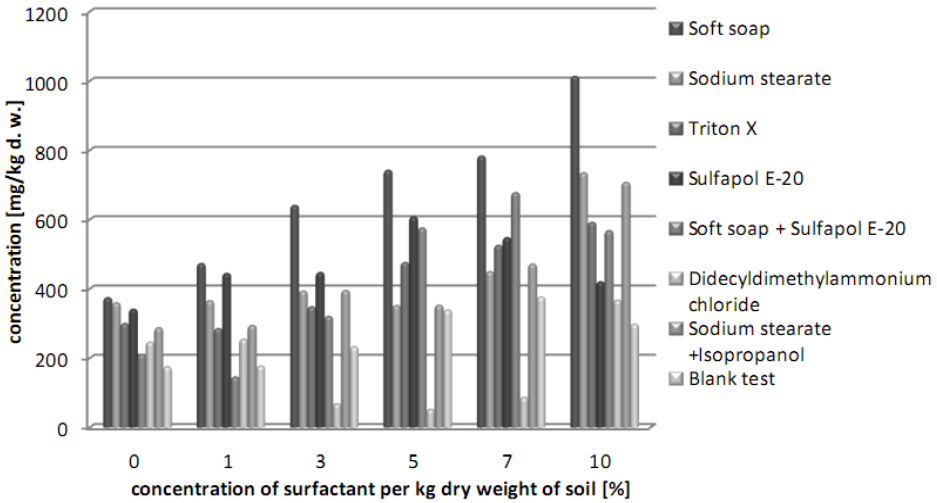


Fig. 2. Graph with the asbestos concentration in soil for various kinds of surfactants, in various concentrations; for each surfactant concentration samples are presented in the order shown in the legend.

The findings confirm that the use of a surface active agent improves the leaching of the asbestos fibers from the soil. The best results were obtained for anionic surfactants at concentrations in the range of 5-10%. For samples with soft soap (especially 10% of surfactant), the concentration of asbestos was the nearest to the real content of asbestos in the soil (0.1%), but the large amount of precipitated residues made the analysis more difficult. The cationic surface active agent (didecyldimethylammonium chloride) caused the lowest leaching of asbestos fibers than pure water, so it is not recommendable for asbestos determination. The influence of surface tension was investigated in samples with sodium stearate as surfactant and 20% of isopropanol in water. Results for these samples are almost similar to those for samples with sodium stearate alone, so surface tension in asbestos determination can be neglected.

## Subjectivism of microscopic determination

In the last stage of our study, the subjectivism of microscopic determination was investigated. Microscopic analysis is a subjective method and to a large extent it depends on the researchers and their experience. The test was carried out for 4 kinds of samples: blank test (pure soil) and for a soil contaminated with asbestos (concentration 0.1%) with the use of surfactants. Every kind of surface active agent was tested: anionic – Sulfapol E-20, cationic – didecyldimethylammonium chloride, nonionic – Triton X. The same microscopic photos were investigated by two different persons. Researcher A had more experience in asbestos fiber counting than had Researcher B. Graphs with the number of fibers counted and with asbestos concentration in the soil, made by two different researchers, for each sample are presented in Figures 3-6.

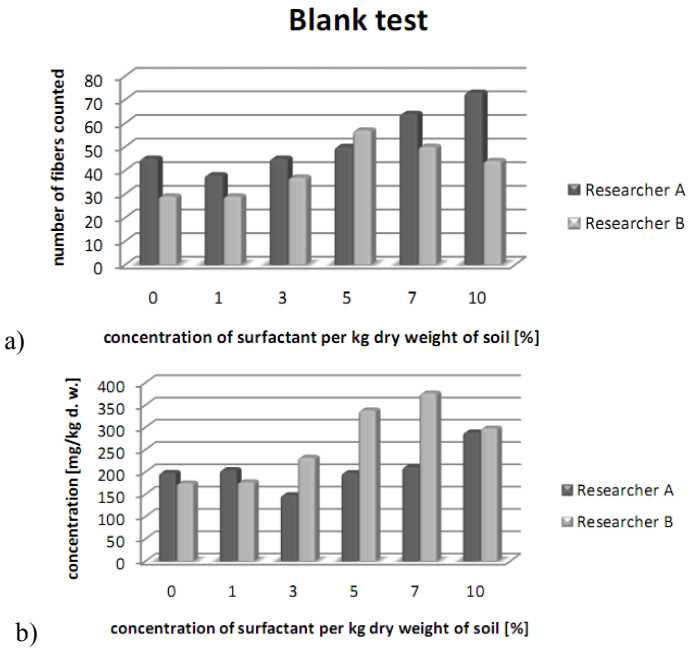


Fig. 3. Graph for a blank sample with the number of fibers counted (a) and with the asbestos concentration in soil (b), at various surfactant concentrations.

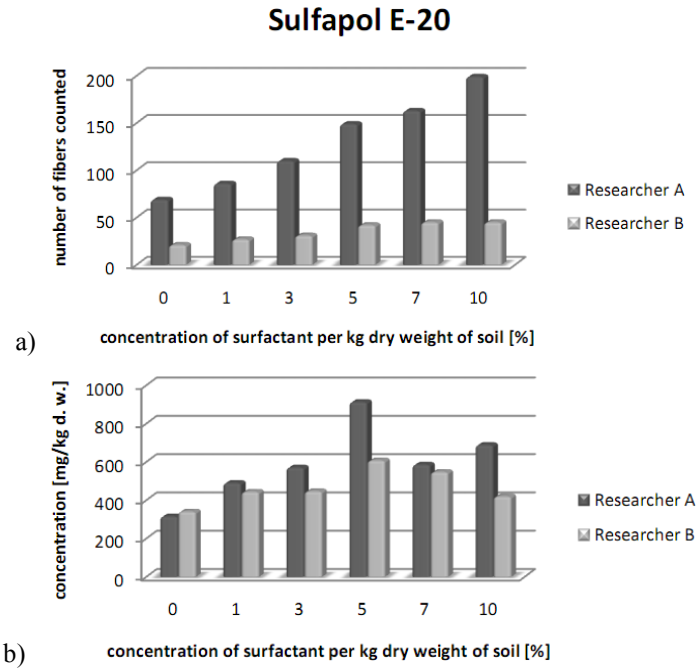


Fig. 4. Graph for anionic surfactant with the number of fibers counted (a) and with the asbestos concentration in soil (b), at various concentrations.

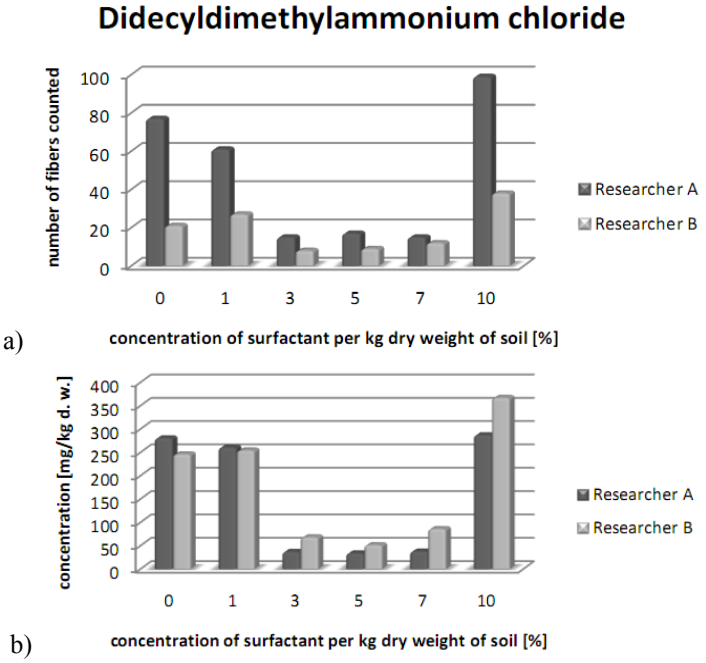


Fig. 5. Graph for cationic surfactant with the number of fibers counted (a) and with the asbestos concentration in soil (b), at various concentrations.

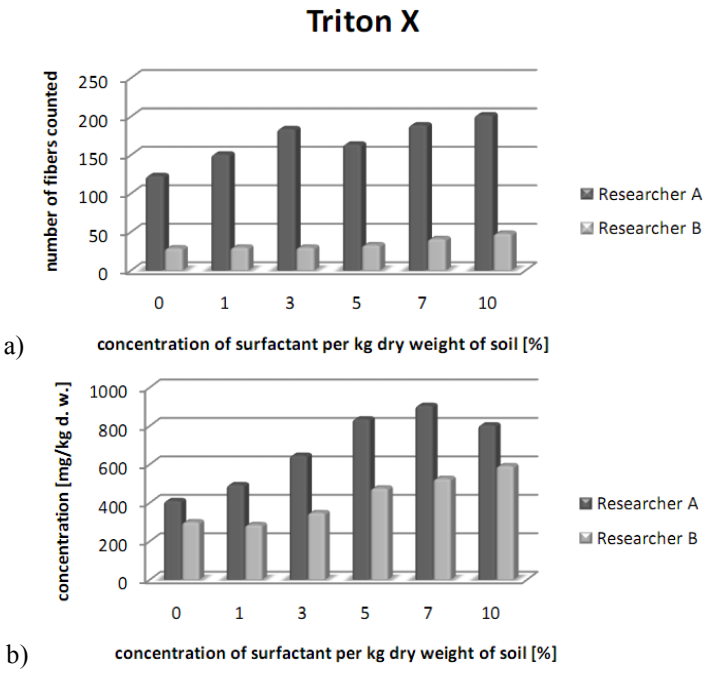


Fig. 6. Graph for nonionic surfactant with the number of fibers counted (a) and with the asbestos concentration in soil (b), at various concentrations.

The presented results show that experience is very important in asbestos determination. In the case of the number of fibers counted in the soil, obtained by two different researchers, the differences are very visible. However, by recalculating the size of fibers to their mass the results are more similar. The fiber mass is exponential depending on its width. Even if there are a lot of thin asbestos fibers in a sample, after recalculating them to their mass, a low value is obtained. This fact confirms that in our method the subjectivism of the method of microscopic determination is greatly reduced.

## Method application

After confirmation of the fact that the target method can be used for asbestos determination in the soil at the boundary concentration (0.1%), environmental samples were investigated. The sample consisted of soil from the ground of a farm building with asbestos cement roof (Fig. 7).

Fig. 7. Place of soil sampling, farm building with asbestos cement roof; village Kurzelów (Włoszczowa District, Świętokrzyskie Province, Poland).

From the Kurzelów soil 2 samples were prepared. For the preparation of the first leachate only soil and pure water were used. The second sample comprised the added surface active agent (Sulfapol E-20) at a mass concentration of 5%. Next, a microscopic analysis was carried out. The results are presented in Table 3.

Table 3. Concentration of fibers in the Kurzelów soil enabling the number of fibers to be counted (columns 2-3) and recalculated to their mass (columns 4-5).

| Kind of samples | Total number of fibers counted | Concentration of fibers in soil [· $10^9$ fib./kg dry matter] | Total mass of fibers counted [· $10^{-3}$ µg] | Concentration of fibers in soil [mg/kg dry matter] |
|---|---|---|---|---|
| Kurzelów soil | **46** | **11.04** | **0.99** | **237.87** |
| Kurzelów soil + surfactant 5% | **71** | **17.03** | **3.47** | **813.43** |

The presented results confirm that the use of a surface active agent improves the leaching of asbestos fibers from soil. The high concentration of asbestos fibers in the soil suggests high environmental pollution with asbestos. For verification of the amount of asbestos in the soil, in Figure 8 this result was compared with references values, i.e., with results for soil intentionally contaminated (in laboratory conditions) with asbestos at a concentration of 0.1% per dry weight of soil.

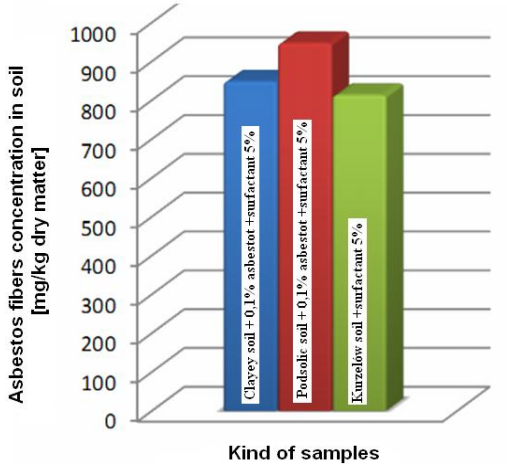


Fig. 8. Comparison of results for asbestos concentration in Kurzelów soil and clayey and podsolic soils contaminated with asbestos of 0.1%.

Concentration of asbestos in the Kurzelów soil is near to the result obtained for the soil intentionally contaminated with asbestos at a concentration of 0.1%, i.e., its boundary concentration. This high environmental pollution suggests that the soil in the vicinity of asbestos-containing materials should be monitored to reduce the risk of asbestos-related diseases and to observe and appropriately act after the eventual contravention of the boundary concentration of the dangerous substance in the soil.

## CONCLUSIONS

The proposed method enables determination of asbestos in the soil at a concentration of 0.1% (boundary concentration). The total mass of the fibers counted should be used in recalculating (comparison with reference values and standards). Anionic surfactants are the best for leaching the asbestos fibers from the soil. The preferred surfactant concentration in the leachate is 5-10%. Experience has an important role in determination of asbestos fibers by counting. Any differences in recalculating the fibers to their mass, obtained by various researchers, are relatively low. The obtained results confirm the opportunity to successfully use the target method, e.g., in environmental monitoring. The results obtained by our innovative methods further support the idea that the development of procedures for asbestos determination is required for better preventing asbestos-related diseases.

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