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Ultrasonic cleaning of catalytic mass grains from a pressure filter

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Abstract: The article presents pilot research on the use of ultrasound during pressure filter backwashing to improve the efficiency of removing impurities from the surface of grains. Ultrasound is widely used in various fields for removing impurities, breaking or crushing. A low-frequency ultrasonic cleaning method was used to remove accumulated iron hydroxide deposits from the catalytic mass grains; this method did not remove manganese from the water. As the preliminary research has shown, the method gives satisfactory results. An improvement in manganese removal efficiency of around 40% was obtained.

Keywords: ultrasonic cleaning, filtration, morphology of grains, catalytic mass

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Introduction

At present, pressure filters are used in most groundwater treatment plants containing elevated iron and manganese concentrations. The filter bed may be filled with sand together with a layer of catalytic material for the removal of manganese from water (Barloková & Ilavský, 2010). Over time, but more often as a result of failure, there is a loss of catalytic ability of filters by the covering of its grains with a layer of impurities, for example, iron hydroxide. Currently, methods for its regeneration are cyclical backwashing with water, water with air or first with air and then with water (Siwiec, 2011). This is to loosen, separate the grains and cause them to move relative to each other. Ultimately, this leads to friction between the grains and flushing of impurities from their surface. However, these three methods

do not always give a positive result. Too low a backwashing performance does not loosen the bed sufficiently for proper grain interaction, while too high a performance causes excessive separation of grains and leaching of lighter bed fractions. In the situation where traditional methods of backwashing fail and it is not possible to regain adequate efficiency of removing impurities on the bed, the operator of the water treatment plant can only replace the expensive filter bed.

Ultrasound is now widely used in various fields of technology and science. Examples include the ultrasonic breaking of kidney stones, removal of the dental plate or cleaning of various elements in so-called ultrasonic cleaners. In the field of environmental engineering, ultrasonic techniques are used in wastewater treatment (Mahamuni & Adewuyi, 2010; Gogate, 2008), removal of pollutions in membrane processes (Doosti et al., 2012; Drakopoulou et al., 2009) or disinfection (Mason et al., 2003). According to the available literature, no one has ever used ultrasound to regenerate a blocked filter bed with catalytic mass. This may be due to the limited availability of research material. A new rinsing method is proposed, which significantly extends the service life of the iron and manganese removal bed and can restore the sorption performance of the blocked bed.

In the application of ultrasound, important parameters include the time and frequency of their action on the element immersed in water (Khanal et al., 2007; Jian et al., 2005). Too long a time causes excessive breaking of grains into small fractions that can be washed out of the filter (Cai et al., 2010). However, too short a time does not allow removal of the accumulated layer of deposit. Similarly, if the frequency is too high, it can break down not only the deposit but also the grains. Too low a frequency will not bring the desired result.

The article presents the results of pilot studies on the method of backwashing the filter bed with a catalytic mass in combination with ultrasound.

1. Methodology

1.1. Subject of research

Material for the study was a bed of a pressure filter used for removing iron and manganese with a catalytic layer Multiman 3M (1.0 m quartz sand and 0.4 m Multiman 3M mass). Catalyst mass material was taken from the layer. The filter was part of the underground water treatment plant system with a maximum production of 37 m³/h using underground water and supplies treated water to rural distribution networks. The Multiman 3M catalytic mass responsible for the removal of excessive manganese content is a filtration material whose main component is the MnO₂ manganese ore (MnO₂ content of minimum 82%).

After several years of operation, it was found that the concentration of manganese reaching treated water increased to 190 µgMn/dm³ and exceeded the limit value (50 µg/dm³) almost four times. Attempts to improve the efficiency of water treatment by increasing the intensity and frequency of backwashing the filter did

not bring the expected results. Inspection of the filter bed, in particular of the catalytic mass, showed that the Multiman 3M grains were completely covered with a layer of iron hydroxide due to aerator failure, and their shape and size changed significantly (Dobrzański & Jodłowski, 2018).

1.2. Analytical methods

The samples were sonicated using a laboratory ultrasonic cleaner with a power of 640 W and a frequency of 35 kHz. Beakers with samples of the analyzed catalytic mass were placed in it. The operating time of the ultrasonic cleaner was changed in the range of 2-60 min.

The assessment of the impact of ultrasound on catalytic mass was carried out on the basis of testing the effectiveness of manganese removal and analyzing changes in morphological characteristics.

The equivalent diameter of the catalytic mass grains was measured on a Morphologi G3 microscopic image analyzer. It allowed the observation of particles with a diameter in the range 0.5-3000 μm . Measured by the device, the equivalent diameter (d_e , mm) is the diameter of the circle with the same surface as the image of the recorded particle. Other analyses morphology parameters were Circularity – the ratio of the perimeter circle to the same surface as the particle divided by the perimeter of the actual particle image (assumes values from the nearest 0 to the value 1.0 for the ideal circle), and Solidity – the actual surface of the particle divided by the surface of the tensile thread that enters the particle (assumes values from 0 to 1.0 – the ideal circle). The measurement was made several times to analyze a larger number of particles.

The analysis of the effectiveness of manganese removal by catalytic mass was made on a laboratory scale by adding a constant amount of mass 500 g to a beaker with prepared 500 ml water with a known concentration of manganese 610 $\mu\text{gMn}/\text{dm}^3$. Manganese concentration was determined according to the spectrophotometric method.

2. Results and discussion

In order to better illustrate the research material, photographs of the surface of the grains of the blocked bed together with a layer of accumulated iron hydroxide were taken with the help of a microscope (Figs. 1 and 2). In cross-section (Fig. 1) it can be seen that the deposit consists of layers alternately made of light and dark material. It is believed that the light-colored layer is iron hydroxide. Layers of dark material are probably catalytic mass dust resulting from grain friction. The deposit layer was estimated to have a thickness in the range of about 100 to 320 μm .

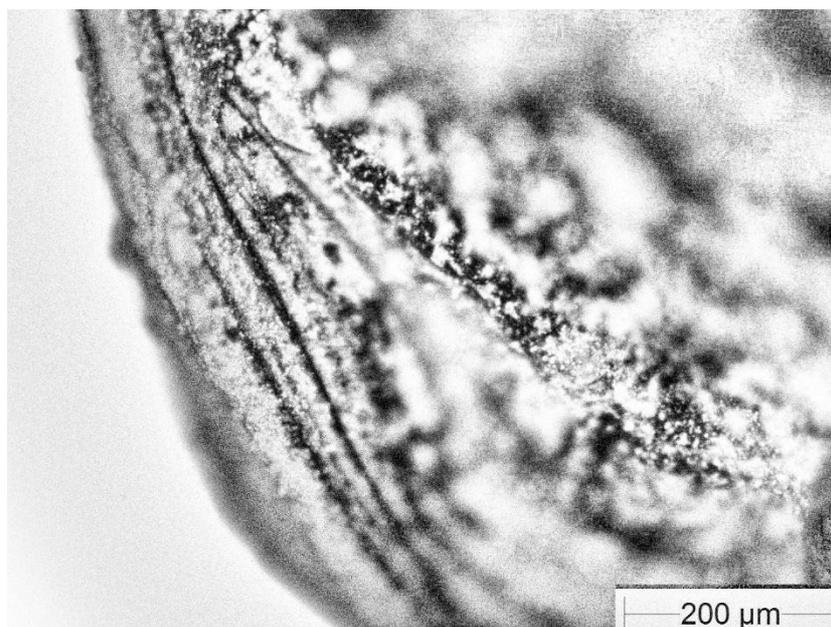


Fig. 1. Photo of the cross-section of the layer accumulated on the surface of the catalytic mass grain (*own research*)



Fig. 2. Photograph of the grain of the catalytic mass together with a cracked shell from accumulated sludge (*own research*)

Test samples were subjected to ultrasounds with a power of 640 W and a frequency of 35 kHz changing the duration of ultrasound in the range of 2-60 min. After this process, the sample was backwashed. Then, the efficiency of manganese removal from prepared control water was checked for each sample. On the basis of the results obtained, a graph is presented in Figure 3.

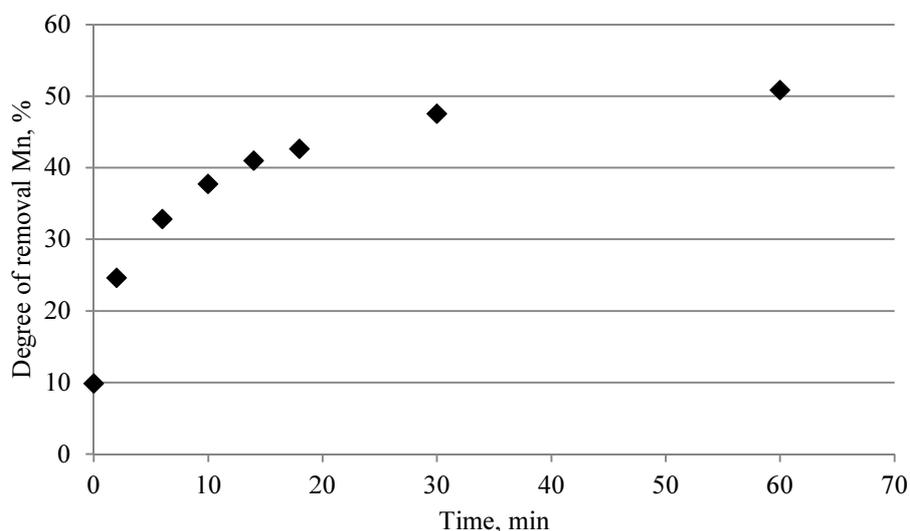


Fig. 3. The degree of manganese removal from water depending on the duration of ultrasound at their constant frequency and power (*own research*)

As can be seen in Figure 3, the maximum manganese removal efficiency was 51%, i.e. 41% higher than for the control sample without the use of ultrasound in conventional backwashing. This translates into a manganese removal efficiency of 3.0 to 6.2 μgMn per gram of Multiman 3M mass for a time of 2 to 60 minutes, respectively. It should be mentioned that for the new catalytic mass the degree of manganese removal was measured at 72%. This corresponds to the manganese removal efficiency for the new catalytic mass of 8.8 $\mu\text{gMn/gMk}$. More importantly, already with a backwashing time of 10-18 minutes, the recovery effect is equally high. It should be mentioned that this is the most commonly used backwashing time range at water treatment plants.

An important aspect was to prevent excessive crushing of the catalytic mass grains. For this purpose, the samples were examined using microscopic image analysis. This allowed the assessment of changes in the shape and size of grains before and after the backwashing process with ultrasound (Fig. 4).

The analysis of particle morphology of the new catalytic mass showed that in the batch used for testing grain equivalent diameter (d_e) in the range of 1.0-3.0 mm was dominated. From the statistical analysis of the d_e value distribution, it was found that the expected d_e value was 1.67 mm with a standard deviation of $\sigma = 0.28$ mm – log-normal distribution.

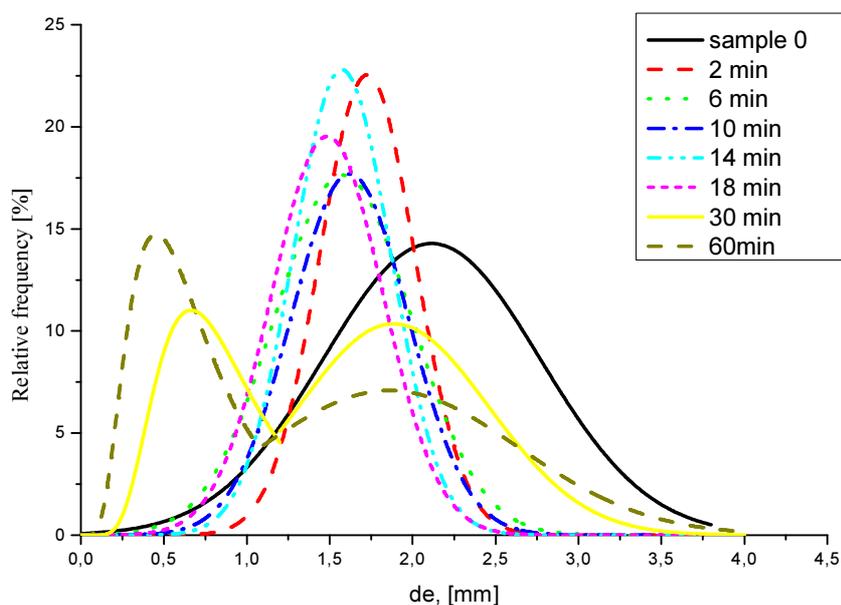


Fig. 4. The distribution of the equivalent diameter depends on the duration of the ultrasound
(own research)

However, based on the results of the analysis of the particles of catalytic mass taken from the filter bed, it was found that the equivalent diameter of the particles was mainly in the range of 1.0-3.3 mm (Fig. 3). The expected value d_e of 2.1 mm and a standard deviation of 0.51 mm were determined.

It can be seen in Figure 4 that the long-term impact of ultrasound on grains leads to damage. In the case of ultrasound operating time in the range of 2 to 18 minutes, the distribution graph moves slightly towards smaller diameters, which proves the removal of accumulated deposit layers from their surface. Furthermore, the equivalent diameter distribution is substantially narrowed down to a range of 1.0-2.5 mm. The expected value of distribution diameter for these samples fluctuates within 1.5-1.8 mm. This value is similar to the grain diameter distribution of the new catalytic mass. Too long a sonication on the sample causes excessive grains breaking. This is evidenced by the second peak of equivalent diameter distribution (for 30 and 60 minutes) appearing within 0.1-1.0 mm diameters, which is not recorded for the remaining samples.

There were no significant changes in shape parameters (Table 1).

The parameters circularity and solidity for most of the tested samples remain at a similar level to the 0 sample. This means that the accumulated layer of deposit was removed evenly from the grain surface. This can be considered an advantage of the ultrasound method. The change in morphological parameters occurs only for 30 and 60 minutes samples. This causes the appearance of a second population of particles depicted in Figure 4 through bimodal distribution.

Table 1. Summary of data regarding changes in parameters of Circularity and Solidity of catalytic mass grains relative to the duration of ultrasound (*own research*)

Time [min]	Solidity			Circularity		
	Average	90 perc.	10 perc.	Average	90 perc.	10 perc.
	[mm]	[mm]	[mm]	[mm]	[mm]	[mm]
0	0.98	1.00	0.96	0.93	0.97	0.87
2	0.98	1.00	0.95	0.92	0.97	0.86
6	0.98	1.00	0.95	0.92	0.96	0.86
10	0.98	1.00	0.94	0.92	0.96	0.86
14	0.98	1.00	0.95	0.92	0.96	0.86
18	0.97	1.00	0.94	0.91	0.96	0.84
30	0.95	0.99	0.91	0.87	0.93	0.80
60	0.95	0.99	0.91	0.86	0.92	0.79

Conclusions

- The proposed method of filter backwashing brings the intended benefits. However, it should be remembered that it does not enable achieving the parameters of the new catalytic mass.
- Relatively short ultrasound times of up to 20 minutes should be used. This allows regeneration of the catalytic mass up to 40%.
- The deposit layer is removed evenly from the grain surface. There are no changes in the morphological characteristics of the grains.

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