

Annals of Warsaw University of Life Sciences – SGGW
 Land Reclamation No 44 (1), 2012: 63–73
 (Ann. Warsaw Univ. of Life Sci. – SGGW, Land Reclam. 44 (1), 2012)

A study of application of chalcedonite as a manganese dioxide carrier

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Abstract: *A study of application of chalcedonite as a manganese dioxide carrier.* Chalcedonite is a sedimentary siliceous rock, which occurs at four deposits in Poland and is included into a group of unique rocks. Chalcedonite is utilized in water treatment technology, mostly as an effective filtration material due to its mesoporous structure and extended outer surface of the grains. This paper presents three different methods of impregnation of the mineral material (MDMC-1, MDMC-2, MDMC-3) by manganese dioxide. As an oxide carrier chalcedonite was used. The results of the chalcedonite surface modification with SEM-EDS technique were presented. It was found that the chalcedonite is a very good manganese dioxide carrier and the modification of the chalcedonite surface changes its chemical composition, structure and value of the specific surface area.

Key words: chalcedonite, siliceous rocks, manganese dioxide, modified materials.

INTRODUCTION

Chalcedonite is a siliceous sedimentary rock and occurs at “Dęborzynka”, “Gapinin”, “Lubocz” and “Teofilów” deposits, located on the Rawska Plateau in the region of Tomaszów Mazowiecki and Nowe Miasto. Only the deposit “Teofilów” is documented and it is now being developed. The surface of this deposit is 577,437 m² and its geological resources were estimated to be 21.587 × 10⁶ kg (21,587 thousand ton) (Tchórzewska and Pabis 1997). The processing of the output consists of multiple breakages and sifting which ends in drying. It

enables preparing the moisture-free material with particular fractions. The end product is chalcedonite sand with grains of 0.8 – 2.0 mm and gravel with the following grain sizes: 2.0–4.0 mm, 4.0–8.0 mm, 8.0–16.0 mm and 16.0–32.0 mm. The chalcedonite sand and gravel have a Hygienic Certificate PZH issued on the 31st of July, 2008 (www.microsil.com.pl). In the territory of the mine more than 1.2 mln sq m of chalcedonite sand with the grains ≤ 2 mm is mined, it is deposited in a settling ditch (Kosk 2010).

The main component of the rock is chalcedony, but quartz, opal, ferric hydroxides, pyrite, manganese compounds and silty minerals also appear. The mineral composition of chalcedonite in volume percent is as follows: chalcedony, opal and autogenic quartz 68.3–95.4%, quartz and other terrigenous components 0.3–6.6%, free and filled pores 2.0–24.7% (Ratajczak and Wyszomirski 1991). The fractions of chalcedony gathered in the settling ditches of the mine and used in the technology of water and wastewater treatment are characterized by a similar mineral composition: chalcedony and autogenic quartz 81.6–88.6%, terrigenous quartz 3.6–14.6%, feldspars 0.3–1.7% and silty rock grits 2.0–8.5% (Kosk 2010). The chemical composition of chalcedonite is almost homogeneous because the content of silicon dioxide exceeds 94% and the

analysis of the surface using the EDS technique showed spectral signals of silicon dioxide and oxygen (Michel 2011). The specific surface area is relatively small and was estimated to be $6.13 \text{ m}^2/\text{g}$ (Michel 2008a) and $3.25\text{--}8.67 \text{ m}^2/\text{g}$ (Ratajczak and Wyszomirski 1991). The specific density of the chalcedonite sand is comparable to that of quartz sand and is $2602 \text{ kg}/\text{m}^3$, and the bulk density for the fraction with the grain diameter in the range of $0.315\text{--}5.0 \text{ mm}$ is $1306 \text{ kg}/\text{m}^3$. After consolidating the bulk density of the material increased to the value of $1538 \text{ kg}/\text{m}^3$ (Siwec 2007).

Chalcedonite is a material with a wide range of application in the sanitary engineering. The basic application of the chalcedonite is water treatment, especially ground water. Its high usability unveils in good hydraulic properties of the material which allows obtaining large mass capacities on the filter and long filtration cycles. Many successful introductions were described when chalcedonite substituted classical filters of quartz sand (www.microsil.com.pl b). The structure of the filtration material, which chalcedonite is, its porosity and complex external surface of the grains can help to free development of the biofilm. It enables effective removal of ammonium from the underground water in the process of nitrification on the chalcedonite bed (Sozański et al. 2008). Chalcedonite does not have significant sorptive properties in relation to manganese(II) (Michel 2008a). Modification of its surface with manganese dioxide leads to creation of a chemically active material which shows effectiveness in manganese(II) removing in the second step filters. The usable capacity

of the material prepared in this way was $0.76\text{--}4.0 \text{ g Mn}/\text{L}$ of the bed, depending on the method of modification and the conditions of the research (Michel 2008a; Michel et al. 2008; Michel 2009). However, it can be taken into consideration as a sorptive material to remove oil spills. Regarding its abilities to soak in fuel and rape oil, the chalcedonite sand shows its effectiveness comparable to diatomite and clinoptilolite (Michel 2006).

Surface modification is a popular solution and as manganese dioxide carriers different materials are used, for instance, zeolite (Anielak 2006; Anielak and Schmidt 2011; Han et al. 2007), diatomite (Al-Degs et al. 2000; Al-Degs et al. 2001), montmorillonite (Boonfueng et al. 2005), bentonite (Eren et al. 2009), glauconite (www.purolite.com), quartz sand (Hu et al. 2004; Lee et al. 2004; Han et al. 2006), carbon nanotubes (Ma et al. 2007), aluminium oxide(III) (Maliyekkal et al. 2006) and polymeric fibre (Moore and Reid 1973; Su et al. 2009). In the water treatment technology for manganese(II) removal naturally made and synthetic filtration materials are used containing manganese dioxide in its composition (Kowal and Świdwerska-Bróz 2009). A high, negative surface-bound charge of MnO_2 causes that it is a good sorbent of metals from water solutions. It has sorptive and catalytic properties which are used for water treatment from solved non-organic pollutions (Calderon Rosas et al. 2010). That is why it is a popular material used in the silt form or bound on the carrier.

The aim of this paper was to work out an effective method of manganese dioxide linking on the surface of the chalce-

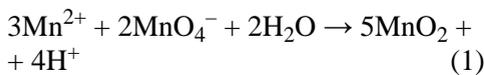
donite sand and to present the characteristic of the obtained material.

EXPERIMENTAL

Sample preparation

Chalcedonite sand used in the studies consisted of 0.8–1.25 mm grains. First it was washed with deionized water several times to remove particulate matter, and then it was dried in air conditions. The material was used to prepare manganese dioxide modified chalcedonite (MDMC). The chalcedonite coating was made using three methods.

The first method of MDMC preparation was similar to the one followed by Zhang et al. (2008). Manganese oxide was synthesized using the reaction of potassium permanganate with manganese(II) sulphate according to the formula:



MnO_2 was precipitated when the reagents were mixed together and the reaction took place until manganese(II) sulphate was completely consumed (excess of potassium permanganate). The 100 g sample of chalcedonite was then placed in the manganese dioxide and potassium permanganate mixture for 24 h at the ambient temperature. The supernatant of this mixture was discarded and the chalcedonite soaked with manganese dioxide was dried in an oven at 100°C to consolidate the coating. The procedure was repeated twice. Next it was washed with deionized water, and then dried at 100°C to prepare the sample for analysis. The

material prepared using this method will be referred to as MDMC-1.

The second method of chalcedonite surface modification was performed identically to modification of diatomite in the work of Al-Degs et al. (2001), which was adopted from the work of Moore and Reid (1973). Chalcedonite after modification was washed with deionized water, and then dried at 100°C to prepare the sample for analysis. The material prepared using this method will be referred to as MDMC-2.

The third method of MDMC preparation was described by Michel and Kiedryńska (2011). This method is similar to the first one but includes MnO_2 precipitation in direct contact with the surface of the mineral carrier and binding with this surface. The finished sample was dried at 100°C just to prepare the material for analysis and MnO_2 was not bound thermally with the surface of chalcedonite. The material prepared using this method will be referred to as MDMC-3. All solutions were prepared with redistilled water and reagent-grade chemicals.

Material identification

The samples of modified chalcedonite and their equivalents after the process of manganese(II) sorption were used as the material for the analysis. Sorption of the manganese(II) was conducted in the flow conditions, and the sorptive was prepared from the manganese(II) chloride and the redistilled water. The shape and elemental composition of the exterior surface of samples were analyzed using the SEM-EDS technique (scanning electron microscope by LEO Co. Ltd. fitted with an energy dispersive X-ray spec-

trum detector by Oxford Instruments Ltd.). The samples consisted of 0.8–1.25 mm grains and were not polished. Before analysis, they were coated with a thin layer of gold.

RESULTS AND DISCUSSION

Chalcedonit sand used for the research does not have a homogeneous grain structure. Chalcedonite is found in the deposit in two forms, that is why there are grains with a porous structure and an extensive external surface and solid grains with small surface development. More specific information on the topic is presented in the work of Michel (2011). The effects of the surface modification of chalcedonite in the current article is shown by the example of grains of porous variety.

The first method of impregnation which was based on thermal bound of the oxide with the surface of chalcedonite led to production of the grains thickly covered by the oxide. It is presented in Figure 1, which shows the overall view of the grain. On the MDMC-1 surface the layer is seen in which with the help

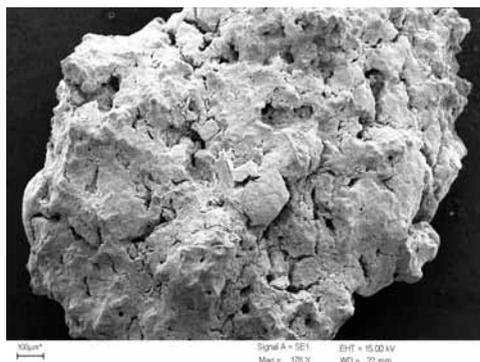


FIGURE 1. The SEM micrograph of the MDMC-1 grain

of the EDS analysis the present of manganese coming from MnO_2 was proved (Fig. 2). The large thickness of the coat causes that the spectral indication of silicon which comes from the bed was not observed. On the other hand, the obtained coat is not continuous and on its part it is subjected to exfoliation, which is clearly seen in Figure 3. The consequence of it was that in organoleptic observations the MDMC-1 material is left loose, pulverous particles of MnO_2 come off the surface of the grains. The grains of the material

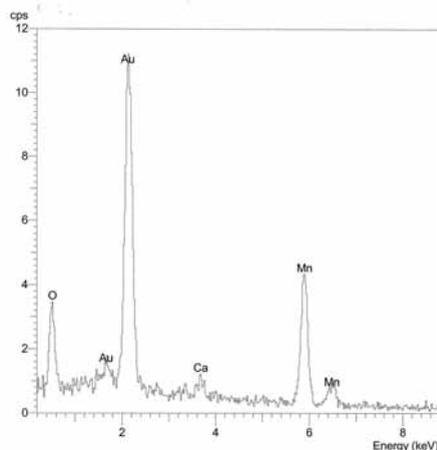


FIGURE 2. The EDS spectrum of the MDMC-1 coat

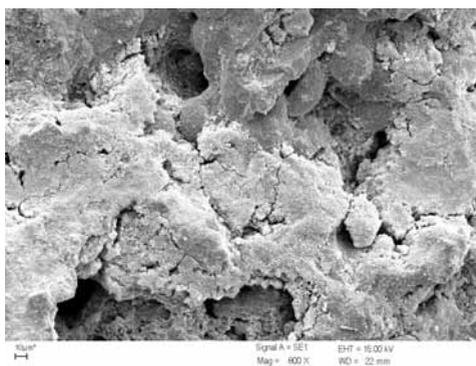


FIGURE 3. The SEM micrograph of the exfoliated MDMC-1 coat

were of brown colour with visible clear parts in which the carrier was revealed. The coat made using the first method was characterized by low durability. MnO₂ had a grain structure (Fig. 4) which is very similar to the surface of quartz sand coated with manganese oxide obtained by Han et al. (2006) using another method of impregnation. Modification of chalcedonite using this method causes an increase in its specific surface to the value of 8.36 m²/g (Michel 2008b). Sorption of the manganese(II) on MDMC-1 did not cause significant changes in the appearance of the coat (Fig. 5).

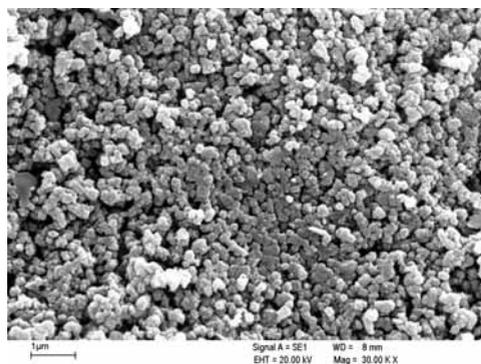


FIGURE 4. The SEM micrograph of the MDMC-1 coat grain structure

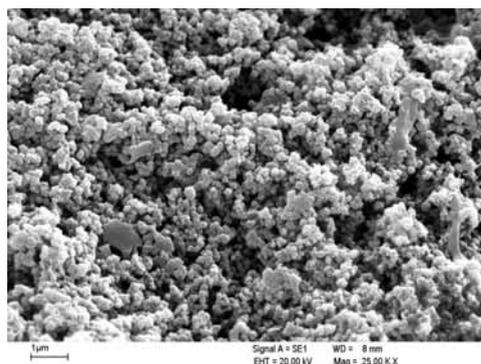


FIGURE 5. The SEM micrograph of the MDMC-1 coat structure after manganese(II) sorption

Chalcedonite modified using the second method was characterized by dark, black-brown colouring. The obtained coat was continuous. Figure 6 presents the overall view of the grain of the MDMC-2 material. Interstices and cavitations on the chalcesonite surface are filled with MnO₂. The EDS analysis of the obtained layer shows strong spectral indication coming from manganese and weak indication of sodium which can be the remains of the preparation (Fig. 7).

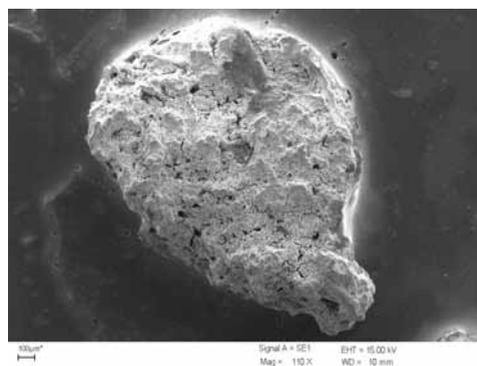


FIGURE 6. The SEM micrograph of the MDMC-2 grain

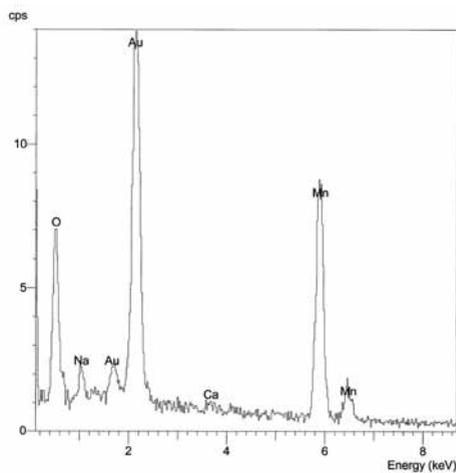


FIGURE 7. The EDS spectrum of the MDMC-2 coat (Michel et al. 2008)

The coat has an irregular and crater structure (Fig. 8), which probably causes an increase in the specific surface area of the modified material which is $10.25 \text{ m}^2/\text{g}$ (Michel et al. 2008) in relation to the carrier surface which is $6.13 \text{ m}^2/\text{g}$. When enlarged the plate-like structure of the oxide is seen (Fig. 9). After the process of sorption of manganese(II) on the MDMC-2 material, it was observed that the plates that appeared in the structure were larger and more distinct (Fig. 10). Al-Degs et al. (2001) follow Golden et al. (1986) that the plate-like crystals are typical of birnessite. It is then highly probable that the second method of im-

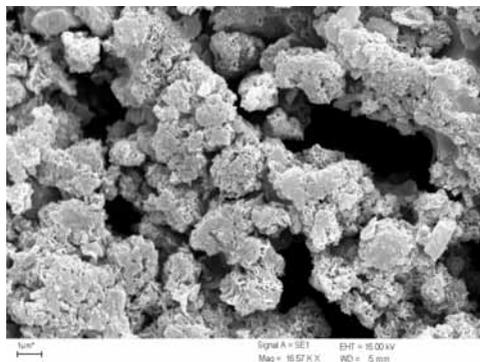


FIGURE 8. The SEM micrograph of the MDMC-2 coat crater structure

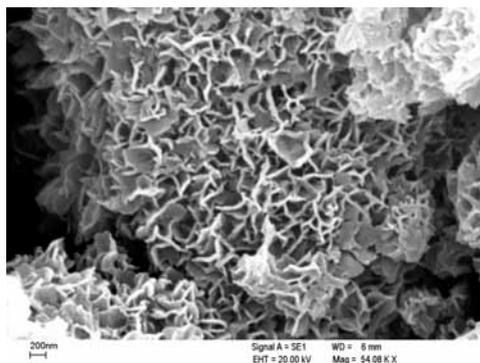


FIGURE 9. The SEM micrograph of the MDMC-2 coat plate-like structure

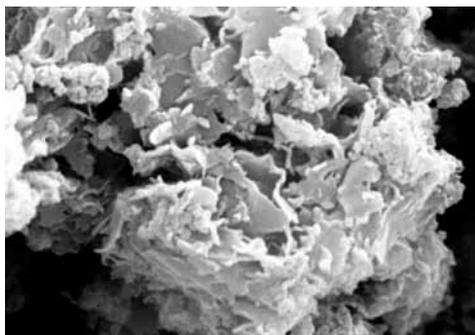


FIGURE 10. The SEM micrograph of the MDMC-2 coat plate-like structure after manganese(II) sorption

pregnation leads to appearance of exactly this kind of MnO_2 on the chalcedonite.

The third method of modification of chalcedonic MnO_2 leads to appearance of the brown, continuous coat which coats the whole grain surface (Fig. 11). The grain is not too much pasted over by the oxide whose larger concentration takes place only in the interstices (Fig. 12). In the organoleptic observations the MDMC-3 material is coherent and the obtained layer does not fracture. This effect was reached by precipitation of MnO_2 in the direct contact with the carrier's surface. MnO_2 has a grain structure seen in Figure 13. The EDS spectrum

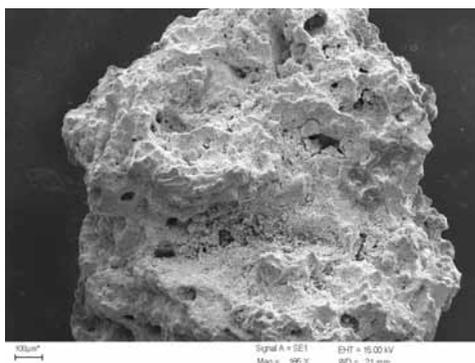


FIGURE 11. The SEM micrograph of the MDMC-3 grain



FIGURE 12. The SEM micrograph of the MDMC-3 coat structure

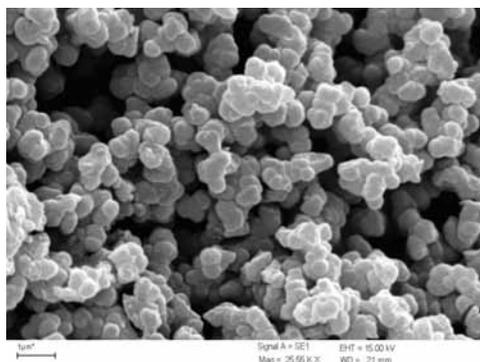


FIGURE 13. The SEM micrograph of the MDMC-3 coat grain structure (Michel 2008a)

of the MDMC-3 surface is presented in Figure 14. The spectral indication of manganese coming from the MnO_2 coat and the spectral signal of silicon were observed too. It comes from chalcodonite (SiO_2) and may testify of a small thickness of the obtained coat. As a result of chalcodonite coating by MnO_2 an increase in its specific surface area takes place. Its value of $6.13 \text{ m}^2/\text{g}$ increases to $9.88 \text{ m}^2/\text{g}$ (Michel 2008a). Sorption of manganese(II) ions causes a change in the appearance of the MDMC-3 material (Fig. 15). Manganese oxides in the form of MnO_2 and MnO were identified on its surface (Michel 2008a). It is necessary

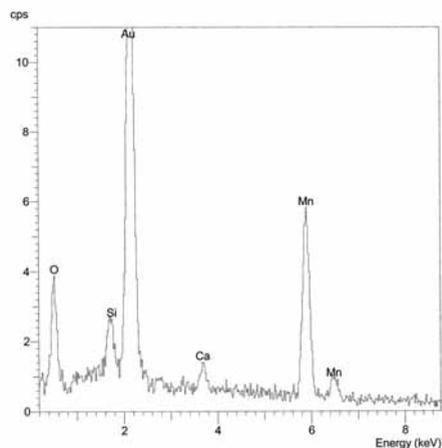


FIGURE 14. The EDS spectrum of the MDMC-3 coat

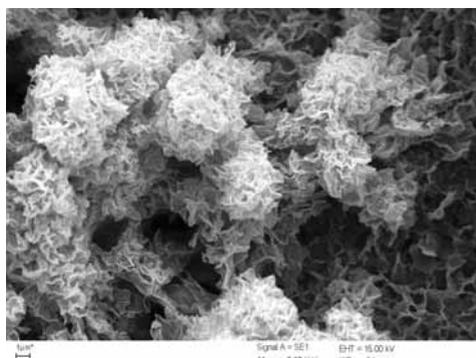


FIGURE 15. The SEM micrograph of the MDMC-3 coat structure after manganese(II) sorption (Michel 2008a)

to point out that the grains of MDMC-3 as well as MDMC-1 and MDMC-2 after the process of manganese(II) sorption were not covered with biofilm.

The value of the specific surface of the MDMC-1, MDMC-2 and MDMC-3 materials is typical of this type of materials. For comparison, quartz sand modified with MnO_2 can be characterized by the specific surface of $9.03 \text{ m}^2/\text{g}$ (Lee et al. 2004), $1.99 \text{ m}^2/\text{g}$ (Hu et al. 2004) and $0.712 \text{ m}^2/\text{g}$ (Han et al. 2006), whose

variable values follow from the preparation of the material and the type of the carrier. The increase in the surface of the material as a result of modification of the carrier with MnO_2 was also observed by other authors (Al-Degs et al. 2001; Han et al. 2006).

To sum it up, the used methods of impregnation allow making the modified material of different quality. The weakest effect was obtained when preparing MDMC-1 material with the thermal method. In comparison, the MDMC-2 and MDMC-3 material are much more durable. The MDMC-3 material is worth paying special attention at because an increased temperature is not used for its preparation (the increased temperature was used in order to prepare the sample with the constant moisture parameters). Taking into account the preparation of the modified chalcedonite in technological scale the second impregnation method is more complicated than the third one as the use of hot NaOH requires special security safeguards. Additionally, it is possible to carry out the modification of the filtrating material by the third method in the flow conditions.

CONCLUSIONS

Chalcedonite is a very good manganese dioxide carrier. The modification of the chalcedonite surface changes its chemical composition structure. The value of the specific surface of chalcedonite increases as a result of the modification in the MDMC-1 < MDMC-3 < MDMC-2 line. The coat morphology and structure depends on the applied impregnation method. The MDMC-3 material was

considered to be the optimal regarding the strength of the coat and the easiness of its production.

The chalcedonite sand was used as a silica carrier for manganese oxides. The suggested methods of coating of chalcedonite by MnO_2 can be potentially applied to other filtrating materials such as diatomite, clinoptylolite or pumice.

Chalcedonite is a very useful material in the water treatment technology. It can be easily affected by coating with an oxide layer; moreover its gains have a meso- and macroporous structure and irregular surface what corresponds to good filtrating properties of this material. It was stated on the manufacturer's website in the form of letters of reference from different water works plants. That is why chalcedonite sand is worth being looked at as a filling of filtering and chemically active beds, especially as its resources for exploitation are enough to be exported and the price is comparable to the price of quartz sand used for filtration.

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Streszczenie: *Badania nad zastosowaniem chalcedonitu jako nośnika tlenku manganu.* Chalcedonit jest osadową skałą krzemionkową, która występuje w Polsce w czterech złożach i jest zaliczana do grupy skał unikalnych. Chalcedonit jest wykorzystywany w technologii uzdatniania wody głównie jako efektywny materiał filtracyjny, na co wpływa jego mezoporowata struktura oraz rozwinięta zewnętrzna powierzchnia ziaren. W artykule zaprezentowano trzy metody modyfikacji materiału mineralnego (MDMC-1, MDMC-2, MDMC-3) za pomocą tlenku manganu(IV). Chalcedonit był zastosowany jako nośnik tlenku. Zaprezentowane zostały efekty modyfikacji powierzchni chalcedonitu identyfikowane techniką SEM-EDS. Stwierdzono, że chalcedonit jest bardzo dobrym nośnikiem tlenku manganu(IV) a modyfikacja jego powierzchni zmienia jej skład chemiczny, strukturę oraz wielkość powierzchni właściwej.

Słowa kluczowe: chalcedonit, skała krzemionkowa, tlenek manganu(IV), materiały modyfikowane.

MS. received May 2012

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