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CALCINATION OF NICKEL MUD

Michaela KLAČANSKÁ, Hana KOBETIČOVÁ, Maroš SOLDÁN,
Marián HARŠÁNI, Marcel KURACINA

SLOVAK UNIVERSITY OF TECHNOLOGY IN BRATISLAVA,
FACULTY OF MATERIALS SCIENCE AND TECHNOLOGY IN TRNAVA,
INSTITUTE OF INTEGRATED SAFETY,
ULICA JÁNA BOTTU 2781/25, 917 24 TRNAVA, SLOVAK REPUBLIC
e-mail: michaela.klacanska@stuba.sk, hana.kobeticova@stuba.sk, maros.soldan@stuba.sk,
marian.harsani@stuba.sk, marcel.kuracina@stuba.sk

Abstract

Different characterizations were carried out on uncalcinated nickel mud and samples calcined in the temperature range of 200 – 600 °C. In the present paper, the phase composition and structure transition of the nickel mud heated from room temperature are indicated by SEM, EDX analysis and Fourier transform infrared spectroscopy. Samples of nickel mud were collected in Sereď, where nickel was produced from Albanian iron-nickel ore. It was found that calcination has effect on the structure and composition of nickel mud. The obtained results provide an important base for the further studies of comprehensive utilization of nickel mud.

Key words

nickel mud, calcination, SEM, EDX analysis, Fourier transform infrared spectroscopy

INTRODUCTION

Calcination is a thermal treatment process of solids by heating materials at a very high temperature. Usually it is conducted in the presence of air or nitrogen gas. This action initiates the repulsion of some volatile materials such as water, organic materials, and carbon dioxide from the sample without any fusion. Sometimes calcination is used to change the physicochemical properties or the constitution of materials (changing phase or changing metal to metal oxide). The calcination temperature is a central parameter in the synthesis of photocatalysts and can play a critical role in controlling their physicochemical properties and photocatalytic activity (1).

Nickel mud was formed by leaching nickel and cobalt from lateritic iron-one. Nickel mud contains chromium oxide, silicon, aluminium, calcium and the rest of nickel is essentially iron concentrate. In the Slovak Republic, nickel was produced from the Albanic iron-nickel ore with a nickel content of about 1% (2).

Nickel mud has a good catalytic activity. Its catalytic activity is affective by the principle of adaptive time constant, surface of area, adsorption of nickel mud, distribution of pore and crystalline structure (3).

Calcination methods were used by a lot of researchers (4, 5, 6). They investigated various types of materials, e.g. red mud, Fe-Ni particles, Fe-TiO_2 and others. In this study, we focused on the nickel mud reserves in Slovakia.

MATERIALS AND METHODOLOGY OF EXPERIMENT

The nickel mud used in this study was obtained from the Albanic iron-nickel in Sered'. For topographic and elemental analysis of nickel mud, we used scanning electron microscope (SEM), EDX analysis and the Fourier transforms infrared spectroscopy (FTIR).

EDX analysis was used for determine chemical composition, concentration of individual elements and identification of reaction products. It was performed on the equipment of energy-dispersive X-ray spectroscopy analyser which was a part of electron scanning microscope of JEOL JSM 7600 F type. Topography of nickel mud was observed at an accelerating voltage 20 kV, current 2 nA and working distance approximately 15 mm. Chemical composition of nickel mud was investigated by software INCA at the magnification of 1 000 times.

Calcination was performed in a muffle furnace at different temperatures (200 – 600 °C) for 5 hrs under static air after crushing the dried nickel mud. Calcination of nickel mud was analysed by FTIR. FTIR was used to investigate the effect of different calcination temperatures on the nickel mud photoactivity. FTIR spectrum resulted from the interaction between IR light that penetrated into thin layers of surfaces of nickel mud and chemical composition of the nickel mud.

RESULTS

EDX analysis shows that nickel mud contains higher percentages of Fe and Al. These elements positively influence the catalytic and sorption properties of nickel mud. Results of EDX analysis are summarized in Table 1. Detail of the surface of nickel mud is in Fig. 1. The areas where the measurements were processed are in Fig. 2.

Table 1 Elemental analysis of nickel mud in wt. %.

Spectrum	O	Al	Si	P	S	Cr	Mn	Fe	Ni	Cu
1	26.72	0.14	0.31	0.05	0.08	0.77	0.99	70.49	0.13	0.32
2	33.60	0.07	0.37	0.03	0.09	1.30	1.01	63.07	0.22	0.24
3	33.12	0.13	0.32	0.04	0.06	1.52	1.03	62.47	0.86	0.45
4	30.18	0.09	0.34	0.08	0.08	0.64	1.05	66.75	0.43	0.36

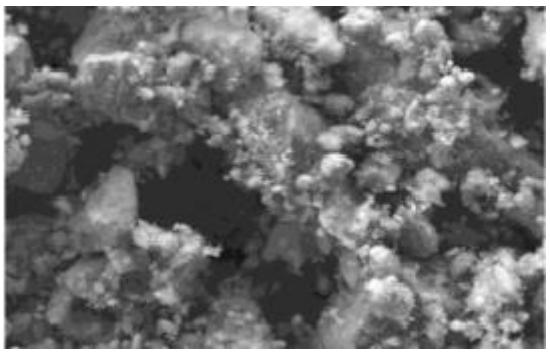

Fig. 1 *SEM of nickel mud (fraction < 500 μm)*
Detail of the surface at magnification of 1 000 times

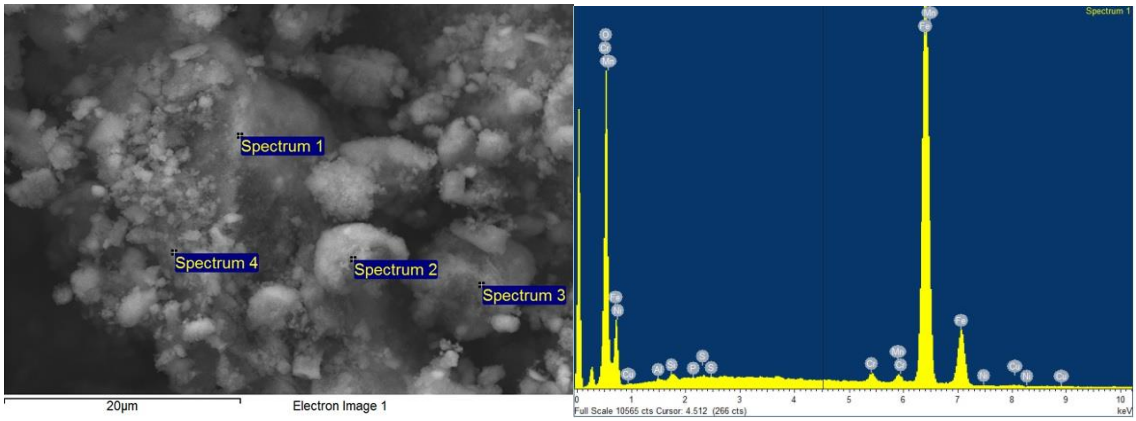

Fig. 2 *SEM-EDX analysis: nickel mud chemistry*

Fig. 3 shows the FTIR spectrum of the nickel mud, calcined at 200, 400 and 600 °C, respectively.

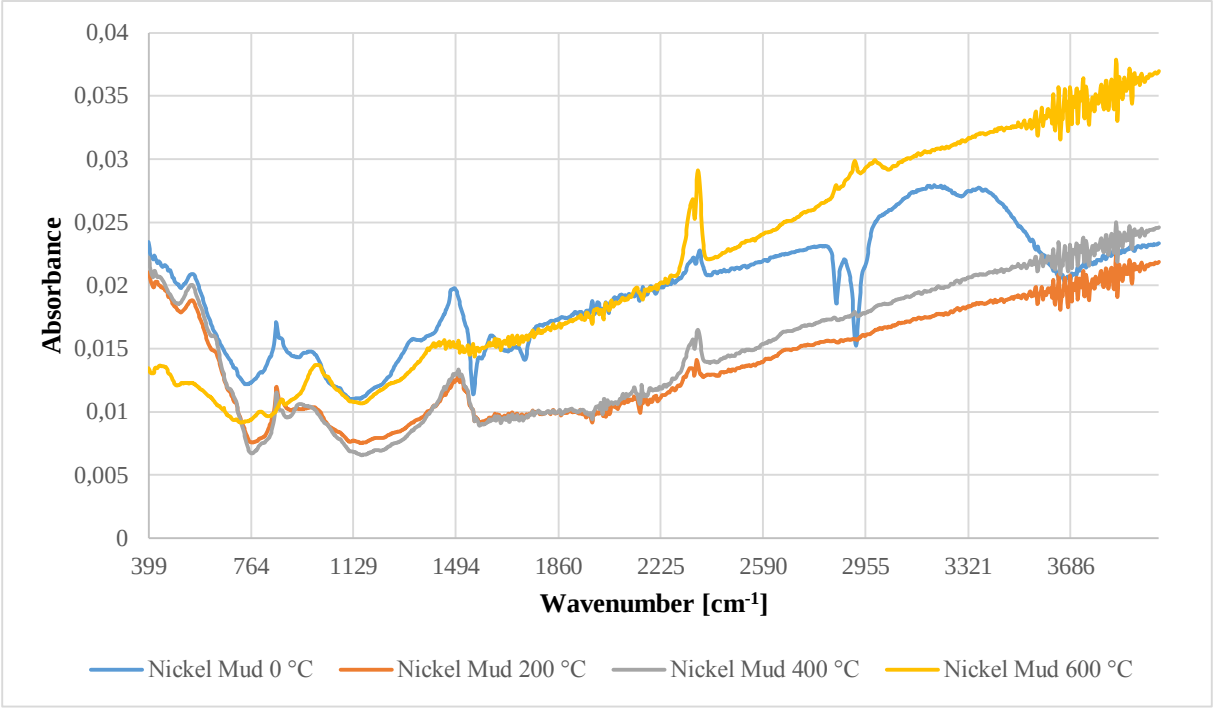

Fig. 3 *Effect of calcination temperature on nickel mud*

In the FTIR spectra of nickel mud, we can observe an intensive band at 1500 cm^{-1} corresponding to the O-H bonds in the bound water molecule. After the thermal treatment, the intensity of the peak decreases. We can observe a smaller absorbent band at 870 cm^{-1} corresponding to Al-O bonds. As a result of the increased temperature, these bonds degrade, resulting in a decrease of the peak. Weak absorption peak at 530 cm^{-1} represents O-Si-O vibrations. Its intensity does not change after the thermal treatment. The smaller absorbent band at 440 cm^{-1} corresponds to the Fe-O bond, and its intensity does not change after annealing. The absorbent band at 2300 cm^{-1} corresponds to binding in the CO_2 molecule, the intensity of this peak increases with increasing temperature.

DISCUSSION

Similar study of authors from China (7) studied the properties of red mud by SEM. The authors found that red mud calcined at a series of temperatures 150 °C to 1200 °C, which indicates that the heat treatment can improve the value of particle diameter and make the particles easy to gather with each other. Different microstructures result from different physical and chemical progresses. With the influence of heating at 150 °C, red mud loses the majority of its physically absorbed water and part of chemically bound water. Therefore red mud can have a gradually increasing value of density.

The UV absorption spectra of calcined Fe–TiO_2 at various temperatures were studied in the article (8) of Thailand authors. The physical and chemical properties of catalysts, crystalline phase and size, surface area, morphology, and absorption properties were affected by the calcination temperature. The photocatalytic reaction followed the first-order kinetics. The calcined photocatalyst at 500 °C provided the highest activity of MO degradation under visible light. This implied that the synthesized catalysts prepared at mild calcination temperature showed higher photocatalytic activity than that calcined at higher temperature (>500 °C).

The morphological changes of Fe–Ni catalyst for the preparation of carbon nanofibre and the calcination of Fe–Ni carbonate into Fe–Ni oxide were investigated in the study (9). The calcination provided at 400 °C the size of the resultant Fe–Ni oxide aggregate to several micrometres and kept the size of its component particles to less than 10 nm. The evolution of CO_2 in the calcination decreased the size of resultant Fe–Ni oxide into several micrometres.

FTIR analysis of red mud uncalcinated and calcined within the interval 400–900 °C was used in the study (6). Calcination temperature has a significant effect on the cementitious activity of red mud owing to the phase transitions during the calcination process. It was found that the red mud calcined at 600 °C in this study had the best cementitious activity, which was due to the formation of poorly-crystallized Ca_2SiO_4.

CONCLUSION

Nickel mud can be probably used as a catalyst or sorbent in various areas of industry. And therefore, nickel mud was investigated for its properties. The current paper dealt with calcination at the temperature range of 200 – 600 °C. For analysis of composition of nickel mud, we used the SEM and EDX analysis. FTIR analysis was used to investigate chemical bonds in dependence on calcination.

Finally, the following significant pieces of knowledge may be summarised:

- EDX analysis has shown that nickel mud consist mainly of Fe (62.47 %), O (33.12 %), Cr (1.52 %), Mn (1.03 %), Ni (0.86 %) and small concentration of other elements.
- in FTIR spectra of nickel mud we found 3 bonds:
  - O-H bonds at 1500 cm^{-1} –> after the thermal treatment, the intensity of the peak decreases.
  - Al-O bonds at 870 cm^{-1} –> increased temperature causes decrease of the peak.
  - Fe-O bonds at 440 cm^{-1} –> intensity of peak does not change after annealing.
- Also O-Si-O vibrations at 530 cm^{-1} were observed.
- Binding in the CO_2 molecule causes absorbent band at 2300 cm^{-1}.

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ORCID:

Michaela Klačanská	0000-0002-2425-4326
Maroš Soldán	0000-0003-1520-1051
Marián Haršáni	0000-0002-3283-6414