The Application of Microwave Energy in Mineral Processing – a Review

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This paper presents a review of the application of microwave energy in the field of mineral processing at the Institute of Geotechnics, Slovak Academy of Sciences. The influence of microwave radiation of rocks on the failure disintegration of minerals and ores as well as, a modification of magnetic properties and thermal processing of rocks was studied. Moreover, the rate of microwave heating of minerals, effect of microwaves on coal desulphurisation and the extraction of diterpanes from coal were investigated.

Keywords: *microwave, complex permittivity, mineral processing, coal, minerals*

Introduction

In 2000, Dr. Ricky Metaxas, the president of Association for Microwave Power in Europe for the Research and Education has written about the radio frequency and microwave heating as a perspective for the millennium (Metaxas, 2000). Most recently, in the field of research and application of microwave energy in mineral processing, a series of papers describing microwave heating of various minerals, modification of physical properties, extraction processes and innovation of mineral processing technology have been published in the world
(Kingman et al., 2004; Kingman, Jackson et al., 2004; Al-Harahsheh and Kingman, 2004; Huang and Rowson, 2002; Havlík et al., 2001; Uslu and Atalay, 2004; Sobek, 2008). The research in the microwave energy application at the Institute of Geotechnics of the Slovak Academy of Sciences has begun in 1993. It is focused on the treatment of Slovak ores and coal. In frame of cooperation with the Faculty of Aeronautic, Technical University in Košice, a technique for the measurement of powder materials dielectric properties was elaborated. The research on the use of microwave heating has been undertaken in these main areas: the measurement of dielectric properties, modelling of microwave heating, drying of minerals and coals, improving of the grindability of materials, influence of microwave on changes in magnetic properties, removal of sulphur from coals, microwave extraction of organic compounds from coal, and the leaching of materials and microwave vitrification of waste.

Basic concept of microwave heating

Microwaves cause molecular motion by migration of ionic species and rotation of dipolar species. Microwave heating of a material depends to a great extent on its dissipation factor. This is the ratio of dielectric loss or loss factor to dielectric constant of the material. The dielectric constant is a measure of the ability of the material to retard microwave energy as it passes through; the loss factor is a measure of the ability of the material to dissipate the energy. In other words, loss factor represents the amount of input microwave energy that is lost in the material by being dissipated as heat. Therefore, a material with a high loss factor is easily heated by microwave energy. An important characteristic of microwave heating is the phenomenon of 'hotspot' formation, where by regions of very high temperature form due to non-uniform heating. Microwave energy is extremely efficient in the selective heating of materials as no energy is wasted in 'bulk heating' of the sample. This is a clear advantage that the microwave heating has over conventional methods.

Dielectric properties of materials

Knowledge of a material dielectric property is critical for proper design of microwave applicators. The measurement of dielectric properties is not an easy task and it requires specialised techniques. The resonant cylindrical cavity technique is based on this simple perturbation theory. It assumes that the change in the stored energy in the cavity between the loaded and unloaded conditions is very small. This means that the electromagnetic fields in the cavity with and without the sample must be approximately equal. Therefore, the dimensions of the sample must be small compared with the size of the cavity resulting in a small frequency shift. In addition, the symmetry of the sample within the cavity is also necessary.

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The effect of temperature on the measurement of real and imaginary parts of permittivity was realized by the resonance cavity method in the TM030 mode at the frequency of 2216 MHz. The measurements of permittivity of minerals were made at the Faculty of Engineering, School of Electrical & Electronic Engineering, University of Nottingham (Lovás et al., 2010).

Dielectric properties of the measured samples were calculated from the change in frequency f and Q-factor (Q = 1/tanδ), measured before (in an empty fused silica tube) and after sample loading. For the calculation the following equations were used:

$$\varepsilon' = 1 + 2.J_1^2(x_{1,n}).\frac{V_c}{V_s}.\frac{f_o - f_s}{f_o} \quad , \quad \varepsilon'' = J_1^2(x_{1,n}).\frac{V_c}{V_s}.\left(\frac{1}{Q_s} - \frac{1}{Q_o}\right) \tag{1}$$

f_o - resonant frequency of the empty cavity [s^{-1}]; f_s - resonant frequency of the cavity with a sample [s^{-1}]; J_1 - root of the Bessel function of the first kind; V_c - volume of the cavity [m^3]; V_s - sample volume [m^3]; Q_o - quality factor of the empty cavity; Q - quality factor of the cavity with a sample; ε' - relative dielectric constant; ε'' - relative effective dielectric loss factor.

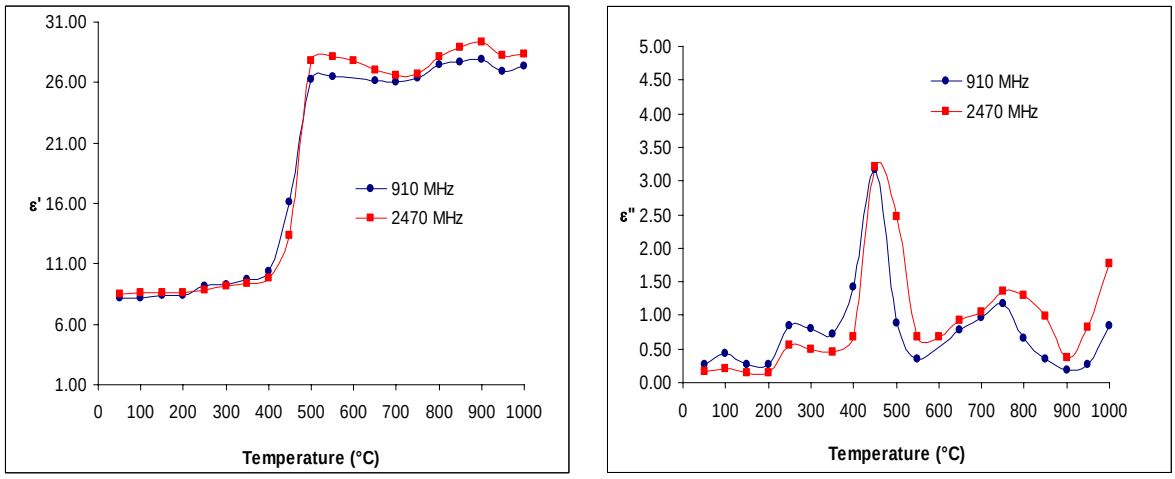

Fig. 1. The dependence of the real (ε) and imaginary (ε parts of the permittivity of chalcopyrite on the temperature and frequency.

The maximum value of real permittivity was found at 500 °C and for imaginary permittivity at 450 °C, after that the real part decreases slowly until 800 °C where it increases again and the imaginary part decreases rapidly until

800 °C, where it exhibits a new peak. These results correlate with the forming of new phases (Lovás et al., 2010).

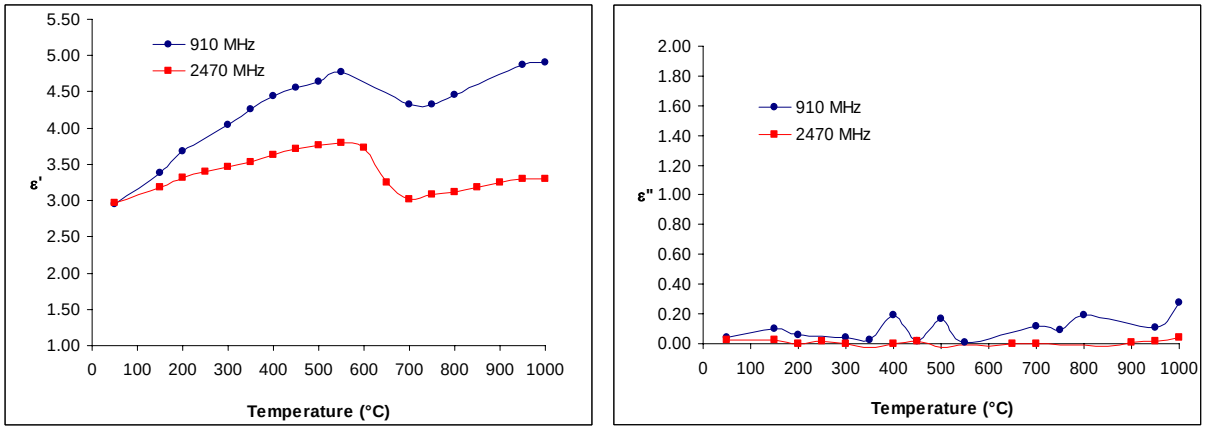

Fig. 2. The dependence of the real (ε) and imaginary (ε parts of the permittivity of magnesite on the temperature and frequency.

The decrease of imaginary permittivity ε'' occurs at a temperature of 180 °C and it is possible that be related to water evaporation. The value of imaginary permittivity increases at 950 °C. It is connected with its

thermal decomposition of $MgCO_3$ to MgO. The real permittivity grows until 600 °C, afterwards the values decrease (CO_2 loss).

Modelling of the microwave heating of materials

Microwave heating entails the conversion of electrical energy to heat either to raise the material temperature. Mathematical modelling of such processes gives a better insight into the physical phenomena, determines relations between the characteristics of the applied microwave radiation and the structure of the material. This establishes optimal heating regimes for material processing. The complex dielectric permittivity describes the behaviour of the material in the microwave field. The imaginary component is proportional to the size of permittivity dielectric loss and can be used as a criterion for assessing the suitability of heating in the microwave oven. Both components of the dielectric permittivity significantly affect the microwave heating process. The heating of substances often leads to significant changes in values of real and imaginary components of complex permittivity.

On the basis of the parameters using Comsol Multiphysics, it was modelled heat insulated sphere of radius 3 cm in the microwave oven in the performance of 1000 W, frequency 2.45 GHz. The model is set up in two steps: firstly, the wave propagation problem is solved, secondly, the heat transfer problem is evaluated. The model is illustrated for the samples of magnesite and chalcopyrite heated up to 20 s. Distribution of temperature is indicated in Fig. 3-4.

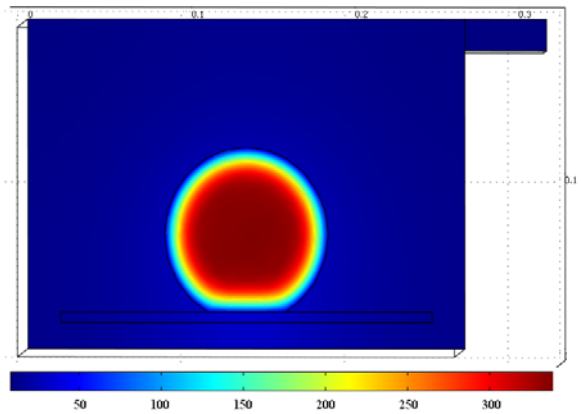

Fig. 3. The distribution of temperature at microwave heating of magnesite.

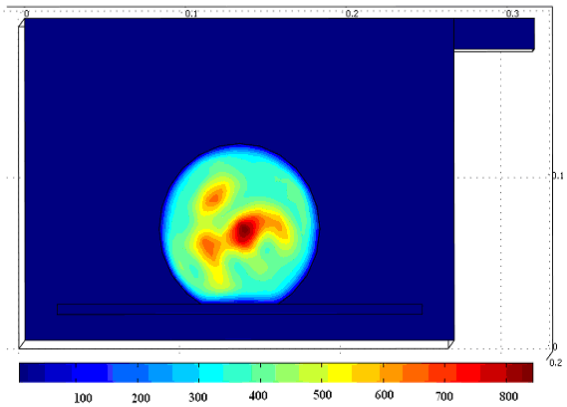

Fig. 4. The distribution of temperature at microwave heating of chalcopyrite.

As it is shown in Figs 3-4 microwave heating propagates from inside to outside. The results of our experiments confirmed that the chalcopyrite sample is heated faster than magnesite.

Microwave heating of minerals

The minerals were separated into 20 g samples with a particle size of 0.2 - 0.5 mm. The samples were irradiated in a domestic microwave oven Whirpool AVM 434 with output of 900 W and at frequency 2.45 GHz. Table 1 shows the test results. The samples were heated during 1 and 5 minutes, respectively.

Tab. 1. Microwave heating of minerals.

Mineral	Locality	Temperature [°C]	
		1 min	5 min
Baryte ($BaSO_4$)	**Rudňany (Slovakia)**	69	107
Galena (PbS)	**Banská Štiavnica (Slovakia)**	741	-
Hematite (Fe_2O_3)	**Hačava (Slovakia)**	118	423
Chalcopyrite ($CuFeS_2$)	**Slovinky (Slovakia)**	780	-
Chromite ($FeCr_2O_4$)	**Turkey**	85	218
Quartz (SiO_2)	**Švedlár (Slovakia)**	140	175
Magnetite (Fe_3O_4)	**Krivoj Rog (Ukraine)**	547	-
Magnesite ($MgCO_3$)	**Lovinobaňa (Slovakia)**	82	128
Pyrhotine (FeS)	**Zlatá Baňa (Slovakia)**	290	-
Pyrite (FeS_2)	**Hnúšťa (Slovakia)**	670	-
Sphalerite (ZnS)	**Banská Štiavnica (Slovakia)**	176	192
Siderite ($FeCO_3$)	**Rudňany (Slovakia)**	152	227
Tetrahedrite ($Cu_{12}Sb_4S_{13}$)	**Rožňava (Slovakia)**	413	-

- not measured

On the basis of the heating rate, irradiated minerals were divided into weakly heated minerals such as quartz, barite and well heated minerals, for instance chalcopyrite, pyrite, galena, magnetite and siderite.

Microwave drying of minerals and coal

The rate of drying of selected granular minerals was studied (Fig. 5). Very fast drying of magnetite and galena was observed. However, rapid and sufficiently effective drying of siderite and in particular of quartz was also monitored.

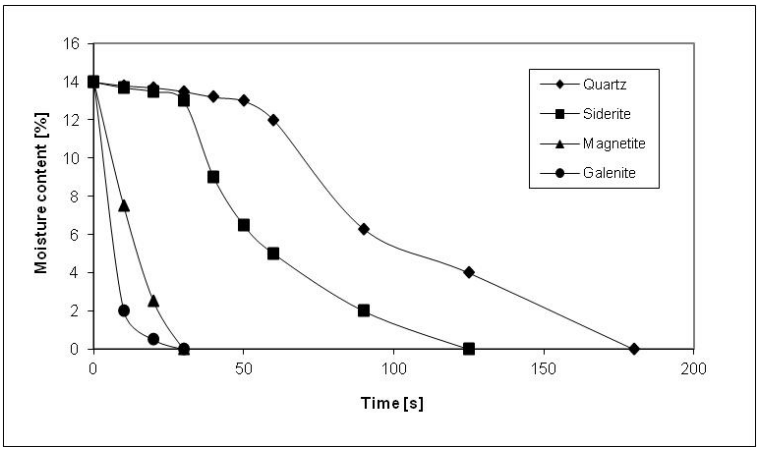

Fig. 5. The microwave drying of selected minerals (grain size – 40 μm).

The microwave drying of minerals depends most of all on their permittivity and their grain size (Florek and Lovás, 1995).

Microwave heating of coal was performed on the samples of brown coal from Handlová mine in a microwave furnace with the output of 500 W. The decrease of moisture on the time of drying was followed. The results are shown in Fig. 6.

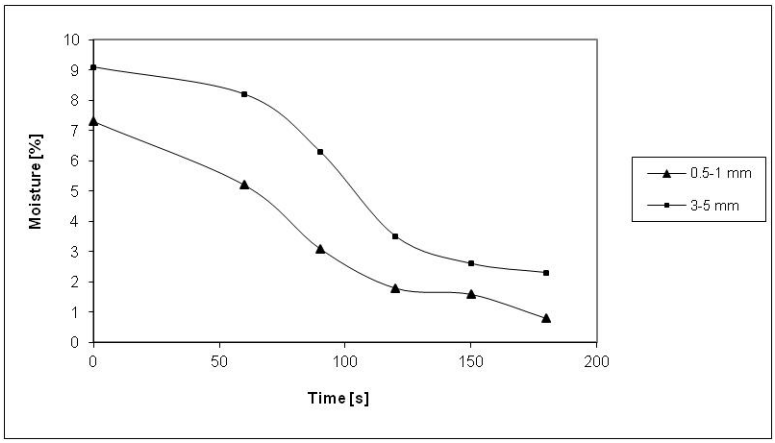

Fig. 6. The drying of brown coal in a microwave furnace.

The advantage of drying in a microwave furnace is mainly drying velocity that is 10-times higher in comparison with a classical drying (Florek and Lovás, 1995). Classical drying process runs from the surface of a sample to the middle and depends on thermal conductivity that is low in the case of coal. Heat and water transport moves in an opposite direction. The speed of drying is limited by the velocity of water diffusion to the surface. Microwave drying has completely different mechanism then classical drying by hot air, because drying runs from the middle to the surface. The "microwave inner heat" creates pressure gradient that efficiently "pumps water" to the surface from where is fast taken by air flow.

The application of microwaves in a thermally assisted liberation

The rapid heating of ore containing microwave energy absorbing minerals in a non-absorbing gangue matrix generates thermal stress. This thermal stress causes microfracturing along the mineral grain boundaries; as a result, such an ore sample becomes more amendable to grinding. An interesting feature is the selectivity of microwave heating in the case of irradiation-containing materials that absorb and transmission components. Microwave radiation penetrates the outer layer of material transmission (A) virtually no heat and it interacts with heated absorbent (B) (Fig. 7). Gradually, the heated material (B) the influence of thermal conductivity of the heated material (A).

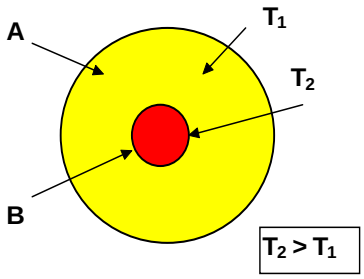

Fig. 7. Microwave heating of heterogeneous material A - transmitting material, B - material heated in a microwave furnace, T_1 - temperature of transmitting material, T_2 - temperature of a material heated in a microwave furnace.

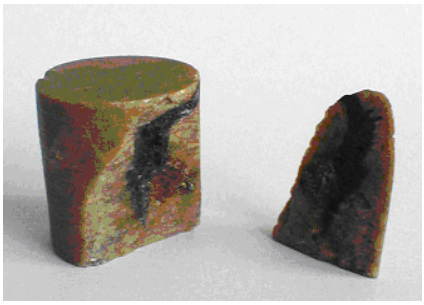

Fig. 8. The disintegration of a siderite roller (light colour) overgrown by a thin layer of chalcopyrite (dark colour) in a microwave furnace.

Selective heating of heterogeneous materials is not possible using conventional heating methods. As an example of selective heating of the microwave heating, a siderite roller overgrown by a thin layer of chalcopyrite was used. After inserting the roller into the microwave oven, the siderite is slowly heated. Microwaves penetrate into the volume and interact with a layer of chalcopyrite, which will heat up very quickly. The roller decomposed into two parts, since the creating of tense caused by rapid increase of temperature during intense heating of chalcopyrite (Fig. 8.).

In the first stage of disintegration, specimens irradiated or non-irradiated with microwaves were after determination of deformation coefficient crushed in a jaw crusher, and in the second stage ground for 20 min in a vibrating grinder under constant conditions. The grain size curves of the specimens obtained after grinding allowed us to determine the maximum size of grains at 80 % yield. The influence of irradiation with microwaves on grindability of specimens was evaluated on the basis of Berry-Bruce comparative test derived from the Bond's work index. The Berry-Bruce test enables to compare grindability of specimens irradiated and non-irradiated by microwaves.

$$RWI = \left(\frac{10}{\sqrt{P_1}} - \frac{10}{\sqrt{F_1}} \right) \Big/ \left(\frac{10}{\sqrt{P_2}} - \frac{10}{\sqrt{F_2}} \right), \qquad (2)$$

RWI - relative working index, F – mesh size of sieve at 80 % passing of feed grains [μm], P – mesh size of sieve at 80 % passing of final (comminuted) product grains, 1- non-irradiated specimen, 2 – microwave irradiated specimen

After the disintegration process, a relative working index was calculated for individual specimens according to (2). The presented values (Tab. 2.) suggest an improvement in grindability that was manifested in the decrease of values of the relative working index after microwave heating.

Tab. 2. Relative working index of grindability of andesite specimens subjected to microwave pre-treatment.

Time of microwave heating [min]	**Relative Work Index**
	grinding 20 min
0	1
1	0.97
3	0.91
5	0.85
7	0.82

After 7 min of irradiation, a high degree of failure was reached. This was confirmed by improved grindability and a decrease in the relative working index by 18 % in comparison with the non-irradiated specimen (Znamenáčková et al., 2002).

Intensification of magnetic separation by microwave radiation

Magnetic separation processes utilize the differences in magnetic properties of the components of treated ores. The separation of weakly magnetic iron ores is limited by the magnitude of magnetic force that can be developed in high intensity magnetic separators. From an economic aspect, the use of low-intensity magnetic separators is more effective but they require the modification of magnetic properties of weakly paramagnetic ores and minerals. The increase of magnetic susceptibility of mineral components can be achieved by phase conversion, induced, for example by magnetic roasting, chemical or electrochemical pre-treatment, irradiation with accelerated electrons, or by the application of microwave energy. Microwave heating (roasting) appears to be a progressive treatment that can ensure the thermal decomposition of iron containing ores with the aim to increase their magnetic susceptibility. An influence of the change of magnetic properties was studied in the case of Cu and Fe minerals.

The experiments of microwave heating were carried out with different size fractions of siderite ore from Nižná Slaná (Slovakia) (Znamenáčková, 2005). The microwave oven Whirlpool AVM 434 with a maximum power of 900 W at frequency of 2.45 GHz was used for microwave heating of 100 g siderite ore. Vibration mixing ensured the uniform heating of irradiated specimens. The magnetic separation of siderite ore specimens was carried out employing a roll-type electromagnetic separator Mechanobr, type 138 T-SEM, intended for dry separation processes.

Tab. 3. Magnetic separation of siderite ore (0.5 – 1 mm).

Microwave heating [min]	**Product**	**Yield** [%]	**$\kappa \cdot 10^{-6}$** [j.SI]	**Content of Fe** [%]	**Recovery of Fe** [%]
10	M	54.3	48,119	42.3	84.4
	N	45.7	20,145	9.3	15.6
	F	100.0	27,058	27.2	100.0
15	M	72.6	288,076	45.6	97.6
	N	27.4	10,147	3.0	2.4
	F	100.0	217,565	33.9	100.0

M - magnetic product, N - non-magnetic product, F- feed.

When the original sample of siderite ore ($\kappa = 947\times10^{-6}$) is subjected to the low-intensity magnetic separation (intensity of magnetic field = 135×10^{3} Am^{-1}), no separation was observed (Tab. 3). The magnetic separation of samples showed a rapid growth of Fe recovery equal to 84.4 % into magnetic products at separation of 10 min heated sample and the recovery equal to 97.6 % at separation of 15 min heated sample. It follows from the results that the time of heating equal to 15 min is sufficient for microwave pre-treatment of siderite ore before its magnetic separation.

The microwave radiation is used for the intensification of magnetic separation of Cu-ores (Lovás, 2005). The thermal decomposition as a result of interaction between microwave radiation and sulphidic minerals makes possible the magnetic separation of weakly magnetic chalcopyrite and tetrahedrite. In case of magnetic separation of chalcopyrite ore, it is possible to obtain the Cu recovery in 62.53 %. The volatile substances (Hg and Sb) releasing at thermic decomposition of tetrahedrite are adsorbed on the surface of active coal. The subsequent magnetic separation of tetrahedrite from the active coal has showed the decrease of the content of Sb from 14.3 % to the value 0.8 % and Hg from 0.8 % to the value 0.1 %. Thus, the microwave energy can be used to improve magnetic separation techniques.

Microwave-assisted leaching

Microwave assisted leaching of chalcopyrite and tetrahedrite

The influence of microwaves at leaching of chalcopyrite concentrate $CuFeS_2$ has been studied in comparison to the classical way of heating. The leaching experiments were performed in different leaching agents: 0.25 M ferric sulphate and 0.25 M ferric chloride. The temperature of the suspension was measured by means of an optical fibre of firm Nortech Fibronic. The results of experiments of leaching of $CuFeS_2$ concentrate with two leaching agents conserving the stoichiometric ratio at various temperatures of the leach are shown in Fig. 9 and Fig. 10, respectively.

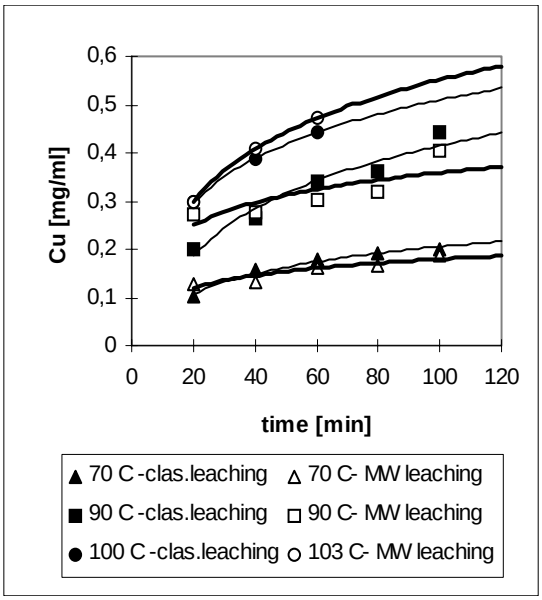

Fig. 9. The kinetics of Cu leaching of chalcopyrite concentrate leached in ferric sulphate 0.25 M Fe_2SO_4 without and with the influence of microwaves at different temperatures. – microwave leaching MW, – classical leaching.

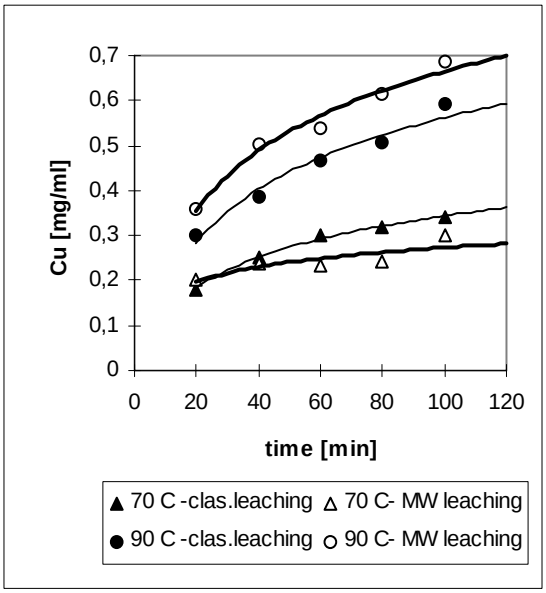

Fig. 10. The kinetics of Cu leaching of chalcopyrite concentrate leached in ferric chloride 0.25 M $FeCl_3$ with classical (class) method and microwaves (MW) at different temperatures. – microwave leaching MW, – classical leaching.

The currently known methods of leaching of chalcopyrite materials in acidic or neutral media do not ensure desirable yields of utility components and are very time-consuming. The influence of microwaves was observed for each of the leaching agents tested at used temperatures after 40-50 min of leaching. The comparison of the effectiveness of the two leaching agents favours the using of ferric chloride at microwave heating (temperature 90 ^{o}C). The use of microwaves in leaching the chalcopyrite concentrate with ferric sulphate becomes effective only when the temperature of the leaching suspension exceeds 100 ^{o}C. The positive influence of microwave radiation on the Cu-recovery was noticed (Lovás, 2005).

Hydrometallurgical method of processing the tetrahedrite concentrate from the Maria mine in Rožňava was described (Sekula, 2008). The microwave leaching of tetrahedrite was realized in leaching solution Na_2S (300 g.l^{-1}). After 15 min was observed the 4-manifold arising of As recovery. The positive influence of microwave radiation was also noticed at the extraction of Ag from tetrahedrite: the 9-manifold arising of Ag recovery was determined (Lovás, 2005).

Microwave leaching of electronic wastes

The investigations were carried out with an electronic scrap with the following composition: Cu - 64.65 %, Al - 4.54 %, Zn - 0.53 %, Pb - 0.16 %. 2 M HCl and 2 M H_2SO_4 were used as the leaching agents. The leaching kinetics was studied at different temperatures in a microwave oven Microsynth at the Institute of Chemical Process Fundamentals of the ASCR in Prague. Microwave device Microsynth consists of two magnetrons with power of 500 W (total power 1000 W), from immersion thermometer based on fiber optic, infrared thermometer and magnetic stirrer. The device has a control unit, to adjust the temperature of the solution, time and performance. The dissolution of Cu was negligible. The recovery of Pb and Al was dependent on temperature, time and leaching method. The recovery of Al was 91 % after 60 min of microwave heating at temperature of 60 °C, while 83 % in the case of conventional heating. At 80 °C all Pb went into a solution after 60 min of microwave leaching. Conventional leaching yielded about 65 %.

The microwave treatment of coal

Desulphurization by the microwaves is closely related to the form of sulphur compound in coal, its chemical structure, as well as the chemical activity of the leaching. Magnetic methods of mineral removal from coal depend on the difference in the magnetic moment associated with mineral particles and that of coal. The microwave heating enhances the magnetic susceptibility of the iron mineral, thus rendering it more amenable to magnetic separation.

The time dependence of microwave heating on decrease of total sulphur in coal (Cígeľ Colliery) (ratio NaOH:coal = 1:1) is shown on Fig. 11. – RMCL process (Radiation Molten Caustic Leaching).

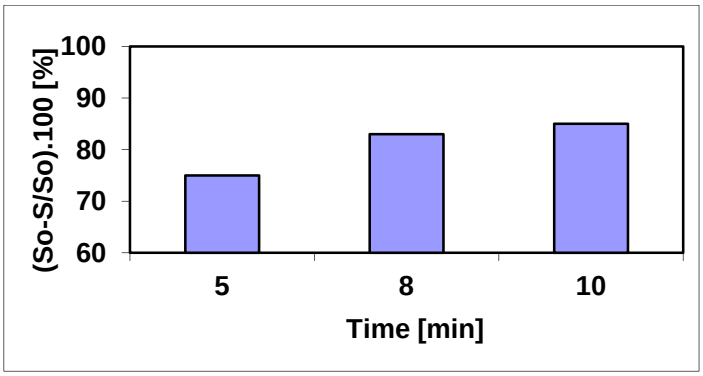

Fig. 11. The influence of the ratio of NaOH and coal on contents of sulphur in coal sample (Cígeľ) after RMCL process, S_o - contents of sulphur basic sample.

From these results, we can say that it is possible to remove 80 % of total sulphur for ratio NaOH:coal =3:1. It can be seen from Fig. 11. that as microwave exposure time increase, the proportion of total sulphur decrease. The sulphur removal increases to 76 % within 5 min and reaches to 85 % in 10 min, but after 10 min it dose not changed significantly (Turčániová, 2006). In muffle oven for temperature 380 °C (for reaction time 40 min) total sulphur decreases from value 2.27 % on 1.0 % (56 %). The main difference between the thermal and microwave heating was extremely short time for desulphurization in the case of microwave experiments.

Magnetic and triboelectrostatic separation of coal

The magnetic separation of raw coal (without pre-treatment) is ineffective. Therefore, the coal sample with particle size of 0.05-0.2 mm was heated before magnetic separation for 10 min in the microwave oven with power of 900 W at 2.45 GHz.

The results of the magnetic separation of coal after microwave radiation are in Tab. 4.

Tab. 4. The results of magnetic separation of coal after microwave radiation.

Grain size [mm]	**Magnetic induction** [T]	**Mass yield of magnetic product** [%]	**Recovery of S_{pyr} in magnetic product** [%]
0.05 – 0.2	0.13	4.32	39.6
	0.24	4.72	43.2
	0.42	10.80	44.7
	0.51	11.54	45.5

Microwave heating of coal is advantageous for subsequent desulphurisation. The use of microwave radiation for the desulphurisation of coal displays potential and may soon be a commercial reality. This would allow the use of high sulphur coals in an environmentally and economically sound way.

Microwave heating of coal is advantageous for subsequent triboelectrostatic separation. The influence of microwave radiation on coal was confirmed by the increase of the content of volatile substances from 30 to 44 wt % and a decrease of the ash from 49 to 18.3 wt %. The analysis of fractions after triboelectrostatic separation showed that at the central position of the splitter the ash and combustible matter in the non-treated sample was 36 and 44 wt %, respectively. At the same position of the splitter, the ash and combustible recovery in the microwave-treated sample were 12.2 and 46 wt %, respectively (Turčaniova, 2006).

Microwave – assisted extraction organic compounds from coal

Biologically interesting diterpane substances were extracted by microwave extraction. The coal powder (20 g) was extracted with dichloromethane in a microwave oven (Whirlpool AVM 434, 500 W, 2.45 GHz) in a distillation flask with a reversing reflux system at the boiling point of dichloromethane (1.5 – 60 min). Obtained extracts were filtered and the solvent was evaporated on a rotary evaporator. The total extract was passed through an SPE column (Chromabond SiOH – 6 ml/500 mg, Macharey - Nagel GmbH, Germany) with *n*-hexane (100 ml). The solvent was evaporated and the purified SPE extract was further separated by column chromatography on silica gel (5 g of Kieselgel 60, granularity 0.06 – 0.2 mm, Carl Roth GmbH, Germany) with 4× 20 ml of hexane. Column chromatography on silica gel was used to isolate diterpenes from the purified total extract for further characterization. Four fractions (F1–F4) were obtained. Saturated diterpenes were expected to elute with the solvent front and thus to be present in the first fraction (F1). TLC analysis of F1 showed the

presence of a single spot with $R_F = 0.91$. This spot was not observed in the other fractions (F2–F4). More polar components (aromatic or unsaturated) eluted with $R_F = 0.48$ and 0.33 in F3 and F4, respectively. GC–MS analysis confirmed presence of diterpanes in F1. The most abundant diterpane, phyllocladane, comprised more than 80 % of all components (based on peak areas in the TIC chromatogram). In order to learn whether phyllocladane can be separated from other diterpanes, the spot with $R_F = 0.91$ was divided into two parts, which were scraped off the plate separately. The upper part ($R_F = 0.91 – 0.96$) of the spot was found to contain less phyllocladane (81.4 %), whereas the lower part ($R_F = 0.87 – 0.91$) was enriched by this compound (85.8 %). To separate F1 with higher chromatographic resolution an HPLC system was used. Fractions were collected in 0.2 min intervals and analyzed by GC–MS. The highest amount of phyllocladane was found in the fraction number five (3.2 – 3.4 min; 88.6 %) (Zubrik, 2009).

Microwave extraction was optimized to obtain the diterpenes in high yield (Tab. 5). Microwave-assisted extraction (MAE) is a very efficient way of extracting organic compounds. When it is compared with classical procedures, e.g. Soxhlet extraction, the extraction times are dramatically shorter (Zubrik, 2007).

Tab. 5. Results of microwave extraction optimization performed with 20 g of coal, solvent dichlormethane.

Time [min]	m_{EXT}[a] [mg]	m_{F1}[b] [mg]	Y_{F1}[c] [‰]
1.5	372.8	42.1	2.11
5	401.8	39.7	1.99
10	388.8	52.8	2.64
20	325.0	50.8	2.54
60	231.9	56.4	2.82

[a] *Mass of total extract,* [b]*Mass of diterpene fraction F1,* [c]*Overall yield of diterpenes related to the original weight of coal:* $Y_{F1}=m_{F1}\times1000/m$ *Coal.*

The optimum extraction time was found to be 10 min; further increase of the extraction time had only minor effects.

Melting of andesite in a microwave oven

The influence of microwave energy on the melting process was investigated on andesite specimens originating from the locality of Ruskov (Slovak Republic) (Znamenáčková, 2003). Microwave heating of andesite rollers is shown in Fig. 12. The roller is melted in 10 minutes. It is evident that the roller is melted from inside.

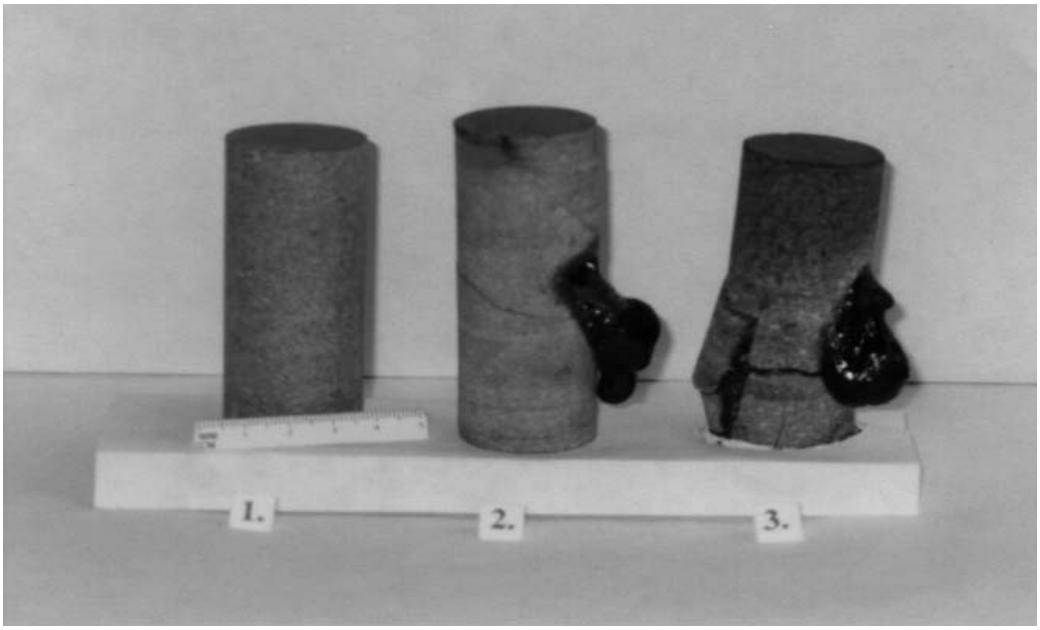

Fig. 12. Heating of andesite in microwave oven
1 - non-irradiated specimen, 2 - irradiated specimen (10 min), 3 - irradiated specimen (15 min).

Microwave vitrification

In Slovakia, the factory for nickel production was closed in 1993, but around 5.5 kt of wastes remain in a dump. This waste was used as a model sorbent of heavy metals (Cu, Cd, Co) from wastewater treatments. Heavy metals (Cu, Cd, Co) were precipitated with waste in concentration 10 mg of ions per 1 g of waste. The mixtures used in experiments contained 30 – 40 % of nickel leaching residue. Colourless container glass and dolomite were used as additives. The chemical composition of mixtures is described in Tab. 6.

Tab. 6. The chemical composition of mixtures.

SiO_2	Fe_2O_3	FeO	Al_2O_3	CaO	MgO	Na_2O	K_2O
48.01	11.63	6.79	2.49	9.78	3.93	8.28	0.37

Microwave vitrification was carried out in a microwave furnace Panasonic NE 2740 at the frequency 2450 MHz and output 1350 W. 120 g of sample was placed in a thermally isolated ceramic crucible. The samples were heated during 40 minutes. Then samples cooled in air. The temperature was measured by a thermocouple Raytek during heating.

The heating of samples was very fast. The temperature 400 °C was reached in 5 minutes in each sample.

After vitrification, the chemical durability (Tab. 7) and mechanical properties (Vicker`s microhardness) were tested.

Tab. 7. TCLP test of waste with precipitated heavy metals before vitrification and vitrified sample.

SAMPLE	Cd [$mg.l^{-1}$]	Co [$mg.l^{-1}$]	Cu [$mg.l^{-1}$]
before vitrification	472.15	417.50	321.50
vitrified	0.19	0.10	1.24

The TCLP (Toxicity Characteristic Leaching Procedure) test of precipitated heavy metals on waste materials has confirmed the necessity of waste stabilization. The microwave vitrification was applied because of a high content of iron in waste. The microhardness was tested by the Vicker`s indentation method, using a Leco microdurometer with a Vicker`s diamond indenter having 136° angle between faces. After focusing the place of indenting, the objective has approached to the sample and diamond indenter indented to the surface of the sample and then the length of indent diagonals has been measured. The sample was submitted to a load of 0.49 N. The Vicker`s microhardness was measured in two zones – matrix and white zones, which were identified in the picture from light microscopy (Fig. 13).

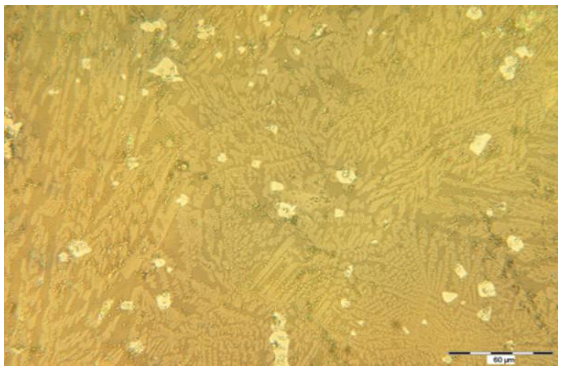

Fig. 13. The picture of vitrified sample from light microscopy.

The results of Vicker`s microhardness measurements are shown in Table 8.

Tab. 8. Vicker`s microhardness [GPa].

MATRIX	WHITE ZONE
6.75 ± 0.64	11.15 ± 1.28

The results proved differences between zones. The highest value of microhardness was obtained in a white area. The material with high microhardness and good chemical durability was obtained in a short time of heating (Pyzsková, 2005).

Conclusion

Microwave heating processes are currently undergoing investigation for application in a number of fields. The advantages of microwave energy may lead to significant savings in energy consumption, process time and environmental remediation. Compared with conventional heating techniques, microwave heating has the following additional advantages.

Higher heating rates: no direct contact between the heating source and the heated material, selective heating may be achieved, greater control of the heating or drying process, reduced equipment size and waste. A rapid heating of investigated samples gives a presupposition of microwave radiation utilisation in the field of rocks thermal processing.

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