MISCIBILITY OF CuO, NiO, AND ZnO IN THEIR BINARY MIXTURES AND ITS IMPACT FOR REPROCESSING INDUSTRIAL WASTES

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Miscibility of the components in the binary oxide systems containing CuO, NiO, and ZnO were studied using their dissolution in diluted H_2SO_4 combined with the examination of the oxides lattice parameters obtained by powder XRD. In samples synthesized by heating the corresponding mixed hydroxides at 900°C in air the following miscibility limits were found: 15 mol.% CuO and 26 mol.% ZnO in bunsenite (cubic NiO), 0.8 mol.% NiO and 1.2 mol.% ZnO in tenorite (monoclinic CuO), and 1.6 mol.% CuO and 0.5 mol.% NiO in zincite (hexagonal ZnO). The data are generally comparable or lower than those available in literature. The miscibility limits are of crucial importance for the recovery and purification of Cu, Ni, and Zn from the industrial sludges by a simple metallurgical scheme, which is currently tested in a laboratory scale.

Keywords: CuO, NiO, ZnO, Solubility, Dissolution

INTRODUCTION

Binary solid solutions of ZnO-CuO [1, 2], CuO-NiO [3, 4], and ZnO-NiO [5, 6] and their heterogeneous mixtures [1, 7, 8] have been studied for several reasons. Due to limited miscibility of the components and their different electronic and structural features, the heterogeneous mixtures of the oxides exhibit interesting properties. CuO-ZnO systems are catalytically active [1], and they can be used as gas sensors [7]. ZnO-NiO ceramics were tested for possible use as thermistors with a positive temperature dependence of resistivity [8]. The miscibility of these oxides has been studied mainly by X-ray powder diffraction and electron microprobe analysis. However, if the miscibility is smaller than about 5 %, such analyses are not sufficiently accurate and sensitive and the results are then accessible as qualitative statement (solubility is very low, about 1 wt.%, etc.). Due to various reasons to study the binary oxide mixtures, diverse synthesis techniques were used. Beside the common ceramic synthesis from the simple oxides [7, 8, 10], heating of nitrates [1, 3, 4], mixed hydroxides [9], and chemical vapor transport [2, 6] were employed. Because the miscibility usually depends on temperature and synthesis route [9], that wide scale of synthesis routes is another reason of the scatter of the reported values of the solubility limits.

In the 1990's Jandová et al. [11, 12] proposed a method for recovery of Zn from industrial wastes (figure 1), which is also related to the elemental composition of oxide mixtures. The method was tested with neutralization sludges, which arise in galvanic plants in Czechia and are deposited in landfills as dangerous waste. This state is unbearable from a global ecological and economical point of view. The Cu, Ni, and/or Zn contents in the sludges usually exceed those in raw materials used for their metallurgical production. However, after the deposition in the landfill, the wastes of various compositions are mixed together and further „contaminated" with soil, and their reprocessing in the future will be much more demanding. Theoretically, the metals could be recovered directly in the galvanic plants using hi-tech procedures such as electrolysis or liquid-liquid extraction, but the investment to these technologies exceeds the economical possibilities of the producers. Some form of the reprocessing of common neutralization sludges produced in the entire Czech Republic would hence be desirable.

The principle of the Jandová method [11, 12] is to calcine the mixture of metal hydroxides and basic salts and then to leach the oxide mixture in diluted acid under mild temperature (the last steps in scheme 1). High dissolution reactivity of ZnO in solutions of low acidity at ambient temperature has already been proposed to leach zinc from polymetallic waste before its electrowining (eg. from metallurgical dusts [13]). However, the same approach could possibly be directly used for the metal purification. If the waste contained a significant amount of Ni or Cu, substituted NiO or CuO of medium dissolution reactivity would be formed [12]. Trivalent metals, Al and Fe, are mainly converted to chemically almost inert spinels. The separation according to Jandová is based on a very fast dissolution
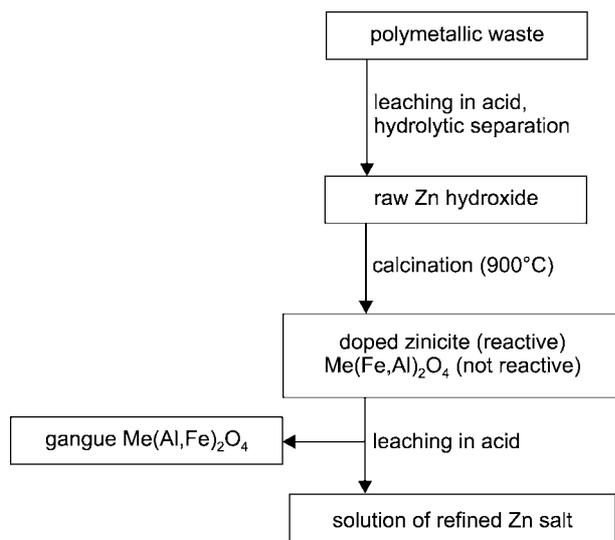
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┌─────────────────────────┐
│    polymetallic waste   │
└─────────────────────────┘
            │
            │  leaching in acid,
            │  hydrolytic separation
            ▼
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│     raw Zn hydroxide    │
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            │
            │  calcination (900°C)
            ▼
┌─────────────────────────┐
│  doped zinicite (reactive) │
│  Me(Fe,Al)₂O₄ (not reactive) │
└─────────────────────────┘
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┌────────────────────────┐   │  leaching in acid
│ gangue Me(Al,Fe)₂O₄   │◄──┤
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              │ solution of refined Zn salt │
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Figure 1. The Jandová scheme of recycling metals from polymetallic waste. It is shown for recovery of Zn [11, 12], but the same scheme is also possible for Cu [15].

of ZnO with respect to all other oxides in the calcines, however, the purity of the product has not been satisfactory, probably due to certain solubility of metal impurities in ZnO. Trivalent metals are practically insoluble in ZnO, however, divalent metals with ionic radii comparable to Zn, such as Cu or Ni, dissolve to a certain extent. Because the method according to Jandová et al. has also been tested for possible recovery of Ni [14] and Cu [15] from oxide mixtures, the question of miscibility concerns also bunsenite (NiO) and tenorite (CuO). The fact, that not pure oxides but their solid solutions are formed by calcination of the polymetallic waste, has not yet been taken into account in optimization of the Jandová scheme, although this fact affects both reactivity of the calcines and purity of the refined products.

The aim of this work was to establish miscibility of CuO, NiO and ZnO in their binary mixtures and to find the influence of their composition on their dissolution reactivity in diluted mineral acid. Because the results should be used to evaluate the recovery of metals from waste sludges according to Jandová et al., the oxide mixtures were synthesized by precipitating metal sulfates and subsequent calcination of the mixed hydroxides at 900°C on air as in the original procedure. This temperature might be too high for an industrial--scale process, but it is necessary to obtain well--crystalline insoluble oxides of trivalent metals. Additionally the solubility of other metals in ZnO was reported to decrease with increasing temperature [1] that is also desired to purify Zn. In the present work the miscibility of divalent metal oxides was determined by a combination of X-ray powder diffraction and examination of dissolution curves of the oxides in sulfuric acid. The results were compared to the data published in the literature.

EXPERIMENTAL PART

Oxide samples were synthesized by precipitating solution of $CuSO_4 \cdot 5H_2O$, $MgSO_4 \cdot 7H_2O$, $NiSO_4 \cdot 7H_2O$, and $ZnSO_4 \cdot 7H_2O$ by the solution of NaOH in molar amount OH/Me 2:1. The resulting mixed hydroxides were left to age at room temperature for a few hours to days, filtered off, washed by water until the filtrate was neutral, dried on air and calcined at 900°C for 3 or 5 hours. The following oxide series were prepared: CuO-NiO, CuO-ZnO, NiO-ZnO, and MgO-NiO (only NiO rich side). The series contained 24, 21, 15, and 7 samples with different ratios. The elemental composition of the solids was checked by AAS (Zeiss 3, flame atomization) and phase composition by X-ray diffraction analysis (XRD, Siemens D5005). XRD patterns were processed by PowderCell for Windows, that enabled to perform quantitative analysis of mixtures and also to refine lattice parameters by the least square method at fixed atomic positions in the crystal lattice. Structure models of tenorite, bunsenite and zincite were taken from ICSD database.

Dissolution kinetics was studied in 0.5 M H_2SO_4 possibly in the presence of redox agents. 100 - 300 mg of oxide mixtures was injected as water suspension (about 2 ml) to 300 ml preheated 0.5 M H_2SO_4. To alter the dissolution rate of bunsenite, appropriate amount of $K_2S_2O_8$ or $N_2H_4 \cdot H_2SO_4$ was added to the acid (more details are given in Results). The dissolving suspension was sampled in appropriate time intervals with a pipette (3 or 5 ml), filtered off, and the stock solutions of the filtrate were analyzed by AAS for dissolved Cu, Mg, Ni, and/or Zn. The dissolution extent was related to the final concentration of the metals after complete dissolution of the oxides. If it was necessary due to a too low reactivity of the oxide under given temperature, the dissolution was completed after increasing temperature or the addition of the oxidant (in the case of bunsenite). Dissolution data processing is described in Results.

RESULTS

The oxide mixtures obtained in this work contained common phases bunsenite (NiO), tenorite (CuO), and/or zincite (ZnO), or more precisely solid solutions with those structures. In the system NiO-CuO we did not observe formation of tetragonal or orthorhombic phases, which were reported by Davies et al. [3,4]. The lattice parameters were refined to reveal the formation of solid solutions. In agreement with literature, at the temperature 900 °C tenorite (CuO) and zincite (ZnO) can only slightly be doped by other metals, and the corresponding lattice parameters are hence very slightly affected by substitution. Bunsenite (NiO) can adopt a few tens percent ZnO and CuO and unlimited amount of MgO that is also reflected by a substantial change of the bunsenite lattice parameter. XRD pattern processing also yielded estimates of the percentage of the individual phases in the oxide mixtures, which is, however, reliable only if the content of the minor

component is above 1-2 wt.%. Except for CuO- and MgO-doped NiO, the dependence of the lattice parameters of the binary solid solutions on the composition was not published yet. Hence, to determine the actual composition of the solid solutions, another approach was necessary, in particular the course of the dissolution in diluted sulfuric acid.

Dissolution of solid solutions in aqueous H_2SO_4

To obtain dissolution curves suitable for further convenient processing, the dissolution should be finished in few tens of minutes to few hours. The filtration of subsamples of the dissolving suspension took about a half of minute that means that reactions finished in few minutes cannot be followed with reasonable accuracy. Reactions proceeding longer than a few hours are too time-consuming. Bunsenite, tenorite, and zincite differ considerably in their reactivity and so the conditions of the dissolution were chosen individually for the individual oxide mixtures, as it follows from table 1 and time scale in figures 2 and 3. To characterize dissolution kinetics of the least reactive pure, CuO- and ZnO-doped bunsenite, temperature was set to 60 or 70°C, and the redox potential was controlled by addition of oxidants. The suppressing effect of the reducing agents on the bunsenite dissolution was already reported [16], but not in so huge range as it is shown in figure 2. The proper choice of the redox agent can hence be used to almost switch on or stop the dissolution reactivity of bunsenite. In the experiments, where the dissolution of bunsenite was required, $K_2S_2O_8$ was added to the preheated H_2SO_4 solution just before the beginning of the dissolution experiment.

To obtain useful data about the elemental composition of the individual phases in the oxide mixtures, the dissolution reactivity of the mixture components must differ sufficiently. The separation of the dissolution of slowly reacting bunsenite from the faster dissolution of tenorite or zincite is enhanced by the sigmoidal shape of the bunsenite dissolution curve (ref. [12], figures 2 and 3). Hence, in NiO doped zincite, the dissolution of bunsenite at 40°C in the absence of an oxidizing agent can be totally neglected before a complete dissolution of zincite. Ni dissolved with the first fraction of dissolved Zn can only arise from zincite (ie. ZnO saturated by NiO), and similarly all Zn
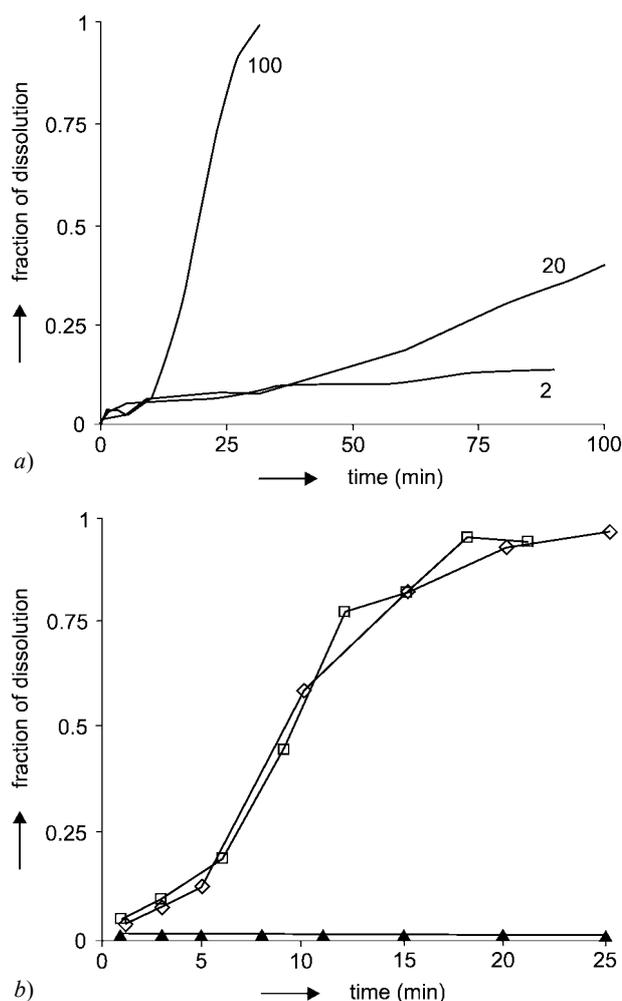

a)

b)

Figure 2. The influence of the redox agents on the bunsenite dissolution.
a) $Ni_{0.8}Cu_{0.2}O$, 70°C, number denotes weight of $K_2S_2O_8$ in mg per 300 ml solution.
b) $Ni_{0.56}Mg_{0.44}O$, 60°C, without redox agents ($\diamond$), with 100 mg $K_2S_2O_8$ ($\square$), with 100 mg $N_2H_4 \cdot H_2SO_4$ ($\blacktriangle$) per 300 ml solution.

dissolved congruently with Ni in the subsequent reaction stage arise from bunsenite (ie. NiO saturated by ZnO). Dissolution of NiO-CuO oxides was interpreted similarly (figure 3*a*). Also faster dissolution of zincite beside tenorite enabled us to distinguish the fraction of

Table 1. Experimental conditions used for dissolution of oxide mixtures in 0.5 M H_2SO_4.

| Oxide mixture	Dissolution conditions	Time for dissolution of components			Resulting information
		zincite	tenorite	bunsenite	
Each	30 or 40°C	$\sim 10^0$ min	$\sim 10^1$ min	$> 10^2$ min	NiO or CuO amount in zincite or tenorite
NiO-MgO	40 - 70°C	< 1 min	$\sim 10^0$ min	$\sim 10^1$ min	Reactivity of NiO
NiO-ZnO, NiO-CuO	60 - 70°C, $K_2S_2O_8$	< 1 min	< 1 min	$\sim 10^1$ min	Fraction of zincite or tenorite, ZnO or CuO amount in bunsenite

a)

b)
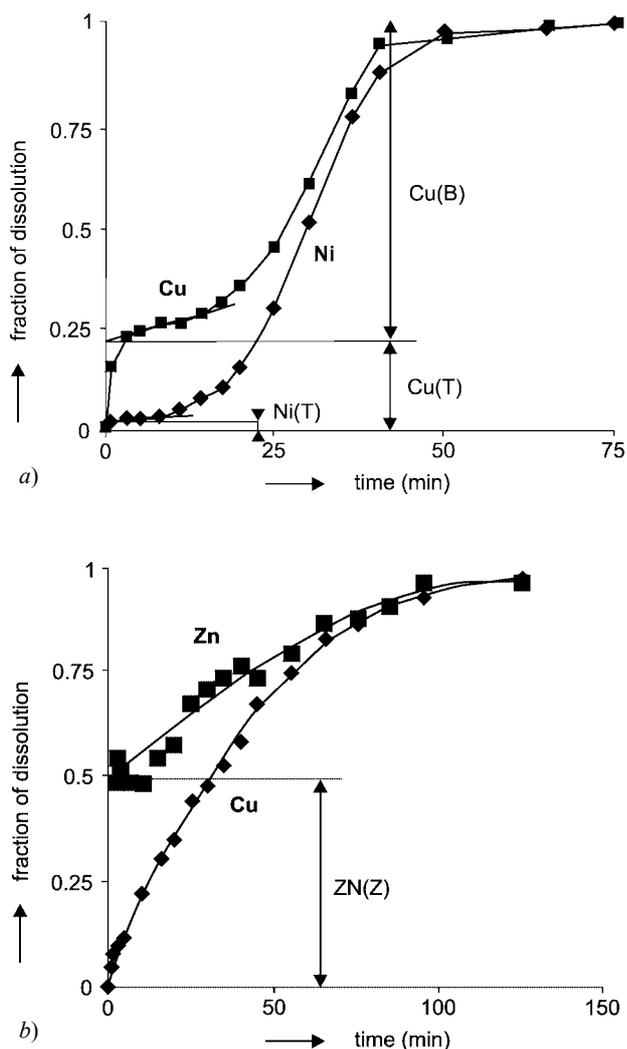
Figure 3. Typical examples of the dissolution curves.
a) 0.21 CuO-0.79 NiO, 70°C, $K_2S_2O_8$. Cu(T): fraction of Cu dissolved from tenorite, Ni(T): fraction of Ni dissolved from bunsenite, Cu(B) fraction of Cu dissolved from bunsenite.
b) 0.99 CuO-0.01 ZnO, 40°C. Zn(Z): fraction of Zn dissolved from zincite, Zn(T): fraction of Zn dissolved from tenorite.

Zn in the two phases of ZnO-CuO mixtures (figure 3*b*). Corresponding mathematical processing of dissolution curves yielded the first column of the miscibility limits in table 2.

The limit of this approach to evaluation of miscibility of the oxides is caused by the fact that if a substituted oxide is oversaturated by the substituent, the new, less reactive phase begins to form as nano-crystalline domains inside the grains of the major, more reactive phase. Presence of such structures was reported for ZnO-NiO mixture and was shown to cause systematic errors in evaluating the miscibility limits by microprobe analysis [17]. The dissolution reactivity of domains, which are much smaller than the grains of the well crystalline oxides detectable by XRD, is bigger than the reactivity of the larger grains of well crystalline oxides of the same phase composition, and causes simultaneous dissolution of more reactive phase (ZnO) and domains of the expectedly less reactive phase (NiO nanocrystalline domains in within ZnO grains). We can hence expect, that the solubility limits obtained from dissolution curves and given in the first column in table 2 are overestimated for the solid state solubility of the less reactive oxide (eg. NiO) in the more reactive one (eg. ZnO). Such estimates are, however, suitable for more detailed evaluation of lattice parameters obtained by XRD.

X-ray powder diffraction

The lattice parameters and the content of the oxide phases were also obtained for the oxide mixture. A rough estimate of the limit of the isostructural substitution in an oxide was obtained from the onset of the crystallization of the phase admixture with the increasing content of the substituting oxide (figures 4 and 5). For example in figure 5 the percentage of ZnO in ZnO-CuO series was plotted against the overall percentage of ZnO (open squares in bottom part of the graph). In the ascending part of the dependence the data set was fitted to line by the least square method, and the total percentage of ZnO in the mixture was found, at which zincite content starts to increase above zero. This percentage is an estimated solubility limit of ZnO in tenorite. Analogously the ZnO solubility limit in tenorite is estimated from the point where the linear

Table 2. Miscibility limits of the binary oxides studied obtained by different manners. The results are given in molar fractions.

| BO in (A,B)O	From the dissolution course	From the onset of BO crystallization	From the lattice parameter of (A,B)O	Published data
CuO in bunsenite	0.19	0.15	0.24	0.35 at 800 °C [9], 0.30 at 1000°C [3]
ZnO in bunsenite	0.30	0.26	0.31	0.24 at 800°C [6], 0.41 at 1050°C [10]
NiO in tenorite	0.015	0.008	0.012	not available
ZnO in tenorite	0.029	0.012	0.01-0.015	0.04 at 800°C [2]
CuO in zincite		0.016	unclear	0.02 at 800°C [2]
NiO in zincite	0.008	0.005	unclear	0.01 at 800°C [6], 0.03 at 1050°C [10]
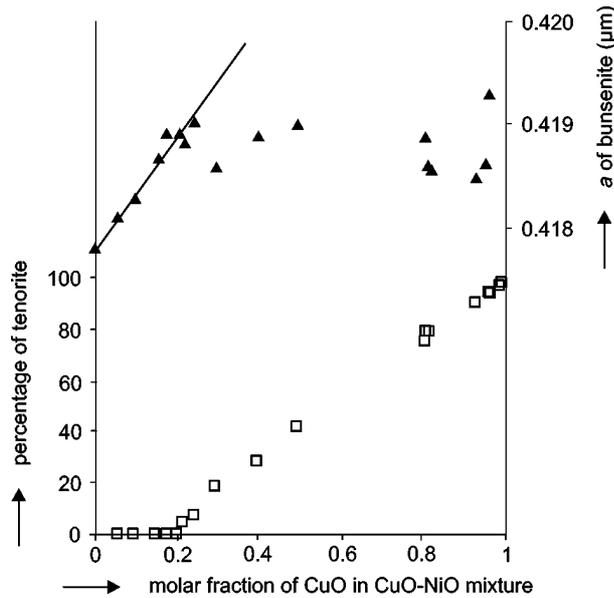
Figure 4. Tenorite content (□) and the lattice parameter *a* of bunsenite (▲) in samples CuO-NiO. Line: *a* of (Ni,Cu)O according to refs. [3, 4, 19].
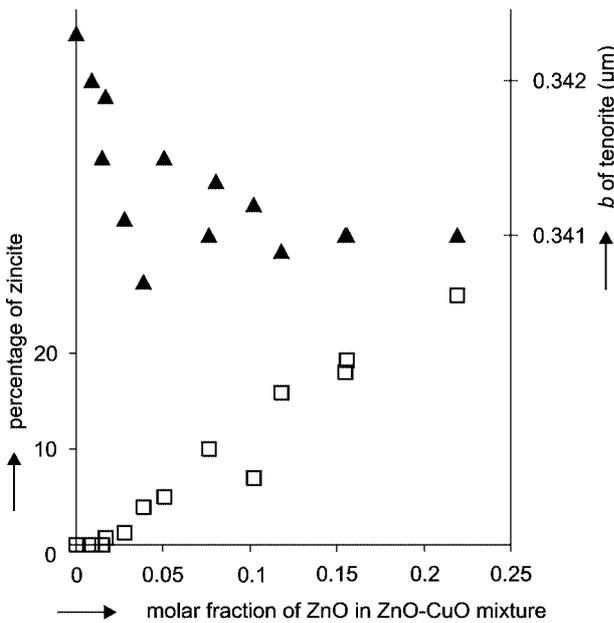

Figure 5. Zincite content (□) and the lattice parameter b of tenorite (▲) in samples CuO-ZnO.

dependence reaches 100% zincite. In such a way, the second column of the solubility limits in table 2 was obtained.

In the second step of the data evaluation, the lattice parameters were plotted against the total fractions of the metals in the oxide mixtures. We supposed that due to the doping the cell sizes depend monotonously on the actual ratio of the dopant in the crystal lattice of an

oxide. It is clearly apparent in figure 4 for CuO-NiO mixture, that the lattice parameter of CuO-doped bunsenite further grows with CuO total content even after the onset of the crystallization of tenorite (the fitted onset of the tenorite crystallization is at about 15 mol.% Cu, but the lattice parameter of bunsenite reaches its maximum at about 22 mol.% Cu). The evaluation of the miscibility limit from the onset of the new phase crystallization is hence lower than a correct value.

In the third step, we assumed that there is a linear dependence of the lattice parameters of the solid solutions on the actual ratio of the dopants according to the Vegard rule. For example we recently found such linear dependences in solid solutions of Fe-Mn oxides with three different crystal structures [18]. Vegard rule was also found to be valid for the MeO doped bunsenite [3, 4, 19] and is shown in figure 4 for CuO-doped bunsenite (the line in the upper part). We hence plotted the lattice parameters of the individual oxide phases obtained by XRD against the content of the amount of the doping oxide obtained from the dissolution course and searched for the finish of the lattice parameter change. An example is shown in figure 6: here with increasing ZnO content the tenorite lattice is distorted up to about 1.2 mol.% ZnO. Then the further increase of ZnO in tenorite do not change the CuO lattice probably due to the formation of ZnO domains in the tenorite matrix. This data evaluation should overcome the errors in the first two columns of the miscibility estimates in table 2 discussed above. The resulting, and probably the most reliable estimates are collected in the third column of solubility limits in table 2. Unfortunately, this approach was not applicable to substituted zincite, whose lattice parameters changed in almost as narrow range as the experimental error of the determination of the lattice parameters.
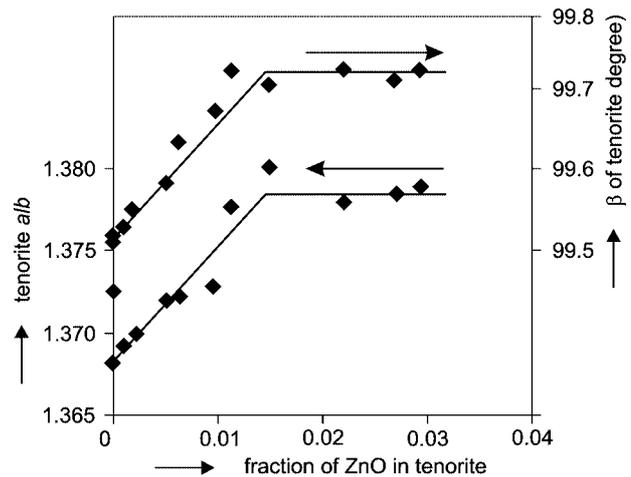

Figure 6. Final evaluation of the miscibility limit. The example of ZnO in tenorite. X-axis: the total molar percentage of ZnO in tenorite samples obtained by the dissolution course in diluted acid, Y-axes: lattice parameters of the tenorite in the samples. The breaks in the dependence represent the real solubility limit of ZnO in the tenorite lattice.

DISCUSSION

The most reliable estimates of the miscibility in the sense of the true isostructural substitution are collected in the third column of miscibility in table 2. The results already published are given in the last column for a comparison. The agreement in the data obtained by us and by others is generally acceptable except for CuO solubility in zincite reported by Locmelis and Binweiss [2], which is probably substantially overestimated. In our samples, the same change in the lattice parameters of ZnO-doped tenorite as reported by Locmelis and Binweiss was produced at much lower CuO content than the reported 4 mol.%. Probably also the literature data on CuO and NiO miscibility with zincite were overestimated, although the difference is not so dramatic. We believe that our study using the analysis of the ratios of the metals dissolving from the oxide mixture in combination with XRD determination of the lattice parameters of the oxide components is much more reliable than the estimates based only on the XRD data processing [2, 6] or a microprobe analysis [8, 17].

Evaluation of the applicability of the Jandová scheme

Solubility limits of NiO and ZnO in tenorite and CuO and NiO in zincite are relatively small. About 1-3% admixtures of divalent metal oxides are inserted into tenorite or are occluded in it in a form of nanocrystalline domains that are highly reactive toward dissolution in sulfuric acid. For the possible hydrometallurgical application this speciation is not important because both are a source of impurities in the final refined solution. For zincite the overall percentage of divalent admixtures is about 1 wt.%. These values represent the least amount of impurities in the refined ZnO or CuO obtained according to Jandová scheme, because these impurities will pass to the solution of refined salt according to figure 1. This result is also in accordance with the purity of the recovered Zn hydroxide concentrate obtained experimentally in a laboratory-scale reprocessing of the industrial sludge. Unfortunately, green NiO (or black CuO) cannot be present in such concentrations if the resulting refined ZnO should be used as a white pigment, which is currently the most economically attractive form of the recycled Zn^{2+} compounds. The method according to Jandová would hence be only applicable if such level of impurities could be tolerated, or if the method served only as a preliminary step before the electrochemical production of metallic Zn. Another complication found in this study is that if a significant amount of Ni was present in the polymetallic waste beside Zn, the bunsenite formed on calcination would behave as a sink for a comparable amount of Zn and so decrease the efficiency of the Zn recovery.

Similar limitations as for ZnO are also valid for possible refinement of CuO. Additionally, CuO is dissolved more slowly than ZnO and hence if Zn was present in the waste, it could not be removed from Cu containing wastes using the scheme in figure 1. The dissolution rates of tenorite and zincite are not sufficiently different to permit their separation in a manner similar to the flowsheet in figure 1, at least if H_2SO_4 is used for dissolution. Further work on increasing the relative reactivities of these phases is in progress in our laboratory.

Bunsenite crystal lattice can incorporate unlimited amount of MgO, about 20 mol.% CuO, and about 30 mol.% ZnO, that excludes the scheme according to figure 1 to be used for separation of Ni from theses divalent metals. Another problem of hydrometallurgical reprocessing Ni wastes is a low dissolution reactivity of bunsenite in acid dissolution. The substitution by CuO and ZnO changes only insignificantly the bunsenite dissolution reactivity, and MgO substitution substantially increases it. On the other hand, we found that the redox conditions could be used to control flexibly the dissolution reactivity of bunsenite. The addition of a sub-stoichiometric amount of a strong oxidant causes a substantial increase of the dissolution reactivity, and in the presence of reducing agents, the bunsenite dissolution can practically be 'switched off'.

CONCLUSIONS

We compared the relative reactivities of CuO, NiO, and ZnO in their dissolution in dilute H_2SO_4. The reactivities decrease in the order ZnO>CuO>NiO. Dissolution of Zn and Cu from their oxides (zincite and tenorite) is much faster than dissolution of Ni oxide-based solid solutions (bunsenite) and could theoretically be used for their hydrometallurgical separation. The dissolution of NiO can be controlled by redox conditions in the extracting solution. The dissolution rates of CuO and ZnO in H_2SO_4 are not sufficiently large to enable the separation of Cu and Zn from their oxide mixtures.

We determined the miscibility limits of CuO, NiO, and ZnO in their binary solid solutions and found the following limits of the Jandová scheme (figure 1):

- Refined solutions of Zn will contain at least several percent Cu or Ni, because such amount of Cu and Ni can be present in zincite in a highly reactive form (either as solid solutions with ZnO or as poorly crystalline domains within ZnO grain). The same will be valid for content of Ni impurity in refined Cu solution.
- NiO can bind several tens percent CuO and ZnO and hence would cause the losses of Cu and Zn in their refinement.

Acknowledgement

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MÍSITELNOSTI CuO, NiO, A ZnO
V JEJICH BINÁRNÍCH SMĚSÍCH
A JEJICH DŮSLEDKY
PRO PŘEPRACOVÁNÍ PRŮMYSLOVÝCH ODPADŮ

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Vzájemná rozpustnost složek binárních oxidů obsahujících CuO, NiO a ZnO se studovala pomocí kinetiky jejich rozpouštění ve zředěné H_2SO_4 v kombinaci se zkoumáním mřížkových parametrů těchto oxidů práškovou rtg difrakcí. Ve vzorcích, připravených zahříváním příslušných směsných hydroxidů na vzduchu při 900°C, byly nalezeny následující meze rozpustnosti: 15 mol.% CuO a 26 mol.% ZnO v bunsenitu (kubickém NiO), 0.8 mol.% NiO a 1.2 mol.% ZnO v tenoritu (monoklinickém CuO), a 1.6 mol.% CuO a 0.5 mol.% NiO v zincitu (hexagonálním ZnO). Výsledky jsou podobné nebo nižší ve srovnání s dosud publikovanými daty. Hranice rozpustnosti jsou klíčové pro získávání a čištění Cu, Ni a Zn z průmyslových odpadních kalů podle jednoduchého metalurgického schématu, které se v současnosti testuje v laboratorním měřítku.