

INVESTIGATION OF ADSORPTION PRODUCTS OF OXYNE ONTO BLENDE AND SMITHSONITE BY IR SPECTROSCOPY

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ABSTRACT. The compounds formed during flotation with oxyme at the surface of smithsonite and blende were studied by IR spectroscopy. By using the potassium bromide pelleting technique the IR spectra were run for blende, smithsonite, oxyme, Zn(II) oxynate, as well as for blende and smithsonite treated with a solution of oxynate. By comparing the obtained spectra the conclusion drawn is that the compound formed at the surface of blende and smithsonite is Zn(II) oxynate.

INTRODUCTION

The solid – liquid interface, however, is more difficult to investigate by IR spectroscopy especially when the liquid is an aqueous solution of reagents, as used in flotation processes.

IR spectroscopic determinations have also been used for surveying the formation of certain species at the surface of minerals during adsorption of various reagents, and there are many reports on this subject [1-10].

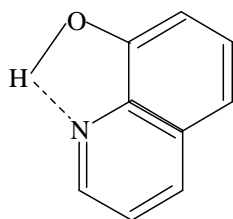
The chelation agents, organic agents acted as precipitants in solution, are lately used in the processes of separation by flotation of ores as well. These reagents are suited to ores containing oxidated minerals and they work as collectors or activators [3-10].

In these investigations and experiments interest in the collector properties exerted by some chelation reagents during the flotation of oxidated minerals [11].

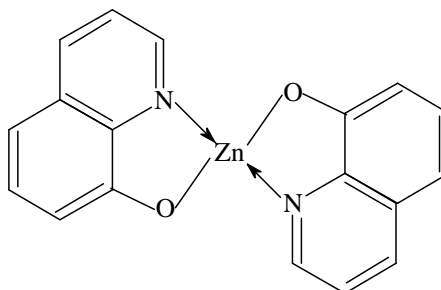
One of this studied reagents, namely 8 – hidroxyquinoline (structure I) or oxyme performs important zinc recoveries in the flotation of smithsonite and blende, and it proves a good collector [12].

In solution, oxyme forms insoluble, stable chelate compounds with many metallic ions, Zn (II) included, and these compounds have (II) type structure.

In the present paper we report some results obtained by an IR spectroscopic investigation, on compounds formed at the surface of smithsonite and blende in the flotation process with oxyme.



Structure I



Structure II

EXPERIMENTAL

The recording of IR spectra was performed by a UR 20, Carl Zeis Jena, double beam spectrometer; KBr, NaCl, and LiF prisms; resolution $0,6\text{-}2000\text{ cm}^{-1}$; region $400\text{-}5000\text{ cm}^{-1}$.

We assumed that zinc oxynate was formed at the surface of blende and smithsonite treated with the chelation reagent. This complex is prepared as follows: 150 ml $0,1\text{ mol l}^{-1}$ aqueous $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ is treated with 75 ml $0,3\text{ mol l}^{-1}$ oxyne in acetic acid 10%; The complex is warmed up and 25 ml oxyne is added, after cooling the pH is established at 9 by NH_4OH , the precipitate it is filtered on crucible G_4 , washed with acetic acid 10% and distilled water, and dried in the drying stove for an hour at 110°C . The preparation method was realised based on personal determinations at different pH values. Thus it was obtained a quantitative precipitation at pH 9, when Zn oxynate contained 18,7% Zn (corresponding to the theoretical composition). By using acetone as a solvent a quantitative precipitation was not possible.

The minerals, blende and smithsonite, was grounded in an agate mortar up to the granulation of approximative $2\mu\text{m}$. A part of the samples was used for the obtaining of IR spectra and another (100 mg) part was treated with 25 ml acetone solution of $10^{-3}\text{ mol l}^{-1}$ oxyne at pH 7, stirred for 5 minutes, filtered through a filter crucible, washed and dried in air. Blende and smithsonite flotation recovery is maximum at pH 7 [12]. By using acetic acid as oxyne solvent a maximum recovery can not be obtained [5].

Using the potassium bromide pelleting technique, we recorded the IR spectra in the region $400\text{-}1700\text{ cm}^{-1}$ for: oxyne, smithsonite, blende, Zn (II) oxynate, smithsonite and blende treated with acetone solution of oxyne. The pellets contain 10 mg Zn oxynate, 10 mg blende or smithsonite treated with oxyne and 600 mg KBr. Blende contains 57,7 % Zn and smithsonite 49,5 % Zn.

In blende case we also used another method for drawing the IR spectra: 10 mg of blende with the granulation of $45\text{-}150\mu\text{m}$ is treated with acetone solution of oxyne, filtered and dried in air. The compound formed on the blende surface is extracted 10 ml of CCl_4 or CHCl_3 , under continuing stirring (operation is repeated for 5 times). The extract is dried on KBr support, which is used to prepare the pellets necessary for drawing the IR spectra.

RESULTS AND DISCUSSIONS

The smithsonite – oxyme system

Figure 1 combines the IR spectra for smithsonite (curve a), oxyme (curve b), zinc oxynate (curve c) and smithsonite after treatment with acetone solution of oxyme (curve d). By comparing the spectra we can notice the presence of bands characteristic to the Zn (II) oxynate from 1580; 1505; 1390; 1330; 1280; 1120; 1040; 915; 868 and 680 cm^{-1} in the spectrum of smithsonite treated with oxyme. Therefore on the surface of smithsonite treated with the solution of reagent with chelatant action is formed Zn (II) oxynate.

In the same way was demonstrated the formation of Cu(II) oxynate on the surface of malaquite [7] and that of Pb (II) oxynate on the surface of ceruzite [11].

The intense bands from 1470; 735 and 610 cm^{-1} in the spectrum of zinc oxynate are situated at 1465; 730 and 620 cm^{-1} , on the smithsonite surface. The band of smithsonite treated with oxyme at 1235 cm^{-1} is shifted against the position in the spectrum of oxyme at 1230 cm^{-1} and against the position of the complex at 1240 cm^{-1} .

Also there can be found some bands at 580 and 465 cm^{-1} which do not appear in the spectra of oxyme and zinc oxynate.

There are, however, numerous bands of oxyme which are absent in the case of smithsonite surface: 1515; 1415; 1215; 1180; 742; 712; 477 cm^{-1} (figure 1b).

The blende – oxyme system

Figure 2 combines the IR spectra for blende (curve a), oxyme (curve b) zinc oxynate (curve c) and blende after treatment with acetone solution of oxyme (curve d), the adsorption product of acetone on blende extracted with CCl_4 (curve e), and extracted with CHCl_3 (curve f).

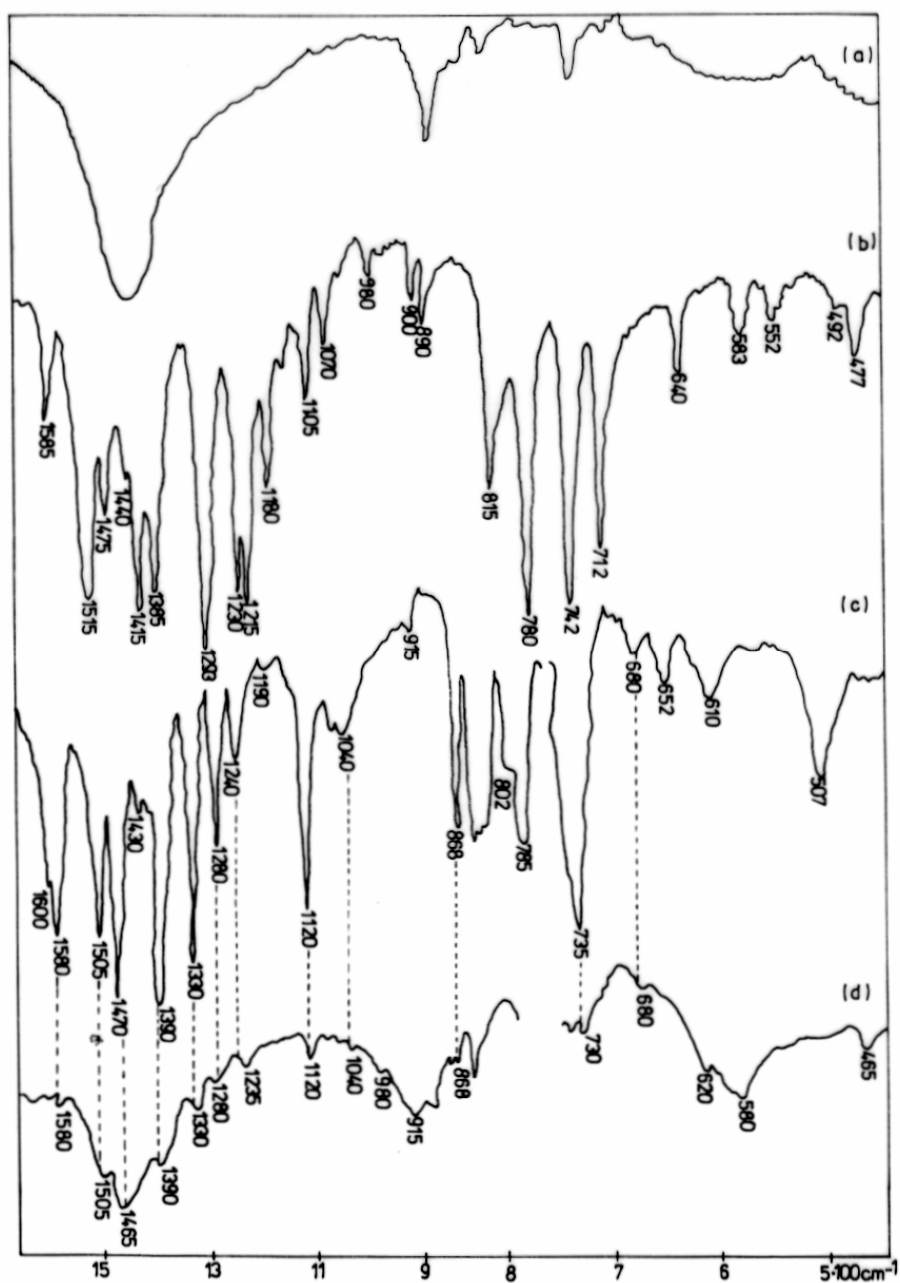


Figure 1. IR spectra indicating the adsorption of oxyne onto smithsonite.
a) smithsonite in KBr; b) oxyne in KBr; c) zinc oxynate in KBr;
d) smithsonite after treatment with acetone solution of oxyne

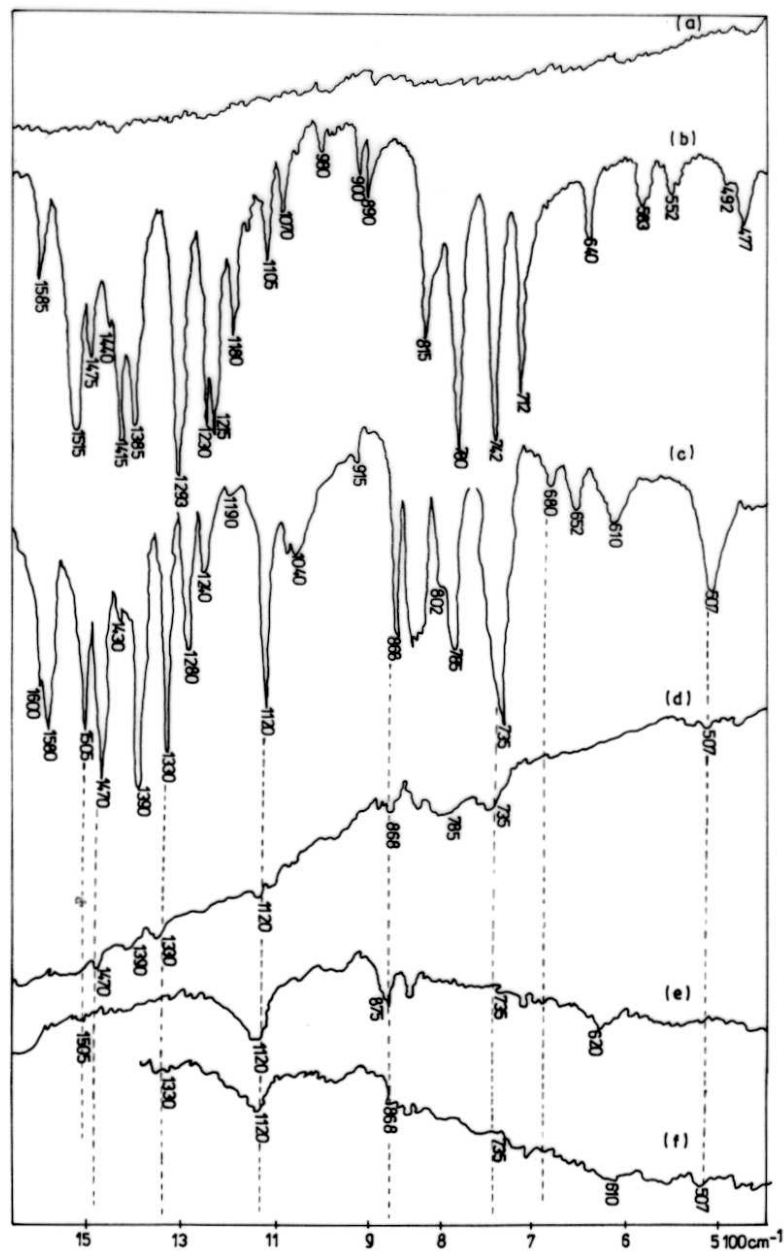


Figure 2. IR spectra indicating the adsorption of oxylene onto blende.
a) blende in KBr; b) zinc oxynate; b) oxylene in KBr c) zinc oxynate d) blende after treatment with acetone solution of oxylene; e) adsorption product of oxylene extracted with CCl_4 ; f) adsorption product of oxylene extracted with CHCl_3

By comparing the a, b, c and d spectra (figure 2) we can notice the presence of characteristic bands for zinc oxynate (II) at 1470; 1390; 1330; 1120; 868; 785; 735 cm^{-1} in the spectrum of blende treated with oxyne (curve d). This bands are not very well defined in spectrum from figure 2d.

The spectrum 2e (adsorbition of oxyne on blende extracted with CCl_4) give evidence of a stonger band at 1120 cm^{-1} from zinc (II) oxynate and a undefined band at 1505 cm^{-1} ; we can see that the Zn (II) oxynate band moves from 868 cm^{-1} to 875 cm^{-1} in blende case.

The spectrum 2f (adsorbition of oxyne on blende extracted with CHCl_3) include a well outlined band at 1120 cm^{-1} and undefined bands at 1330; 868; 610 and 507 cm^{-1} of Zn (II) oxynate spectrum.

CONCLUSIONS

The studied IR spectra indicate the presence of characteristic bands from zinc oxynate in the smithsonite an blende spectre treated with oxyne. We can notice that at the smithsonite and blende's surfaces was formed Zn (II) oxynate during flotation using oxyne as collector.

In the case of blende, the IR spectra correspond to adsorbition product of oxyne extracted with CCl_4 or CHCl_3 , have to complete the previous results.

To explain that in some cases appears misplaced bands, that suggest the possible complex formation between the mineral surface and reagents with a different stoichiometric composition than Zn (II) oxynate, may be caused to a smaller concentration of reagents fixed onto mineral surface.

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