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THE LEACHING BEHAVIOR OF OCEAN POLYMETALLIC NODULES IN CHLORIDE SOLUTIONS

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Laboratory investigations have been undertaken in order to determine the influence of major parameters on the process of leaching of metal values from ocean nodules by means of model "sea-water", that is - acidified chloride solutions. The effect of leaching conditions, i.e. time, temperature, stirring rate, liquid/solid ratio and HCl concentration was examined on leaching efficiency. It was found that concentration of hydrochloric acid greatly influences leaching of metals from ocean nodules. At HCl concentration exceeding 4.0 M and at 30 °C of temperature all valuable metals were almost completely leached out from the feed. Elevation of the leaching temperature allowed for reduction of the initial HCl concentration. Deep-sea nodules studied in this work exhibited somewhat different leaching behavior that reported in the literature.

INTRODUCTION

Since the 1960's manganese nodules have been recognized as a prospective source of metals and their investigation is stimulated by the progressive depletion of land-based mineral resources (Agarwal et al., 1976). These nodules are an unique and complex deposit requiring an equally unique extraction process suitable to their specific properties. Manganese and iron oxides are the major components of nodules while copper, cobalt and nickel - finely disseminated in the oxide matrix - make their economic value (Sridhar et al., 1976; Agarwal et al., 1976; Fuerstenau et al., 1973). Unfortunately, nickel, copper and cobalt are not present in separate mineral phases and therefore they cannot be liberated and beneficiated by means of widespread physical methods. Unique chemical compositions as well as dissemination of Cu, Ni, Co and heavy metals in Fe/Mn oxide phase require the application of chemical reductants to decompose the oxide matrix and to liberate the colloidal particles of metallic compounds for leaching.

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Numerous metallurgical processes for recovering of metals from nodules have been investigated in recent thirty years (Han et al, 1974, Han and Fuerstenau, 1975; Han and Fuerstenau, 1980; Fuerstenau and Han, 1983; Jana, 1993; Kohga et al, 1995; Acharya, 1999. High moisture content (30 - 40 %), high porosity (about 60 %), and inability for common physical beneficiation eliminate the application of simple and economically feasible smelting processes for ocean nodules. Hydrometallurgy remains the only rational route of metals recovering from this specific raw material. In rational selection of the most effective hydrometallurgical process, we look for the lowest consumption of reagent and for the best recovery of metals in a relatively simple process. However, processes considered for the commercial application must strictly involve a reducing step of insoluble MnO_2 matrix to soluble Mn(II) ions and releasing the valuable metals for further recovering (Han and Fuerstenau, 1980; Shinn et al., 1984; Kawahara and Mitsuo, 1993; Chen, 1996). Hydrogen chloride, Fe(II) ions, sulfur dioxide and some organic compounds are frequently used as reductants.

The hydrometallurgical processes can be divided into several types with respect to leaching reagents used. For example, hydrochloric acid (Jana, 1993) is comparatively cheap leaching agent and can be obtained from the sea water and reused. Both ammonia (Chen, 1996, Acharya, 1999); and elevated temperature/elevated pressure leaching processes (Han and Fuerstenau, 1975) can selectively extract nickel, copper and cobalt from the feed nodules and therefore, reduce the consumption of leaching reagents.

We now report experimental results on hydrochloric acid leaching, ammonia leaching and elevated temperature, elevated pressure leaching processes of the Pacific's polymetallic nodules. Leaching behavior of these nodules in hydrochloric acid acidified model "sea water " has been studied in detail.

EXPERIMENTAL

A sample of polymetallic nodules was taken from northeastern Pacific's subequatorial part of Clarion/Clipperton Fracture Zone (CCZ). Particularly, nodules from eastern nodule-bearing area of CCZ within the Interoceanmetal (IOM) pioneer investor claim. The air-dried nodules, which density was 2.53 g/cm^3 and bulk density of 1.23 g/cm^3 , were crushed with a hammer and subsequently ground in the ball mill for about 1 hour. The particle size distribution of ground nodules used as a feed in the leaching experiments is given in Table 1.

Table 1. Particle size distribution of deep-sea nodules

Size fraction	Weight %
-0.045	10.06
-0.071+0.045	38.7
-0.160+0.071	29.27
-0.300+0.160	13.26
+0.300	8.71

Thermal analysis revealed 13.06 % of the moisture content in the examined nodules. Feed material for leaching experiments was the mixture of all particle fractions. Chemical composition of the feed is given in Table 2. Leaching experiments were carried out in a three-necked, round-bottomed glass reactor with the reflux condenser and a stirrer of adjustable stirring rate. Samples of 30 g of nodules were leached in 300 ml of solution. Reagent grade chemicals dissolved in distilled water were utilized during the examinations. The temperature was kept constant using thermostat throughout each test.

Table 2. Chemical composition of polymetallic deep-sea nodules

Element	Cu	Mn	Fe	Ni	Co
Content, % wt.	1.05	22.51	3.82	1.12	0.13

Additional preliminary tests were performed in 0.6 M $\text{NH}_3 \cdot \text{H}_2\text{O} + 0.6 \text{ M } (\text{NH}_4)_2\text{CO}_3$ in the presence of SO_2 at temperature -90°C and at liquid/solid ratio - 10:1. A sample of nodules was also leached at 120°C and 0.7 MPa of O_2 pressure with liquid/solid ratio of 10:1 and H_2SO_4 added in amount of 0.75 kg/kg of nodules.

After each leaching experiment and after periodical sampling, a solid-liquid separation was performed. The filtered aqueous phase was colorimetrically analyzed for cobalt with 0.20 % of Nitroso-R-Salt and 25 % of CH_3COONa solutions while manganese was determined colorimetrically as MnO_4^- due to oxidation with KIO_4 . Moreover, selected solid residues after leaching were digested and analyzed with AAS for nickel, copper, cobalt, manganese and iron to verify the results of leaching.

RESULTS AND DISCUSSION

PRELIMINARY EXPERIMENTS

In order to determine the leaching behavior of examined nodules at conditions previously reported in the literature (Agarwal, 1976; Fuerstenau et al., 1973; Han and Fuerstenau, 1980; Jana, 1993; Sridhar et al., 1976; Kohga, 1995), several checking experiments were performed. The experiments involved hydrochloric acid and sodium chloride leaching, ammonia- SO_2 leaching and high temperature, high pressure sulfuric acid leaching.

Figure 1 shows the recovery versus leaching time plot for cobalt and manganese during the leaching of nodules with hydrochloric acid - sodium chloride aqueous solutions at 30°C . The relationships in Figure 1 indicate that about 80% of cobalt and nearly 70% of manganese were extracted after 1.5 hours of leaching of nodules with aqueous $\text{HCl} + \text{NaCl}$. The results based on the chemical analysis of the solid residue (9.6 g) after leaching for 3 hours are shown in table 3. The reported results of similar leaching: 99% of copper, 99% of nickel, 93% of cobalt, 92% of iron and 96% of manganese recovery (Jana, 1993), were better than these of above experiment.

Table 3. Leaching recovery of metals in HCl – NaCl solution at 30 °C

Element	Cu	Fe	Mn	Ni	Co
Assay of residue , %	0.135	3.37	16.69	0.293	0.068
Recovery , %	96.19	71.77	76.27	91.63	83.26

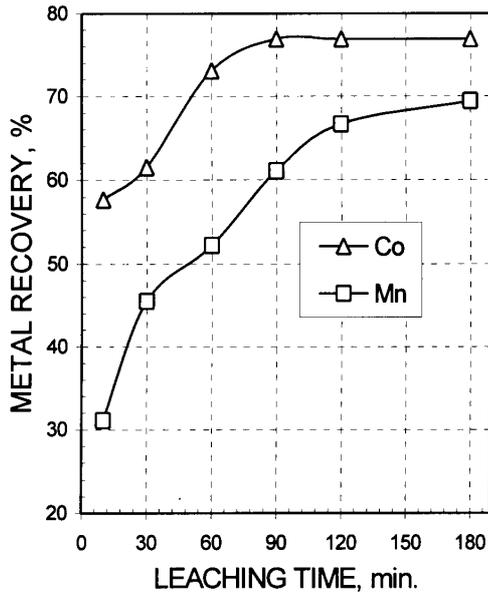


Fig. 1. The recovery – time relationship for HCl - NaCl leaching of nodules (NaCl - 4.0 M, HCl - 2.75 M, temperature – 30 °C, stirring rate - 900 rpm, liquid/solid ratio - 10:1)

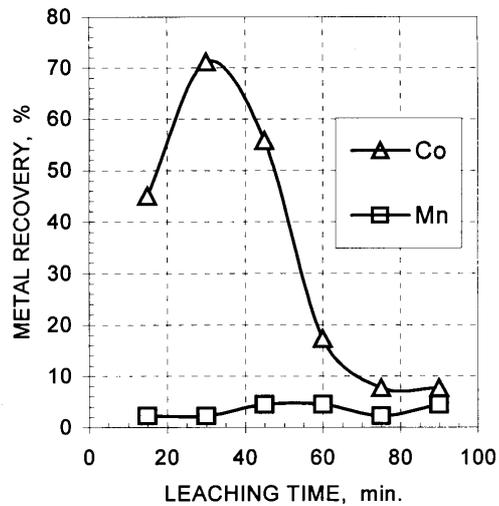


Figure 2. Recovery of Co and Mn during the leaching of nodules in NH₃ - SO₂ solution. (NH₃ · H₂O - 0.6 M, (NH₄)₂CO₃ - 0.6M, flow rate of SO₂ -100 ml/min., temperature – 90 °C, stirring rate – 900 rpm, liquid/solid ratio - 10:1)

Figure 2 shows the Mn and Co recovery - time plots for ammonia - SO₂ leaching of nodules at 90 °C. The observed relationships evidently show that cobalt was greatly extracted in half an hour while the most of manganese remained in the solid after leaching. The results based on the analysis of the residue (29.26 g) after leaching for 30 minutes are given in Table 4.

Table 4. Leaching recovery of metals in NH₃ – SO₂ solution at 90 °C

Element	Cu	Fe	Mn	Ni	Co
Assay of residue , %	0.638	3.46	21.85	0.821	0.064
Recovery , %	41.30	11.66	5.33	28.50	51.98

Literature data (Kawahara and Mitsuo, 1993) reported different results of nodules' leaching with NH_3 - SO_2 solutions. Recovery of metals increased with the time of leaching and 99% of copper, 98% of nickel, 95% of cobalt, 3% of manganese and 1% of iron was leached after 1.5 hour. Figure 3 reveals that recovery of cobalt increases with the leaching time up to 30 minutes, but thereafter remarkably decreased.

Literature reported recoveries (Kawahara and Mitsuo, 1993) were higher than these in Figure 2. The reason for this discrepancy may originate from cobalt precipitation or adsorption on precipitated Fe and Mn solid compounds (Osseo-Assare and Fuerstenau, 1979) since the final pH observed was as high as 4.8. Moreover, liquid to solid ratio applied in the literature (Kawahara and Mitsuo, 1993) was much higher. Therefore, a different leaching tendency was observed in presented examinations.

The results of leaching of nodules with oxygenated sulfuric acid at elevated temperature and pressure (120 °C and 0.7 MPa of O_2 pressure) based on analysis of the solid residue (71.58 g) after leaching of 100 g of nodules for 3 hours, are given in table 5.

Table 5. Recovery of metals during the elevated temperature and pressure leaching of nodules

Element	Cu	Fe	Mn	Ni	Co
Assay of residue, %	0.289	4.12	24.08	0.532	0.128
Recovery, %	80.30	22.80	23.43	66.00	29.52

Table 6. Leaching recovery of metals in 6 M HCl at 30 °C

Element	Cu	Fe	Mn	Ni	Co
Assay of residue, %	0.03	4.12	0.09	0.009	0.0013
Recovery, %	99.58	84.25	99.94	99.88	99.85

These leaching results differ from those reported in the literature (Han and Fuerstenau, 1975) where 80 % of nickel, 90 % of copper, 30 % of cobalt, 3.2 % of manganese and 2.0 % of iron was extracted from the feed nodules at 195 °C, 0.7 MPa (7 atm) of O_2 and 10 g H_2SO_4 /kg of nodules.

The results of preliminary leaching tests reveal that the examined nodules exhibited somewhat different leaching behavior than other nodules described in the literature. Particularly, low metal recoveries were achieved with studied nodules at similar leaching conditions. Nevertheless, general tendencies in metal leachability were similar.

LEACHING WITH HYDROCHLORIC ACID

The leaching systems using hydrochloric acid were systematically examined since their essential practical meaning. Figure 3 shows the concentration versus time relationships for leaching of Co and Mn from nodules with aqueous 6.0 M HCl. It can be observed that recovery of cobalt and manganese slightly increases with leaching

time. Over 95 % of cobalt and 98 % of manganese was extracted after leaching in 6.0 M hydrochloric acid for half an hour at 30 °C. Table 6 provides the results of leaching based on analysis of the solid residue (4.38 g) after leaching for 2 hours.

The results presented in Table 6 indicate that even at temperature as low as 30 °C the leaching with 6.0 M solutions of hydrochloric acid resulted in recovering of almost all nickel, copper, cobalt, manganese and the most of iron from the feed nodules.

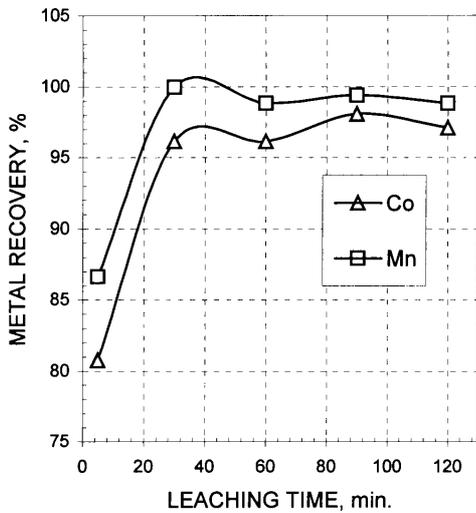


Fig. 3. Recovery of Co and Mn during the leaching of nodules with hydrochloric acid solution. (HCl – 6.0 M, temperature – 30 °C, stirring rate - 900 rpm, liquid/solid ratio - 10:1)

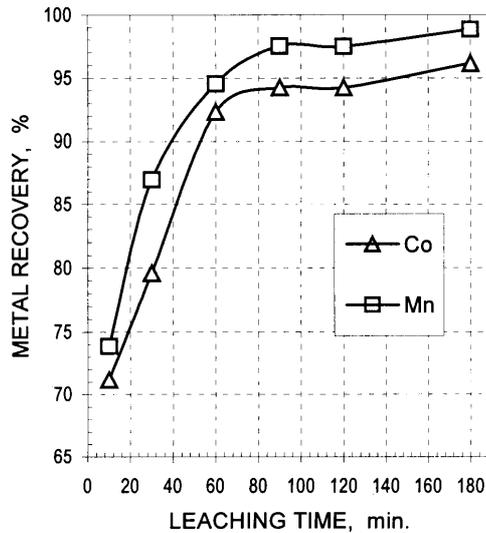


Fig. 4. Recovery of Co and Mn from nodules during the leaching in HCl + NaCl solution. (NaCl 0.55 M, HCl 5M, stirring rate 900 r/min., temperature 30°C, liquid to solid ratio 10:1)

Based on the idea that leaching of nodules would be performed on a ship fixed at the spot where the ocean polymetallic nodules are mined, the leaching behavior of nodules in acidified with HCl model "sea water" has been studied in detail. Since the content of Cl^- in the ocean water from CCZ is 19.6 g/L, the model "sea water" used in the subsequent tests contained 0.55 M of NaCl.

EFFECT OF LEACHING TIME

The metal recovery vs. leaching time plots for extraction of nodules with a model "sea water" containing 5.0 M HCl is showed in Figure 4. It is well seen that leaching for only 5 minutes leads to more than 70 % of cobalt and manganese recovery. Observed recovery of both cobalt and manganese was therefore high and further increased with leaching time. After 1.5 hour of the process, almost all manganese and

about 95 % of cobalt was leached out from the feed nodules. Extension of the leaching time over 2 hours resulted in additional increase of the recovery of Co and Mn to about 95 % and 98 %, respectively.

The results of leaching of nodules with model "sea water" containing varying concentrations of HCl are shown in Figure 5. The recovery of cobalt and manganese from nodules increased with the increase of hydrochloric acid concentration at 30 °C. However, this dependence is rather complex. At hydrochloric acid concentration less than 3.0 M recovery of cobalt and manganese was very low. The reason for this was to low concentration of hydrochloric acid required to reduce insoluble Mn(IV) to Mn(II) ions. This reduction step was necessary for cobalt, copper and nickel (associated with iron- and manganese-containing minerals) to be leached.

EFFECT OF HYDROCHLORIC ACID CONCENTRATION

The leaching recovery of cobalt and manganese rose sharply with hydrochloric acid concentration between 3.0 M and 5.0 M and reached above 93 % for Co and above 98 % for Mn in 5.0 M HCl.

EFFECT OF STIRRING RATE

Experiments have been performed at stirring rates within the range of 300 - 1100 rpm and the results were presented in Figure 6.

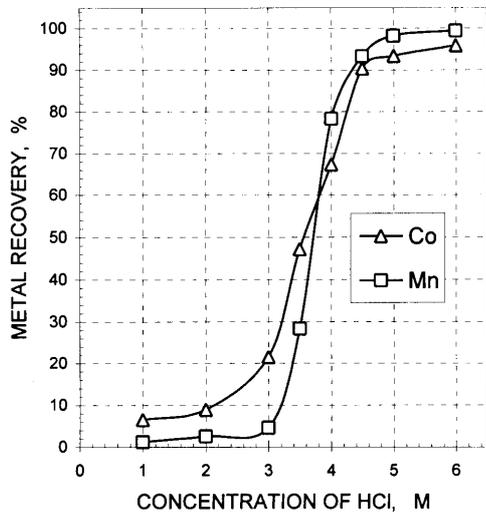


Fig. 5. Effect of concentration of hydrochloric acid on leaching recovery for Co and Mn.. (NaCl - 0.55 M, tem. - 30 °C, stirring rate 900 rpm, liquid/solid ratio - 10:1, leaching time - 1.5 hour)

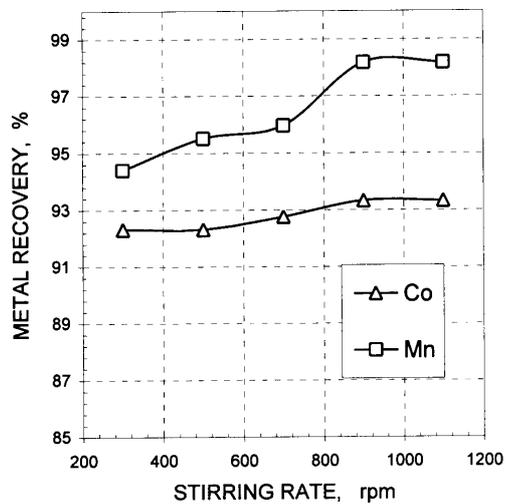


Fig. 6. Effect of stirring rate on recovery of Mn and Co from nodules (HCl + NaCl solution). (NaCl - 0.55 M, HCl - 5.0 M, temperature - 30 °C, liquid/solid ratio - 10:1, leaching time - 1.5 hour)

It was found that recoveries of cobalt and manganese are barely influenced by stirring rate. At 300 rpm more than 90 % of cobalt and manganese was leached out. These recoveries increased gradually with the stirring rate and almost all metals were transferred to the solution at stirring rate 1100 rpm under studied experimental conditions.

EFFECT OF LIQUID TO SOLID RATIO

Liquid to solid ratio in the leaching system was studied within the range from 2:1 to 10:1 (Figure 8). The experimental results revealed that liquid to solid ratio remarkably influenced the observed recoveries of metals from nodules leached with acidified "sea water". The decrease of liquid to solid ratio resulted in remarkable decrease of the recovery of cobalt and manganese. Only about 30 % of cobalt and manganese was recovered from nodules during the leaching when liquid to solid ratio was 2:1 (100 g nodules were leached in 200 ml of solution). There are two possible reasons for the observed effects:

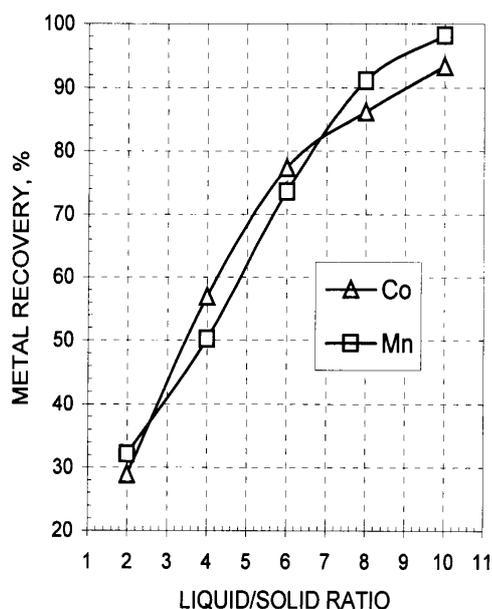


Fig. 7. Effect of liquid/solid ratio on recovery of Co and Mn. (NaCl - 0.55 M, HCl - 5.0 M, temperature - 30 °C, stirring rate - 900 rpm, leaching time - 1.5 hour)

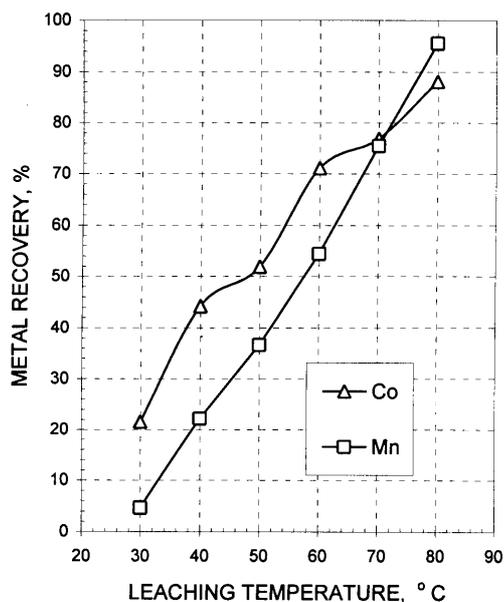


Fig. 8. Effect of leaching temperature on recovery of Co and Mn from nodules. (NaCl - 0.55 M, HCl - 3.0 M, leaching time - 1.5 hour, stirring rate - 900 rpm, liquid/solid ratio - 10:1)

- In a hydrochloric acid solution, insoluble Mn(IV) being in the form of MnO₂, must first be reduced to Mn(II). Then, remaining metals can be leached (there are 0.41 moles of manganese, 0.07 moles of iron, 0.002 moles of cobalt, 0.02 moles of nickel and 0.016 moles of copper in 100 g of nodules). To leach out the entire amount of these metals, 1.92 mol of Cl⁻ is theoretically required. At liquid to solid ratio 2:1, the amount of Cl⁻ available was 1.11 mol. This is only a half of Cl⁻ required to reduce Mn(IV) to Mn(II) and to leach all metals.)
- The Mn(IV) begins to be reduced to Mn(II) only at certain hydrochloric acid concentration. If concentration of hydrochloric acid is lower than this level, the Mn(IV) is hardly reduced to Mn(II). Consequently, copper, cobalt and nickel associated with manganese-containing minerals can not be extracted, except some copper, cobalt, and nickel from the surface of iron- and manganese- containing minerals.

On the other hand, at liquid to solid ratio of 10:1, the recovery of both Mn and Co from the feed nodules has reached 98 % and 93 %, respectively.

EFFECT OF LEACHING TEMPERATURE

The effect of leaching temperature on recoveries of Co and Mn from nodules is given in Figure 8. The observed relationships revealed that leaching temperature substantially influences the leaching of nodules. At temperatures ranging from 30 to 80 °C and with 3 M HCl in the model "sea water", the recovery of cobalt varied from 21.55 % to about 90 % while the recovery of manganese varied from 4.66 % to 95 %. These results based on the analysis of leaching residue (7.4 g) after leaching for 1.5 hour at 80 °C are given in table 7.

Table 7. Leaching recovery of metals in 3 M HCl at 80 °C.

Element	Cu	Fe	Mn	Ni	Co
Assay of residue, %	0.086	2.75	16.16	0.155	0.044
Recovery, %	97.98	82.24	82.36	96.56	91.65

From the experimental results it comes that concentration of hydrochloric acid in the leaching solution could be greatly reduced by means of raising the temperature of leaching. Based on above data, we have examined both two stage counter-current and multiple fresh solid leaching systems under following conditions: leaching temperature – 30 °C, stirring rate - 900 rpm, leaching time - 1.5 hours, 30 g of nodules, 300 ml of 0.55 M NaCl and 5.0 M HCl aqueous solution.

Semi two-stage countercurrent leaching of nodules reveals that analyses of the final liquid gave recoveries of 43.1 % for Co and 59.6 % for manganese. The results of these experiments, based on the analysis of final solid residue (28.39 g), are given in table 8 .

Table 8. Leaching recovery of metals in HCl – NaCl at 30 °C during two-step process.

Element	Cu	Fe	Mn	Ni	Co
Assay of Residue, %	0.23	3.08	19.47	0.48	0.07
Recovery, %	91.71	69.48	67.26	83.78	79.62

The multiple, four-stage leaching of fresh solid gave the final solution containing 0.22 g/L of cobalt and 33.5 g/L of manganese. Table 8 shows the weights of feed nodules and volumes of leaching liquid at each stage of the multiple-stage leaching experiment.

Table 9. Weight of feed nodule and volume of leaching liquid during the multiple-stage experiments

Stage number	I	II	III	IV
Feed nodule weight, g	30	29	28	27
Liquid volume, ml	300	290	280	270

CONCLUSIONS

- Concentration of hydrochloric acid greatly influences leaching recovery of metals from ocean nodules. At temperature of 30 °C, high recovery of metals from raw deep sea polymetallic nodules can be obtained by leaching them with hydrochloric acid solution of concentration exceeding 4.0 M.
- Concentration of hydrochloric acid in the leaching solution can be substantially reduced by raising the temperature or by adding sodium chloride.
- Leaching recovery of metals from raw nodules was barely influenced by stirring rate ranging from 300 rpm to 1100 rpm under experimental conditions.
- Copper, cobalt and nickel can be selectively leached from raw deep-sea polymetallic nodules with ammonia-SO₂ solutions or with H₂SO₄ under high temperature and high pressure. Both manganese and iron remain in the solid residue.
- Studied polymetallic nodules taken from Clarion/Clipperton Fracture Zone at northwestern Pacific exhibit somewhat different leaching behavior than the nodules reported in the literature. Though, general tendencies in metal leachability were similar.

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Przeprowadzono badania laboratoryjne nad wpływem najważniejszych parametrów na proces ługowania metali z rozdrobnionych, polimetalicznych konkrecji oceanicznych, pobranych z pola Clarion-Clipperton na Pacyfiku. Konkrecje te zawierają mangan i żelazo jako składniki główne zaś miedź, nikiel i kobalt stanowiły cenne do odzysku metale. Jako czynnik ługujący zastosowano tzw. „modelową wodę morską”, tj. zakwaszone roztwory chlorkowe. Określono wpływ warunków ługowania: czasu, temperatury, szybkości mieszania zawiesiny, stosunku masowego fazy ciekłej do stałej (l/s) oraz stężenia HCl na wydajność szybkość ługowania.

Przeprowadzone badania wykazały, że stężenie kwasu solnego jest parametrem, który najbardziej wpływa zarówno na szybkość jak i na wydajność ługowania metali z badanych konkrecji oceanicznych. Dla stężenia HCl powyżej 4.0 M i w temperaturze 30 °C obserwowano prawie całkowite przeprowadzenie metali z surowca do roztworu po ok. 30 minutach ługowania. Podwyższenie

temperatury ługowania do 80 °C pozwalało uzyskać znaczne podniesienie szybkości procesu i istotnie zredukować stężenie kwasu solnego w roztworze. Zaobserwowano, że zmiana stosunku fazy ciekłej do stałej w zakresie od 3 do 10 prowadziła do wzrostu wylugowania z 30 do 93-97 %. Porównanie uzyskanych wyników z danymi literaturowymi pozwoliło stwierdzić, że choć badane konkreje oceaniczne zachowywały się podczas ługowania chlorkowego nieco odmiennie w porównaniu z konkrejami opisywanymi w literaturze, to ich podatność na ługowanie w badanych warunkach daje możliwość odzyskania zawartych w nich metali.