

CARBONYLATION AS A SEPARATION TECHNIQUE FOR REMOVAL OF NONRADIOACTIVE SPECIES IN CONJUNCTION WITH CALCINE DISSOLUTION PROCESSING OF HANFORD TANK WASTE

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ABSTRACT

Much of the waste generated from five decades of weapons production in the U.S. Department of Energy complex contains highly radioactive constituents. With the high cost of permanent disposal space, it is necessary to separate as many of the nonradioactive species from the radioactive as possible. This paper discusses the transfer of carbonyl processing technology from mineral beneficiation and powder metallurgy operations to the separation of Fe and Ni from radioactively contaminated waste streams. Samples of simulated composite Hanford Tank Waste residue were first processed with a calcine/dissolution technique which resulted in a residue powder consisting of 31.9 pct Fe and 3.3 pct Ni. Because of the specification for waste glass compositions, these two constituents become important in determining the number of waste glass logs produced. Pyrometallurgical reduction of the residue powders, followed by subsequent carbonylation, extracted up to 92.0 pct of the Fe and 95.7 pct of the Ni. The resultant product contained as little as 4.9 pct Fe and 0.3 pct Ni. At this level, Fe would no longer be a limiting constituent in the waste glass.

INTRODUCTION

Much of the waste generated from five decades of weapons production in the U.S. Department of Energy complex contains highly radioactive constituents. Large amounts of this waste are in the form of tank waste consisting of salt cake, sludge and supernate liquid containing significant quantities of Fe, Ni, and Bi. With the high cost of permanent disposal space, it is necessary to separate as many of the nonradioactive species

away from the radioactive species as possible. This paper discusses the transfer of carbonyl processing technology from mineral beneficiation and powder metallurgy operations to the separation of Fe, Ni, and Bi from simulated composite Hanford Tank Waste residue (SHTWR) based on pioneering test results. Figure 1 presents the concept of high- and low-level waste separation using calcine/dissolution processing followed by carbonyl treatment.

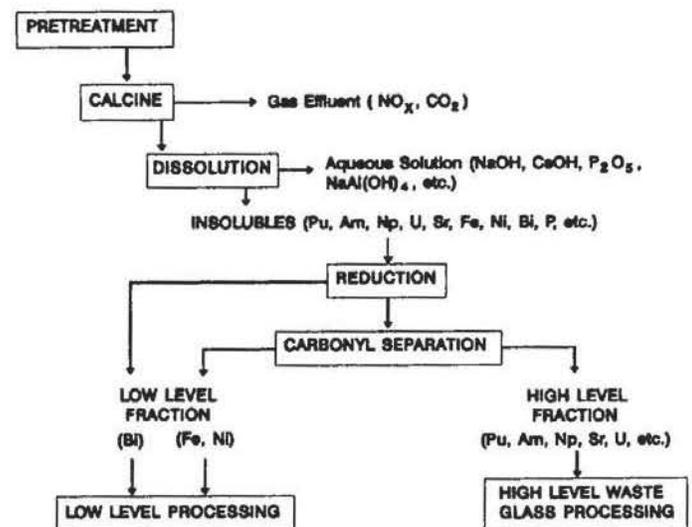


Figure 1 - Simplified flow diagram for the calcine/dissolution concept with carbonyl separation.

Today, carbonyl technology is commercially applied to recover Ni metal from concentrates and to form Ni and Fe powders, which serve as feed stock for powder metallurgy. In addition, research has been conducted to indicate that this process can be applied to beneficiate chromite concentrates and upgrade ilmenite ores to

synthetic rutile through the removal of excess Fe.¹⁻² This latter application is most similar to radioactive residue processes for removal of Fe and Ni because of the similarity in the free energy of formation for the oxides of Cr and Ti, and radionuclides such as Pu, Cs, Np, Sr, and Am, in that these radionuclides (1) are not easily reduced to the metallic state and (2) do not tend to form volatile carbonyls. In the beneficiation investigations,¹⁻² it was possible to extract significant quantities of Fe from the ores by carbonylation after pyrometallurgical reduction. The process was able to obtain an increase in the Cr:Fe ratio by a factor of three¹ and extract over 90 pct of the Fe from ilmenite to convert it to synthetic rutile.² The limiting step in Fe removal appeared to be solid-state reduction of iron oxide to metallic Fe rather than the carbonylation step.

Carbonylation was conducted by injecting high-pressure CO into a reaction chamber at relatively low temperature (140° C) with a chemical promoter to form volatile carbonyl compounds from contact between the metal surfaces and the gas. Formation of volatile carbonyl compounds is limited to Fe and Ni, and possibly Cr and Co, because of the highly selective nature of the carbonyl reaction. It is necessary to reduce the contained oxides of Fe and Ni to the metallic state by reductive roasting prior to carbonylation. The resulting carbonyls are vapor transported out of the pressure reactor into a decomposition chamber. Here the carbonyls are disassociated to metal and CO at higher temperature (300° C), but lower pressure, and the metal plates out on available surfaces. The CO can either be recycled or burned to form CO₂, similar to a wood-burning stove. Safe handling techniques and equipment have been developed for the toxic CO and carbonyls³ and this equipment appears adaptable to the facilities designed to handle the more permanent and severe hazards associated with radioactive waste.

The objective of carbonyl processing of radioactive waste is to form gaseous carbonyls of the nonradioactive metallic species and vapor transport them to a decomposition chamber. The high-level waste, at reduced volume, remains as the residue. It is anticipated carbonylation will lead to (1) large decontamination factors because radioactive species are chemically different from Fe and Ni, (2) simple, flexible, low-temperature

treatment consistent with hot-cell operations, and (3) minimum secondary waste streams since the CO effluent can be easily treated.

EXPERIMENTAL PROCEDURE

Simulated composite Hanford Tank Waste residue from the calcine/dissolution process was prepared at Westinghouse Hanford Company according to procedures developed by Colby⁴ and Delegard.⁵ The mixed chemicals were calcined at 850° C for 2 h, washed in water, and the insoluble portion filtered and dried. The recipe was formulated on the assumption that calcination would eliminate nitrates and nitrites as well as any organic carbon. This formulation is based on composite amounts of species in the waste tanks. However, after calcine/dissolution the waste residue is similar to a Bi phosphate residue. Table 1 summarizes the composition of SHTWR.

Precarbonylation treatment of SHTWR consisted of solid-state reduction with H₂ at 650-, 850-, and 1,000° C. No evidence of residue melting was observed, but some sintering was apparent after the 1,000° C reduction, although the sample crumbled readily during handling. All reductions were performed in a large high-temperature H₂ reduction furnace. The charges were loaded into a cool zone of the heated furnace, the furnace was purged with He, the charges were then pushed into the hot zone and the He switched to H₂ at 12 standard liters/minute (SLM). At completion the procedure was reversed except the sample was moved gradually into the cooling zone and allowed to cool to ambient temperature before removal.

Carbonylation tests were performed in 4.5-L volume, high-pressure reaction vessel made of type 410 stainless steel.¹ A nominal 0.15-SLM CO flow was maintained through the reactor to vapor-transport part of the metal carbonyls from the reactor. The gases were passed at atmospheric pressure through a 30-mm-diameter borosilicate tube maintained at 300° C in a 450-mm-long tube furnace. Iron and Ni carbonyls decomposed in the furnace hot zone back to CO and the respective metals, which plated out on the borosilicate tube. Figure 2 is a schematic drawing of the experimental carbonyl processing system.

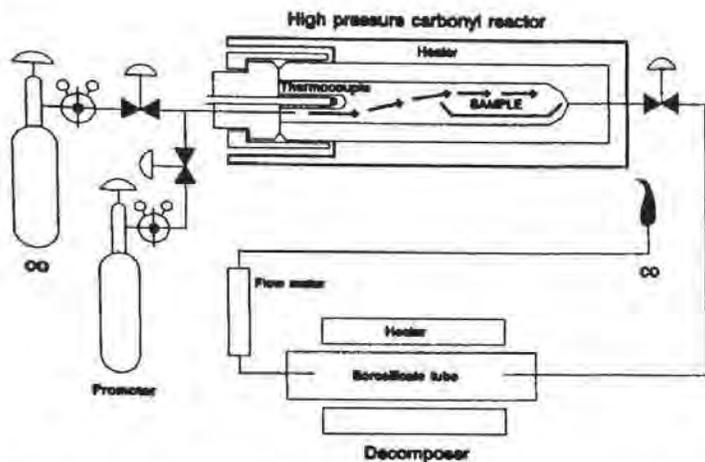


Figure 2 - Experimental carbonyl processing system.

The amount of metal extracted by carbonylation was calculated from charge weight loss and by chemical analysis of the residual charge. Total metal in the samples was determined by inductively coupled plasma (ICP) emission spectrometry. The metal that plated out in the decomposer on the borosilicate glass also was examined by ICP to determine if any elements besides Fe and Ni were transported out of the reactor in the CO stream.

RESULTS AND DISCUSSION

Test parameters and the resulting weight and composition changes resulting from solid-state reduction and carbonylation SCHTWR are summarized in table 2. Weight loss from reduction at all three temperatures is considerably more than would be expected from reduction of just the Fe and Ni oxide fractions listed in table 1. Even the additional loss of Bi oxide by reduction/volatilization does not account for the observed total mass loss. It is thermodynamically unfavorable for the other oxides to be reduced at these temperatures as indicated by the Ellingham diagram, figure 3. The reduction weight losses in table 2 in conjunction with the composition defect of 17.42 pct in table 1 indicate that the surrogate residue is not completely dewatered during filtration and drying. Loss of the retained moisture and volatilization of Bi at the higher reduction temperature then account for the observed high weight losses during reduction. Retention and possible pick-up of moisture during handling between drying and reduction, or reduction and carbonylation, would tend to lower the composition values determined by ICP.

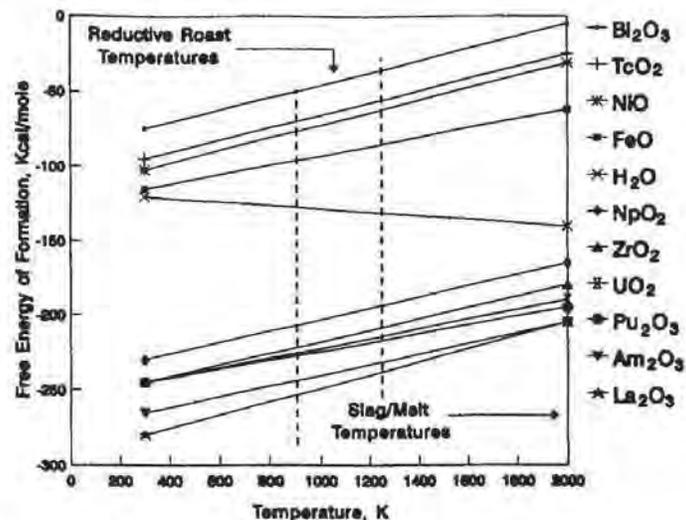


Figure 3 - Ellingham diagram for selected elements.

The initial carbonylation test was performed on surrogate residue reduced at 850° C. A 4-h carbonylation time and H₂S promoter to Fe mole ratio of 1:20 was used, based on optimum mineral beneficiation data.¹ The result was a rather low 4 pct weight loss corresponding to an estimated 9.7 pct combined Fe and Ni extraction. One-third of the residue was further carbonylated for 66 h without promoter and resulted in a 33.3 pct weight loss. Combined, the two carbonylations produced a 35.8 pct weight loss. The data in table 2 are for the two carbonylations combined. The reason for the failure of the H₂S promoter to accelerate the carbonyl reaction comparable to that observed in previous mineral beneficiation application is not clear at present.

Surrogate residues reduced at 650- and 1,000° C were carbonylated simultaneously for the extended time. Two separate charges from each reduction were treated, with one from each reduction retained in the alumina boat without any disturbance prior to carbonylation. The data in table 2 are for charges that were pulverized between reduction and carbonylation, but the weight losses and analysis of the final residue for the other two undisturbed charges were essentially identical. Carbonylation weight loss was in excess of 30 pct for all samples. The lowest residual Fe and Ni content and best metal extraction was realized on surrogate residue reduced at 650° C. This indicates that 650° C is sufficient to metallize essentially all the Fe and Ni oxides, but does not rule out the possibility that even lower reduction temperatures may be

Table 1 - Simulated composite Hanford Tank Waste residue

Oxide	Fe ₂ O ₃	Bi ₂ O ₃	NiO	P ₂ O ₅	SrO	ZrO ₂	SiO ₂	Na ₂ O	CeO ₂	CaO	Al ₂ O ₃	MnO	SO ₃	Total
Wt pct	35.04	8.65	3.17	3.01	4.25	5.85	4.04	3.27	6.03	3.03	2.13	3.78	0.13	82.58

Table 2 - Summary of Fe, Ni, and Bi composition changes during solid-state reduction and carbonylation of simulated composite Hanford Tank Waste residue

Solid-state reduction			
Temperature, °C	650	850	1,000
Charge weight, g	10.22	64.8	10.31
Time, h	6	4	4
Weight loss, pct	17.6	34.9	31.9
Carbonylation			
Temperature, °C	140	135	140
Time, h	75	70	75
Pressure, psig	1,500	1,500	1,500
Weight loss, pct	34.2	35.8	37.9
Iron content, wt pct			
Starting	31.9	29.7	31.9
After reduction	40.5	49.6	49.6
After carbonylation	4.9	11.3	13.6
Nickel content, wt pct			
Starting	3.3	3.0	3.3
After reduction	4.0	4.5	4.5
After carbonylation	0.4	0.3	2.2
Bismuth content, wt pct			
Starting	10.3	9.6	10.3
After reduction	12.9	ND	0.5
After carbonylation	16.9	ND	0.8
Extraction, pct			
Iron	92.0	84.6	86.4
Nickel	93.4	95.7	69.6
Bismuth ¹	NA	ND	96.7

ND Not determined.

¹Extracted by volatilization during solid-state reduction.

NA Not applicable.

Table 3 - Elements in plated out metal

Element	Fe	Ni	Bi	Ca	K	Mg	Na	S	Si	Zn	Ce	Sr	Zr
Wt pct	85.7	13.0	0.1	0.1	0.5	0.1	0.2	0.8	0.2	0.1	UD	UD	UD

UD Undetectable.

Table 4 - Distribution of elements in residue before and after carbonylation experiments

Element	Mn	Na	S	Al	P	Si	Ca	Sr	Zr	Ce
SCHTWR ¹ , wt pct	2.9	2.9	0.06	1.3	1.7	2.3	2.7	4.4	5.2	5.3
Carbonylated, wt pct										
650°C reduction	0.5	1.5	1.5	1.9	2.8	2.7	3.8	7.1	6.5	14
1,000°C reduction	0.5	0	0.9	2.1	3.3	3.3	4.5	8.7	9.6	14

¹Table 1 analysis normalized.

feasible. No Bi reduction or volatilization is evident at 650° C.

Reduction weight loss at 850- and 1,000° C was considerably higher compared to 650° C, but the carbonylation residues had higher Fe and Ni fractions and showed reduced metal extraction percentages. Most of the additional weight loss is obviously due to removal of the Bi by reduction and/or volatilization as indicated by the Bi content dropping below 1 pct after reduction at 1,000° C. Nearly 97 pct of the Bi was missing from surrogate residue reduced at this temperature.

While higher reduction temperatures would be expected to enhance metallization of Fe and Ni, two factors resulting from the elevated temperature appear to hinder carbonyl formation. As temperature increases, sintering becomes more prominent and this inhibits the carbonyl reaction due to reduced metal surface area and possible fusion with other constituents in the charge. Agglomeration, although not severe, was observed in waste reduced at 1,000° C, but not at the other temperatures. Second, accelerated diffusional alloying of Fe and Ni at the higher temperature would tend to be more resistant to carbonylation than separated pure metals.

The metal plated out in the decomposer from the CO stream flowing through the reactor during carbonylation and depressurization analyzed 85 pct Fe, 13 pct Ni, and 2 pct minor elements, as summarized in table 3. All the metal carbonyls formed during the test passed through this decomposer with an approximate 2 min residence time. This was sufficient to decompose all the carbonyls, as evidenced by a blue colored CO safety flame and absence of metal plating on the glass at the outlet-end hot zone of the decomposer. The data in table 3 shows all the minor elements are at concentrations below 1 pct. Use of H₂S as the promoter would account for the S and the borosilicate decomposer glass may contribute some other elements such as Si. No surrogates for the radioactive elements (Ce, Sr, Zr) were detected even though they were present in substantial quantities in the starting and carbonylated SHTWR, as indicated by the data in table 4, which summarizes the concentration change of elements other than Fe, Ni, and Bi in the SHTWR.

CONCLUSIONS

Laboratory tests have shown that carbonyl processing techniques developed for mineral beneficiation can be applied to SHTWR to extract Fe, Ni, and Bi. Radioactive element surrogates in the waste were not extracted and remain in the residue. Free energy of formation data indicate that transuranics, strontium, and mixed fission products would behave similarly because they do not reduce to metal and oxides do not directly form carbonyl compounds. Pyrometallurgical reduction of the residue powders, followed by subsequent carbonylation, extracts in excess of 90 pct Fe and Ni. The resultant product contains less than 5 pct Fe and 0.50 pct Ni. At these levels, Fe and Ni are no longer limiting constituents in vitrification of the high-level waste for permanent storage. Bismuth and other volatile elements can be extracted by condensation in the reduction step. While minor quantities of Fe, Ni, Bi, and other element radionuclides may be present in the extracted products, it can be treated as low-level waste. These results show that reductive roasting and carbonylation hold promise as a separation technique for removing Fe, Ni, and Bi from high-level wastes to produce a significant reduction in volume.

The potential for direct metal deposition into net shapes by the carbonyl process is another interesting aspect of this approach. Besides the commercial powder synthesis, it has been possible to deposit tubes, flat plates, and even ceramic-fiber composites with this process.⁶ Hence, it could be feasible to fabricate, in an immediate fashion, uncontaminated metal components from contaminated metal debris. Potentially, waste containers could be deposited in molds at the decontamination facility without the necessity of hands-on or robotics metal forming. Virtually any waste component capable of tolerating 300° C can be directly encapsulated within the new containers, without welding or brazing.

The further development of the carbonyl process for metal/radionuclide separation requires focusing on several issues: (1) Decontamination factor determination, (2) Process acceleration with the help of promoters and other techniques, and (3) System design

interface with the decontamination and decommission activities. Radioactive testing of this process on actual Hanford Tank Waste is in progress.

The process provides an efficient high-level/low-level separation between transuranics, strontium, and mixed fission products and Fe, Ni, and Bi. It will reduce the amount of waste glass required because of reductions in elements cited in glass specifications; i.e., Fe, Ni, and Bi. It will not have a substantial impact on secondary waste streams because H₂ and CO can be recycled or easily separated. The technology is well developed without significant deployment issues and appears to be compatible with remote processing because of its simplicity, low temperatures, and limited moving parts. Carbonylation should not significantly change the acid dissolution characteristics of the original calcine/dissolution residue so that if subsequent treatment requires acid dissolution, the same steps could be repeated, but on a reduced volume of material.

ACKNOWLEDGEMENTS

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FOREWORD

The organizing committee is pleased to provide the proceedings of the fifth biennial Spectrum meeting, Spectrum '94. The theme of this meeting was "Continuing the Job of Environmental Restoration and Compliance." The meeting was held in Atlanta, Georgia, August 14-18, 1994.

Spectrum '94 was an international technical topical meeting. The technical program committee was charged with collecting stimulating technical papers emphasizing the heavily regulated area of environmental restoration and waste management, where much of the activity and many of the problems find their genesis and their solution in policy or legal action. As a result, there are many classes, tutorials, and meetings that appropriately address legal, political, and policy issues. Spectrum '94, however, was formulated with the technologists and their changing world in mind. The program was devised to facilitate technical exchanges between scientists and engineers while providing an element of interface with the changing world and with those who live in it and must accept and believe in the science and technology that are being applied and practiced by the professionals around the world. The international flavor of the meeting was manifest in the 519 papers, 160 of which were offered by non-U.S. authors representing 32 countries on 6 continents!

The plenaries, Impacts of Enhanced Trade Agreements on Commercialization of Environmental Technologies in the Global Market and Gaining Consensus for Regulatory and Environmental Change, were the notable exceptions to the technical nature of the meeting. In these cases, political personalities were invited to provide what were essentially tutorials for technical people to deal with some of the major nontechnical issues of the day. The sessions were organized to provide balance and understanding and to remind the meeting participants that purely technical solutions are not sufficient for problems in international environmental restoration and waste management.

The organizing committee would like to express thanks to the meeting sponsors: the American Nuclear Society (ANS), the Fuel Cycle and Waste Management Division of the ANS, the Savannah River and the Atlanta Sections of the ANS, the European Nuclear Society, the Canadian Nuclear Society, the Atomic Energy Society of Japan, the International Atomic Energy Agency, and the U.S. Department of Energy Environmental Management Office of Technology Development. Thanks are also extended to the U.S. Department of Energy, which provided the financial support for the preparation and printing of these proceedings.

Our special thanks go out to the large volunteer effort of the members of the various committees who gave their free time to make this meeting a success. These people are the key to organizing a meeting of this type, and their willingness, energies, and abilities in dealing with the problems and issues involved are greatly appreciated. With regard to the proceedings, special thanks go to the administrative staff at Westinghouse Savannah River Company, including Pamela Sapp, Laura Jordan, and Stephanie Doetsch; and to the ANS Scientific Publications Department staff.

These proceedings are the legacy left by the authors. The committee hopes that the documents will find regular use as technical references, which will be the very best way to reward the authors for their efforts.

Clyde W. Frank
Cochair

John L. Steele
Cochair

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*Paper was not available at publication time.

NEW PROCESSES AND TECHNOLOGIES: HIGH LEVEL WASTE

Table 3. Batch adsorption data showing the effect of mixing time on the removal of cesium from MVST W-25 supernate^a

Sample ^b	Mixing Time (h)							
	(2)		(24)		(72)		(144)	
	D	%R	D	%R	D	%R	D	%R
Duolite CS-100	35	20.0	34	15.0	42	20.0	44	22.0
SRR	763	79.3	736	79.5	764	79.2	641	78.7
CST	662	77.4	672	77.5	672	77.7	958	83.8
KCoFC ^c (Gr.)	46,200	99.6	36,900	99.5	36,300	99.5	26,000	99.3
Blank tube		0		0		0		

^aEach batch test was conducted by mixing 50 mg of adsorber with 10 mL of MVST W-25 supernate for the mixing times indicated. D = distribution coefficient in (mL/g), and %R = percentage removal.

^bSRR - formaldehyde/resorcinol resin (425 to 595 μm); CST - crystalline silicotitanate (fine powder); and KCoFC - granular potassium cobalt hexacyanoferrate (250 to 595 μm).