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Chattanooga Shale — An Important U.S. Mineral Resource

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ABSTRACT

Chattanooga shale, a major component of the eastern Devonian shales, covers a wide area from lower Kentucky through Tennessee and into northern Alabama. It contains sizable resources of oil and several critically needed metals. Leaching experiments with sulfuric acid have demonstrated that excellent trace metal solubilization can be obtained from hydroretorted Chattanooga shale, i.e., the solid residue remaining after the raw shale has been processed to produce oil and/or gas.

INTRODUCTION

The present situation on imported oil is a reminder that the U.S. is also dependent on foreign sources for several other strategic resources. Recent investigations(1,2) have emphasized the lack of U.S. reserves, resources, and production capacity for supplying certain essential metals. Future supply and demand figures for non-fuel minerals(3) reemphasize the vulnerability of the U.S. in these areas. The U.S. is totally dependent on imports for supplies of chromium, cobalt, tantalum, and tin (Table 1). Except for cobalt, even resources of these metals are extremely

Table 1. Elements Critical to U.S. Needs[a]

Element	Reserves[b]	U.S. Mining Capacity[c]	Resources[b]	Percent Imported, Primary	Import Sources 1976–1979	Stockpiles (1978)[b]	
						Government	Industry
Cr	0	0	3	100	S. Africa Rhodesia Turkey	2	1
Co	0	0	70	100	Zaire Zambia Finland	2	0.4
Ta	0	0	2	100	Thailand Canada Malaysia	1	1
Sn	1	<1	4	>99	Malaysia Thailand Bolivia	3	0.3
Ni	1	15	70	92	Canada	0	0.2
Nb	0	0	40	100	Canada Brazil	0.5	1
Mn	0	0	50	100	Gabon Brazil Australia	2	0.7
Al	2[d]	5	10	93	Australia Jamaica Guinea	1	0.5
Pt	0	<1	100	>99	S. Africa U.S.S.R	0.5	0.5
Sb	5	30	7	94	Bolivia China Mexico	1.5	0.5

[a] Data from Commodity Data Summaries, Bureau of Mines.

[b] Years of supply, based on annual U.S. demand (1978).

[c] Percent based on annual U.S. demand (1978).

[d] Based on bauxite and alumina.

limited. The U.S. also imports large amounts of aluminum, antimony, manganese, nickel, niobium, and the platinum metals. Although the U.S. possesses long-term resources of these critical elements, they are imported mainly for economic reasons.

Various avenues for supplying these critical metals from such materials as fly ash, coal conversion wastes, clays, shales, etc., have been examined. One potential route to the domestic production of some of these metals involves their extraction as byproducts from shale-oil wastes. Both western(4-7) and eastern(8)

(Devonian) shales have been analyzed for element content; the former sometimes contain anomalously high contents of some elements in very localized areas. By way of contrast, the eastern Devonian shales are spread over much wider areas than the western oil shales and apparently contain relatively more uniform contents of some of the above critical metals. One major component of the eastern shale, known as the Chattanooga, which covers a wide area extending from lower Kentucky through Tennessee and into northern Alabama, contains sizable, but very low-grade, resources of several of the critically needed metals. It should be emphasized that, apart from the economic factors involved, self-sufficiency in these critical materials would be very desirable because some of these metals are imported from geographic areas of questionable political stability. Furthermore, future OPECs in mineral areas could be avoided by our ability to generate these critical materials from our own resources, even if they are low-grade and uneconomical to recover at the present time. For these reasons, an exploratory experimental investigation was initiated to determine the feasibility of extracting some of these metals from eastern Devonian shales. Samples of both raw and hydroretorted Chattanooga shale (shale heated under hydrogen pressure to recover organic carbon as either gas or oil) were leached with sulfuric acid to recover "critical" materials such as cobalt, chromium, nickel, etc. Excellent trace metal solubilization was obtained, as determined by the inductive coupled plasma method of analysis.

Chattanooga Shale — A Source of Critical Elements

During the 1944—1960 period, considerable effort was expended in investigating the Chattanooga shales, mainly for their uranium content. Although of low grade ($\sim$60 ppm), it is estimated that deposits in 12 counties of Tennessee alone contain several million tons of uranium. The highest mineral content is apparently contained in the counties located near or on the Eastern and Northern Highland Rim of the Cumberland Plateaus in Tennessee. The early investigations(8,13,16-20) were summarized recently by Mutschler, Hill, and Williams.(9)

Although the discovery of extensive uranium deposits in the western sandstone areas of the U.S. lessened interest and work in the Chattanooga shale during 1960–1975, depletion of the higher grade U.S. reserves along with growth of the nuclear power industry has renewed interest in the lower grade resources. A recent study(10) by the Mt. States Mineral Enterprises, Inc., commissioned by the Department of Energy, indicated that a coproduction plan involving the production of synthetic oil from shale by a new process ("Hytort")(11,12) along with uranium production and possibly other metal byproducts, was not only feasible but economically viable. The "Hytort" process, developed by the Institute of Gas Technology (IGT), is a method of increasing the oil yield from shale by retorting the shale under a hydrogen pressure of 300–500 psi at temperatures of 540–800°C (1000–1500°F). Estimates of the oil resources available in the eastern shales recoverable by aboveground retorting by this process amount to over 400 billion barrels.

The amounts of major materials and elements occurring in Chattanooga shale are:(9) [1] Minerals — total silicates (which includes quartz, 20–30%) average 65–70%; pyrite and marcasite, 5–10%; organic matter, 10–20%. [2] Elements — aluminum and iron, 5–8%; potassium, 3%; calcium, magnesium, sodium, titanium, 0.4–0.6%; sulfur, 4–8%, and phosphorus, 0.15–1%. In Table 2 values are listed for the minor constituents found in the Chattanooga and other Devonian shales. The trace element concentrations reported by O'Neil,(13) which were used as a basis for byproduct recovery value by Mountain States, appear to be too high for several elements, notably cobalt, nickel, vanadium, and much too great for niobium. These observations are based on recent analyses of shale samples performed at ORNL and by the U.S. Geological Survey,(14,15) using more refined analytical techniques.

Generally the Tennessee samples not only possess the highest concentrations of desirable metals but also among the highest organic carbon content, although the element content varies from site to site and vertically within a particular drill hole. IGT has indicated that they are using organic carbon content in excess

Table 2. Constituents Present in Devonian Shales

Location (county, state)	Investi-gator[a]	Percentage				Parts per million										
		Organic C	S	Al	Fe	Co	Cu	Cr	La	Mn	Mo	Ni	Nb	U	V	Zn
DeKalb, TN	O'N	10	5.5	6	5.5	230	200	100	n.d.	500	300	500	630*	60	800	500
DeKalb, TN	L	14	7		7	70	150	30	n.d.	150	150	150	n.d.	100	150	<300
DeKalb, TN	IGT,[b] ORNL	14	4.8	6	7	55	130	150	35	150	260	200	10	75	280	315
Overton, TN	L	9	4.6	6	4	55	120	n.d.	n.d.	500	80	210	10-20	44	325	360
Christian, KY	ILL	7	1.5	6	3.5	16	150	80	n.d.	300	100	140	n.d.	20	290	210
Martin, KY	L	2.2	2.1	7.5	6	21	*	n.d.	n.d.	*	30	87	<10	11	160	n.d.
Perry, KY	L	4.3	2.3	7.5	6	21	110	77	70	200	50	110	24	16	360	240
Rowan, KY	L	10.5	5.6	6	6	57	74	70	50	390	120	130	n.d.	56	160	76
Bullitt, KY	IGT	13.2	5.3	5	5.9	20	93	81	n.d.	180	160	140	10	50	190	100
Lewis, KY	IGT,[b] ORNL	13.8	2.7	7.5	5.5	20	140	110	35	1100	220	280	10	40	1700	1600
Carroll, OH	L	2.4	2.1	7	4	22	67	n.d.	n.d.	280	29	100	10-20	10	200	100
Wash., OH	L	1.2	2.0	9	4	24	46	n.d.	n.d.	370	12	80	10-20	7	160	100
Jackson, W.VA	L	2	1.5	7	6	20	110	80	70	390	33	82	26	9	170	95
Lincoln, W.VA	L	3	2.3	7	7	26	140	140	70	190	48	155	22	12	310	120
Wise, VA	L	2.8	2.3	8	5	n.d.	53	n.d.	n.d.	*	26	110	15	13	275	n.d.
Sangamon, IL	ILL	<1	<1	6	6	20	40	100	n.d.	300	50	85	n.d.	10	240	120
Cattaraugus, NY	L	1.5	1.5	8	5	18	80	110	70	540	21	90	24	7	214	145

[a] Investigator and/or Laboratory

O'N — O'Neil, thesis, PA State Univ., State College, PA (1956).
L — Leventhal, U.S. Geol. Survey, Denver, CO (1978–1979).
IGT — Inst. of Gas Tech., Chicago, IL (1979)
ORNL — Oak Ridge National Laboratory, Oak Ridge, TN (1980).
ILL — State Geol. Survey, Urbana, IL (1977).

[b] Organic carbon and sulfur determined by IGT, remaining analyses determined by ORNL.

n.d. — not determined.
* — analyses not meaningful.

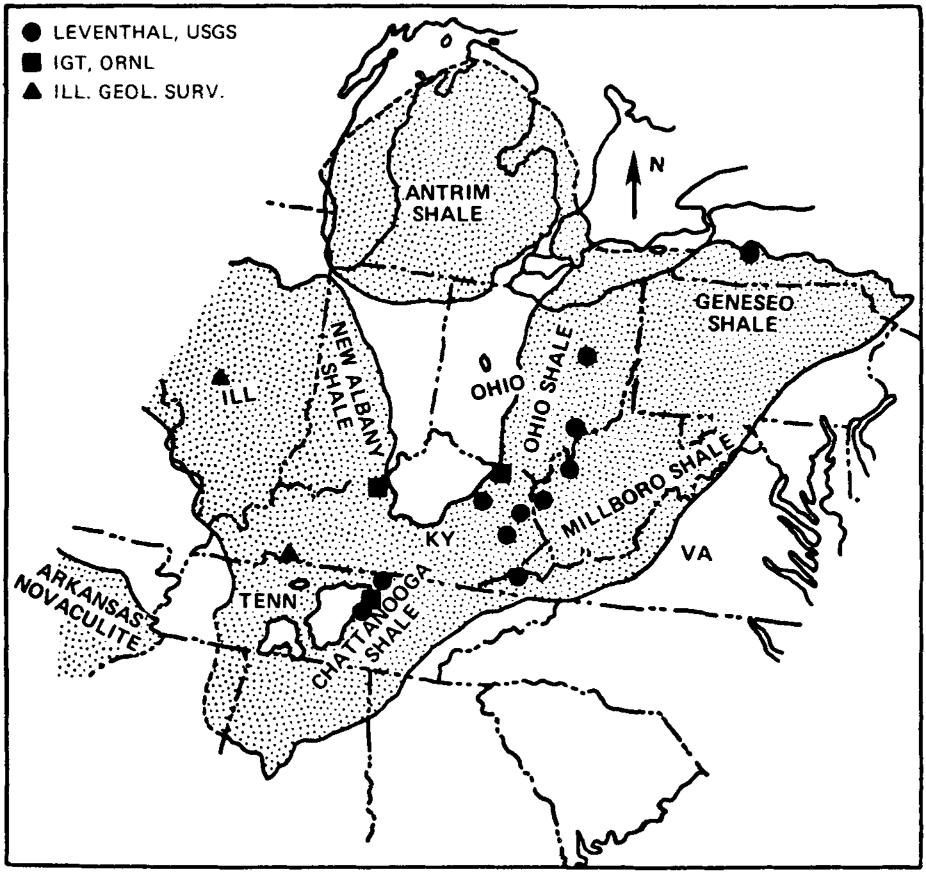

Figure 1. Locations of samples analyzed for organic carbon and other elements in Eastern U.S. Devonian shales.

of 10% as a screening value for the most desirable shales from an oil-producing standpoint. Leventhal(14,15) has presented downhole plots which graphically reveal the correlation between organic carbon content, sulfur, and metallic constituents in the shales. Figure 1 shows the geographical distribution of the various shale samples analyzed and detailed in Table 2.

Previous leaching studies(16-20) on the Chattanooga shales were performed for the primary purpose of extracting uranium. Two processes showed promise: (1) using dilute sulfuric acid as the dissolvent in a countercurrent mode; (2) using oxygen pressure in which leaching and acid production are simultaneously achieved. Since our experiments were aimed at leaching metals from hydroretorted shale, it appeared that using the oxygen pressure leach method would have defeated this purpose as it would have reoxidized the metals previously reduced during the retorting process.

Other investigators have tried nitric, hydrochloric and sulfuric acid as lixivia on coal fly ash.(21) Their studies indicated hydrochloric acid was somewhat better than sulfuric acid for recovering metallic constituents; however, the high sulfur content present (4–8%) in Chattanooga shale lends itself to the production of sulfuric acid on site, thus reducing process costs. Apart from the difference in cost between using hydrochloric rather than sulfuric acid, use of the former would require more expensive corrosion-resistant equipment.

Experimental Methods

For simple laboratory experiments, weighed portions of raw and retorted shale were refluxed in 4M sulfuric acid for eight hours. The leaching solutions were filtered from the solid residues and analyzed for the desired metallic constitutents by the Inductive Coupled Plasma (ICP) method of analysis. The washed residues were dried, heated in a muffle furnace at 500°C to burn off the filter paper and any remaining organic matter, and dissolved in a mixture of nitric and hydrofluoric acids. The resulting solutions were analyzed for the same constitutents sought in the leach solutions. Material balances were calculated from these data and analyses of the original raw and retorted shales (six samples – three of each). These were treated by an $HF-HNO_3$ vapor phase technique(22) to dissolve essentially the entire sample. Leaching experiments were run on only three of the six original samples, one raw and two hydroretorted. However, experiments were performed on two different sized materials – finely crushed (through 200 mesh) and 4–16 mesh to determine the effect of size on leachability.

Additional experiments to determine the effect of "roasting" temperature on leachability were performed in a similar matter. Initially, both raw and hydroretorted shale samples were heated in the muffle furnace at various temperatures for three hours before leaching. Later, hydroretorted shale samples were roasted at 800° and 900°C for only 30 minutes before leaching.

Other methods of recovering oil from the Chattanooga shale are being investigated. These include steam-oxygen gasification, hydrogasification and/or spent shale combustion. If an oxidizing atmosphere is employed, then sulfur dioxide (convertible to sulfuric acid) will be produced and the metal species present in the shale will be converted to oxides, similar to what is produced when the shale is roasted after the hydroretorting. It is important that the temperatures involved in these processes should be kept below 800°C so that the remaining silicate structure will retain its porosity,(17) thus providing a readily leachable solid.

Hydroretorting and leaching dissolves 40—45% by weight of the original raw shale, leaving a residue which largely consists of quartz and some remaining silicate. This residue is quite friable, even upon hand crushing. Preliminary tests indicate that compaction may be an effective step in disposal of the shale residue.(23)

RESULTS AND DISCUSSION

The data summarized in columns 1—3 and 5 of Table 3 were obtained from two sets of essentially duplicate experiments. Agreement between the solubility yields from the two sets of experiments was very good. Except for uranium, recovery of every constituent was better from hydroretorted shale than from the raw shale, and in some cases extraordinarily so. In the case of uranium, the drop in solubilization from raw to hydroretorted (not roasted) shale was 78 to 73.4% (average recovery from both sizes); this may be caused by some reduction of uranyl ion (VI) to uranous (IV) during hydroretorting, and the former would be more soluble in sulfuric acid solutions. The analytical data supplied by the

Table 3. Observed Percentage Elemental Recovery from Chattanooga Shale by Leaching with 4M H_2SO_4 (8-hr Reflux) — with and without Roasting

Element	–200 Mesh Shale		4–16 Mesh Shale, Roasting Temperature[a] (°C)							
	Raw	Hydroretorted	Raw		Hydroretorted					
	RT	RT	RT	500	RT	500	800	800[b]	900[b]	1000
Aluminum	61	73	39	65	78	77	70	74	65	12
Iron	26	99	21	95	99	99	96	98	96	84
Cobalt	35	97	35	100	100	100	97	99	96	27
Chromium	69	76	55	64	77	84	76	84	58	25
Copper	30	57	28	89	30	88	79	86	68	12
Manganese	71	90	59	92	92	92	81	90	80	43
Molybdenum	41	48	30	97	51	98	95	96	87	54
Nickel	38	93	35	97	95	97	90	92	88	29
Uranium	79	77	78	87	70	82	64	71	54	18
Vanadium	50	90	32	88	90	96	86	92	73	27
Zinc	83	80	76	95	95	94	88	96	80	29
Rare Earths (La,Ce,etc.)	72	74	44	70	70	75	52	67	67	54

[a] Samples were roasted in air prior to leaching except those noted RT, room temperature.

[b] 30-minute roast; all others were roasted for 3 hours.

ICP analyses are believed to be accurate to ±5% for most of the elements listed. Uranium analyses (by the optical fluorescence method) are estimated to be precise to ±10%. Material balances were examined for each duplicate set of experiments — those yielding poor results were discarded and the experiments were repeated.

The Swedish Ranstad process(24–27) which was referenced in the Mountain States report,(28) suggested roasting of the shale followed by crushing before leaching with sulfuric acid. Therefore the effect of "roasting" temperature on recovery of the various constituents in both raw and hydroretorted shale (4–16 mesh) was investigated and these results are also presented in Table 3 (columns 4 and 5-9). These samples were heated in the muffle furnace for three hours before leaching in the manner described above. Heating the raw shale to 500°C before leaching markedly increases solubilization, probably because the "roasting" removes all the organic material and most of the sulfur. However, yields from "roasted" hydroretorted shale are even better since every element listed shows a solubilization greater than 75%. At 1000°C, definite phase and/or structural changes (sintering) must have occurred in the shale, because solubility yields of every element fell drastically — even alkalis such as potassium and sodium were "locked" into the shale. At 800°C some changes had begun, but recoveries (although less than 500°C values) were still satisfactory for nearly all elements.

These leaching experiments indicate that solubilization values considerably better than the 60% recoveries, used by Mountain States in its study,(28) can be achieved, e.g., 98% for molybdenum. Since solvent extraction processes using amine extractants have demonstrated uranium and molybdenum yields greater than 95%, it appears that, at least for these elements, economic worth was underestimated.(28) In Table 4 the value of each important constituent in Chattanooga shale is shown (Mar. 1980 prices listed in *Engineering and Mining Journal*), based on its percentage of recovery given above in Table 3. The dollar values listed are based on metal prices, except for aluminum, where bauxite ore

Table 4. Value of Recoverable Resources

Element	Concentration	Recovered value based on 1980 prices ($/ton of shale)	Percent imported	Percent of U.S. demand supplied[a]
Al	6%	22.00[b]	93	30
Fe	5%	3.60[c]	36	2
Co	60 ppm	3.70	100	20
Cr	100 ppm	0.50	100	0.5
Mn	350 ppm	0.40	95	0.8
Mo	200 ppm	4.80	0	10
Ni	200 ppm	1.20	95	3
Nb	20 ppm	1.20	100	10
U	65 ppm	4.80	0	20
V	300 ppm	3.60	36	100
Oil	0.5 bbl/ton	15.00	50	0.6
Total		$60.80		

[a] Supplied by processing 10^5 tons of shale/day.

[b] Value based on bauxite.

[c] Value based on iron oxide.

prices were used and for iron, where bulk iron oxide prices were used. Also in Table 4, the percent of U.S. demand of each element which is imported is given, along with an estimate of how much of this consumption could be met by processing 100,000 tons of Chattanooga shale per day. Only in the case of vanadium (where the U.S. possesses considerable supplies) would a sizable overage result. Additional studies presented elsewhere(29) indicate that the combination of oil and by-product metal recovery is economically justified on the basis of present market values.

Conclusions and Recommendations

Recent analyses of Devonian shales indicate: (1) that the Chattanooga shale has a higher concentration of uranium than any other U.S. shale; (2) it generally contains more of the strategic metals than other shales; (3) it is among the best of the eastern shales for oil yields, based on the organic carbon content.

At the present time, the "Hytort" process appears capable of producing oil efficiently from Chattanooga shale although there may be alternative methods for lowering costs by decreasing the amount of expensive hydrogen required for retorting. The leaching step for extracting metals from hydroretorted or roasted shale appears to be a simple operation; however, the processes required for separating and obtaining each of the desired elements in an acceptable form for commercial purposes will have to be developed. Proper disposal of the wastes from these operations will require a detailed knowledge of the processes mentioned above.

Because previous judgments about commercial prospects were largely based on uranium as the sole product, they should be reexamined in light of the developments discussed above, to wit:

(1) IGT hydroretorting results which could make oil a major product;

(2) The DOE-funded Bendix-Mountain States study which indicates that recovery of by-product metals is an economic incentive;

(3) Strategic resource studies which show that Chattanooga shale byproducts could supply appreciable U.S. demands for some strategic metals;

(4) Acid leaching studies which indicate that hydroretorting and/or "roasting" significantly enhances the potential recovery of by-product metals.

Based on the above considerations, the prospects appear promising for providing significant quantities of some of the U.S.'s strategic material needs in the long-term, although the processes have to be developed and the economics demonstrated.

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