COMPARATIVE FLOTATION TESTS OF NONMAGNETIC TACONITE USING CATIONIC REAGENTS

By

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A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science in Metallurgical Engineering.

Signed

Lieh-Leng C. Cheng

Approved

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Golden, Colorado

Date *<u><i>Aug* **·**³⁰ *1954***.</u>**

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CONTENTS

INTRODUCTION

In order to meet the ever-increasing demands of the iron and steel industry, a great deal of research is being devoted to the development of methods of concentration for large tonnage of the low-grade iron-bearing formation in the Lake Superior iron ranges. This low-grade iron-bearing formation is called "taconite," which is a term generally restricted to the Mesabi range's Biwabik iron formation **above the Pokegama quartzite and below the Virginia slate (Staff Report, 1947, p. 20-52).**

Taconite is not a simple material of constant uniform composition; to quote a statement by Dr. E. W. Davis of Mines Experiment Station at Minneapolis, Minn.: "Taconite has been defined as a herd, compact, siliceous rock, 25 to 35\$ in iron, that varies widely in chemical composition and in the nature and distribution of its iron content."

Taconite itself is not a marketable iron ore, unless it is up-graded to meet the blast-furnace specifications for iron and impurity content. Because of the variability of the formation materials, each type of taconite has its own physical and chemical characteristics. So, in general, each requires a flowsheet comprising different ore-dressing and agglomeration methods.

The ore under Investigation was a nonmagnetic taconite obtained from the Butler Brothers Mining Company of Minnesota. Chemical and X-ray analyses showed that the ore contains a considerable amount of ferruginous carbonate, or impure siderite. The presence of this mineral can seriously affect the selectivity of the quartz float in amine flotation of iron ore (Roe, 1951, p. 119) (Mortsell and Schwalbe, 1950, sec. 8706).

The Investigation concerned the following topics: (I) A survey of the literature on taconites and different concentration methods, (II) physical and chemical analyses of the ore sample, and (III) comparative cationic flotation tests using several types of amines as collectors and starches as depressants.

OBJECT OF THE INVESTIGATION

The iron content of taconites usually varies between 25 to 35\$ with correspondingly high silica; taconites are too low In iron and too high in silica to meet the blast furnace specifications. Therefore, taconites must be beneficiated to a commercial product which contains over 50\$ Iron and less than 12\$ silica.

It was the primary objective in this work to investigate the concentration of taconites by flotation. The subject selected for investigatIon was the comparative flotation of the silicate minerals by means of amine-type collecting agents with the simultaneous depression of the iron minerals by various types of starches.

GENERAL DISCUSSION OF THE **ACCOMPLISHMENTS IN THE INVESTIGATION**

After extensive studies of the properties of nonmagnetic taconites from the Mesabi range, 10 major producers of iron ore, working in conjunction with Battelle Memorial Institute, classified the nonmagnetic taconites into three main groups on the basis of mineral composition and physical association: Group A — Ores which are principally a mixture of iron oxides and quartz, with good liberation at 150-mesh; Group B — Ores 'which contain substantial quantities of Iron in the form of impure iron carbonate or iron silicates or require a very fine grind for liberation; Group C -- Ores falling between A and B In regard to mineral composition or liberation.

The results of chemical analysis and sink-float tests indicate that the nonmagnetic taconite under Investigation must belong to Group B of the above-mentioned classification because it not only requires a grind through 270 mesh for mineral liberation, but also contains appreciable amounts of impure iron carbonate (5.57% siderite). How seriously will **these factors affect the scheme of concentration? The following quotation is taken from a very recent report**

(Scott and Wesner, 1954, p. 635-641):

"The ferruginous carbonate was more abundant in taconite than minnesotaite 1/. . . • Just how it will respond In the various concentration schemes remains to be established. Evidence at hand indicates that it is extremely toxic to flotation separations, seriously impairing selectivity and increasing reagent consumption. Excessive reagent consumption is peculiar to this impure siderite, since that consumed by high-grade specimen siderite was only one fourth as much."

$1/$ Minnesotaite is a complex iron silicate with a molecular **structure similar to talc, except that the major part of** the magnesium is replaced by iron.

Flotation results showed that none of the tests produced a concentrate containing more than 50% iron and less than 10% insoluble. In flotation Test 9, a concentrate of 50.1% iron and 21.4% insoluble was obtained. A duplicate run gave only 49.8% iron and 22.0% insoluble, with a correspondingly low iron recovery of about 64%. Generally, over 0.25 pound per ton of the most favorable amine collectors (Dodecylamine Acetate or CoCo Amine Acetate) is required to produce a concentrate of over 40% iron and less than 37.5% insoluble. For the starch additions, at least 0.25 pound per ton of the most favorable starch (No. 152 Canary Dextrine) is required to bring the iron recovery 2/ to above 70% and at the same

2/ **Iron recovery is based on the assay of the prepared ore for the flotation feed (30.6% Fe and 51.7% Insol). The total iron recovery or over-all iron recovery is even less. ___**

time maintain the iron content above 40% in the concentrate. The consumption of the flotation reagents was not excessive, because the flotation feed had been acid-scrubbed and all the **particles finer than 20 microns had been removed by elutriation. The flotation feed, after acid-scrubbing and removal** of minus 20-micron particles, still contained 2.24% impure **siderite. Nevertheless, the grade of the concentrate and the iron recovery were far from those desirable.**

Based on the present experimental results, the cationic flotation method of concentration is not entirely successful as a treatment-method for the type of nonmagnetic taconite under investigation. It is doubtful that this taconite will be amenable to other methods of concentration.

GEOLOGY OF THE IRON FORMATION IN THE LAKE SUPERIOR REGION AND ITS RESERVES

The iron ranges in the Lake Superior Region include the Mesabi, Vermilion, and Cuyuna ranges In Minnesota; Marquette and Menominee ranges in Michigan; and the Gogebic range, which lies partly in Michigan and partly in Wisconsin

The deposits are all pre-Cambrian sediments. The Vermilion is attributed to the Archean, and the others to Middle or Upper Huronian. Originally the Iron was present In the form of hematite, greenalite, and siderite, which made up 20 to 30\$ of the whole iron-bearing formation. This iron-bearing formation Is part of a thick series of sediment ary rocks which have been metamorphosed into quartzites, slate, cherts, marbles, and jaspers. The metamorphic series

Fig. 1 - Idealized Cross-Section of Mesabi Range Near Babbitt.

ranges up to about 1000 feet in thickness. A typical crosssection of a portion of the Mesabi range is shown in Fig. 1.

As shown in Fig. 1, the taconites include all of the Mesabi iron formation interbedded between the Pokegama quartzite and Virginia slate, excluding the commercial ore bodies. The estimated reserves are very large. A staff report by prominent men in the iron ranges states (Staff Report, 1947, p. 26):

"The known depth of the formation is about 700 feet on **the average, and varies from one to three miles in width,** and has a strike length of slightly more than 100 miles. **Based on the above dimensions of the ore body, the calculated tonnage is about 380 billion long tons, or about 100 billion** lon_{μ} tons equivalent concentrates. At the present rate of **extraction, the reserves are more than adequate for generations**

to come.

The main ore bodies of magnetic taconites lie east of Hibbing between the Aurora and Babbit areas in a strip about 20 miles long. Glacial drift scantily covers most of the deposit, but great areas of the formation are still exposed. Approximately 1.7 billion tons of concentrate can be produced from these magnetic taconite deposits (Hubbel, 1949, p. 84). **At present, only the magnetic taconites are being treated, because of the development of the wet magnetic separation process•**

However, nonmagnetic taconites are the most common and make up the greater portion of the reserves. The problems

of concentration on this type of taconite offer the greatest challenge to ferrous metallurgy.

DIFFERENT COMMERCIAL METHODS OF CONCENTRATION OF IRON-FORMATION MATERIALS

In the past decade, research on the concentration of taconites has been actively pursued both by leading steel and iron companies in the iron districts and by private institutions. The different commercial methods of concen**tration may be summarized as follows:**

Magnetic Separation - Magnetic separation is a relatively inexpensive high-capacity operation* It is applied to the taconites in which the major iron-bearing mineral Is magnetite. Ores in which the iron content is equally distributed between hematite and magnetite are not economically concentrated by this process. An alternate proposal is to **convert hematite to magnetite by a reduction roast, followed** by magnetic treatment. And again, the high cost of furnish**ing the heat for the roasting, as well as high Initial cost of equipment, has led to the abandonment of the process as uneconomical•**

The magnetic treatment of the magnetic taconites consists essentially of crushing the ore to about 1/4 Inch, rejection of a tailing magnetically, fine grinding of the partial concentrate, and magnetic concentration of the ground material.

Grinding to about 150 mesh is necessary for final concentration* The ratio of concentration is about 3 tons to 1. In most cases, some 40 to 50% of the tonnage can be rejected as satisfactory tailing before and during the grinding operation, and the balance of the tailing is rejected after grinding has been completed. A concentrate of around 60% iron is readily obtainable.

Gravity Concentration - Gravity concentration affords a relatively inexpensive method of treatment for the somewhat coarse-grained and friable banded taconites. But the process Is not applicable to the finely disseminated type of taconites. Furthermore, the recovery of iron units from most gravity concentration processes Is very low. In some taconite benefIciation mills, gravity methods are adapted as a preliminary treatment or a step in the flowsheet. In so doing, it is claimed that a good amount of coarse high-grade concentrate could be obtained.

Flotation - Flotation offers an unusually attractive field of investigation, because it Is particularly applicable to finely ground ore particles. Generally, for nonmagnetic taconites, grinding to about 150 mesh is required for commercial liberation of the hematite from the gangue minerals. But the adverse facts are that taconite particles finer than 325 mesh absorb an excessive amount of flotation reagents, and also affect the selectivity of the flotation reagents used. Another factor which must be considered is the presence of

Impure siderite or ferruginous carbonate, a common and undesirable iron-bearing component of taconite. L. A. Roe of the Bjorksten Research Laboratory, Inc., Madison, Wis., **states (Roe, 1951, p. 119):**

"In the amine flotation of silica from iron ores, it has been noted that certain minerals poison the float. The presence of as little as of siliceous siderite, a mineral component of some Mesabi Range ores, can seriously affect the selectivity of the quartz float."

At the present time, flotation as a method of concentrating the low-grade taconites is still in the experimental stage•

PROPERTIES OF THE ORE TESTED

PHYSICAL ANALYSES:

Microscopic ExaminatIon of Sample Specimens - A few typical samples of the nonmagnetic taconite were selected and mounted in bakelite. The surface of the specimens was well polished and cleaned with alcohol. A high-power polarizing microscope in the Research Foundation Laboratory was **used for examining the specimens. The principal iron-bearing minerals identified, excluding siliceous gangue, listed in the order of their quantities, are tabulated below:**

> **1. Hematite (FegOg).** 2. Magnetite (Fe_3O_4) . 3. Limonite or Goethite (Fe₂O₃. x H₂O). **4• Pyrite (FeSg).** 5. Siderite (FeCO₃).

Microscopic examination of several specimens reveals that the taconites vary greatly in their physical structures. Generally, the magnetite was present in larger crystals than the needle-like or platy hematite. Magnetite has the property of yielding somewhat to pressure; a needle drawn across a smooth surface will often leave a distinct mark, but this is not a true scratch, as the sides of the mark are unbroken. Both magnetite and hematite have steel-gray appearance.

Limonite or goethite, which are very hard to distinguish from one another, appear as an orange-colored powder; they are rather resistant to chemical attack. The crystals with curved surfaces,, and somewhat dissolved by repeated application of concentrated HC1 droplets, were identified as siderite.

During ore preparation for flotation feed, a few grams of plus 100-mesh grains were examined with the microscope. Quite a few crystals of rutile (TiO₂) and apatite were identified. They are characterized by their crystallographic **systems and colors; tetragonal with reddish Internal reflection of grey sphalerite-like crystals for the former, and hexagonal with violet color for the latter. A small amount of mica was also observed.**

X-Ray Diffraction Analysis - To confirm and complete the identifications of minerals present in the taconite, X-ray diffraction analysis was employed. A Norelco Geiger counter spectrometer was used to record the "d" values of **compounds present in the sample.**

The X-ray diffraction results are tabulated in Table I. The following operating conditions were used for the X-ray spectrometer.

SAMPLE-TACCILTE $\frac{1}{2}$ $\frac{1}{\sqrt{2}}$ DATA DIFIFRACTION $\underline{X-RAY}$ THE $\pmb{\mathfrak{t}}$ \mathbf{H} ТАВЫЕ

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 (Cont) nuation of the X-Ray Diffraction Data.)

The "d" values are characterized by interplanar distances of each individual
mineral: they are used to identify the minerals from the data cards on the
basis of their relative intensities, by comparing with the experiment $\pmb{\mathfrak{t}}$ Note 1.

CHEMICAL ANALYSIS:

About 200 pounds of the nonmagnetic taconite sample was crushed to minus 6 **-mesh size, mixed well in the cement mixer, and split with a Jones riffle until several 5-kilogram grams** portions were obtained.

The samples for chemical analysis were taken from one of the 3-kilogram portions of ore, ground to minus 100 mesh, and thoroughly mixed* The head sample was submitted to Mr. A. C. Pierce for assay. Since siderite is suspected of being detrimental in the flotation process, a portion of the head sample was sent to Charles 0. Parker and Company, an assay firm in Denver, for the determination of siderite (FeCO^) by Cook's Method, in which the ore is treated with HCl in **C0g gas atmosphere, cooled, and titrated with permanganate.**

The results of the chemical analysis are tabulated below:

Total Fe Insol Siderite Lime Alumina Magnesia 28.40\$ 54.78\$ 5.57\$ 0.80\$ 0.68\$ trace (All percentage is expressed by weight).

SINK-FL0AT TESTS:

Heavy liquid separation Is based on the fact that minerals by virtue of their specific gravity differences can be separated by floating or sinking in liquids of a density intermediated between the densities of the minerals to be separated. It is an accurate method for liberation studies in the laboratory.

Two heavy liquids with specific gravities of 2.703 and 2.800 were used in tests to separate tails and middlings respectively. These liquids consisted of mixtures of carbon tetrabromide (CBr^) of 2.952 In specific gravity and carbon tetrachloride (CCI4 **) of 1.632 in specific gravity.**

To separate the heavier Iron oxide minerals from the lighter minerals, which are essentially silica, a 1 **-kilogram sample of ore was ground and passed through a set of Tyler screens ranging from 65 mesh to 325 mesh. Eight 20-gram portions of the material retained on each screen were weighed. The sink-float tests were then performed on these 20-gram samples. The results are tabulated in Table II.**

In the flotation tests, the degree of liberation of the valuable minerals from the gangue in the grinding circuit plays an important role In determining the over-all economic recovery. For complete liberation, the ore should be ground much finer; however, the valuable minerals lost in fine particles during removal of the fines must be taken Into consideration.

In the case of the amine flotation of siliceous gangue from Iron ore, the fine particles have to be thoroughly removed to attain the maximum effects with the minimum amount of flotation reagents. It is generally agreed that particles finer than 20 microns (about 750 mesh) should be removed from the pulp. As shown in Fig. 2, the percent iron loss in the form of fine particles (minus 20 **microns) is**

		Tyler	-65	-100	-115	-150	-170	-200	-250	-270
		Std.Screen	to	to	to	to	to	to	to	to
		Scale	$+100$	$+115$	$+150$	$+170$	$+200$	$+250$	$+270$	+325
	\mathbf{z} HEAD Assa	$%$ Fe	32.0	30.2	31.2	31.7	<u>30.6</u>	30.2	30.0	28.2
		$%$ Insol- uble	49.40	52.24	50.24	49.84	51.30	52.28	52.06	54.86
SINKS		$Wgt.$ in GmS	9.42	9.57	9.18	9.83	9.70	9.55	9.33	9.10
		Individual $%$ Weight/	47.1	47.9	45.9	49.2	48.5	47.8	46.2	45.5
		% Fe	50.0	<u>51.6</u>	54.6	$\overline{55.2}$	55.8	56.4	57.5	<u> 30.2 </u>
		σ % Insol- uble	21.92	20.44	16.40	16.40	15.16	14.12	10.28	10.00
	ဒါ	$%$ Fe Dis- tribution	73.9	81.8	83.0	88.1	89.4	91.6	94.1	95.3
ပ္ပါ DDLIN 그는	2.80 O	Wet. in Gms	3.08	2.65	0.92	0.77	0.65	0.51	0.45	0.40
	Ю ρZ	"Individual % Weight	15.4	13.2	4.6	3.8	3.3	2.5	2.3	2.0
		5 Fe	39.6	28.8	17.6	19.8	13.4	13.8	12.4	10.7
		$%$ Insol- uble	37.80	54.50	55.24	50.16	60.92	78.00	80.00	82.70
		$%$ Fe Dis- Stribution	19.2	12.5	2.68	2.45	2.00	1.30	1.01	0.70
$\frac{28}{10}$ QTA	$\boldsymbol{\omega}$ \circ 2	Wgt. in Gms	7.50	7.78	9.90	9.40	9.65	9.94	9.21	10.50
		Indivudual % Weight	37.5	38.9	49.5	47.0	48.3	49.7	46.0	52.5
		$%$ Fe	5.4	4.4	8.8	6.2	5.4	4.2	3.0	$\overline{2 \cdot 2}$
	ت	$%$ Insol- uble	89.04	90.92	83.20	87.04	88.12	90.84	95.00	97.2
	ကိ	$%$ Fe Dis- tribution	6.45		5.50 14.32	5.45	8,50	7.08	4.83	4.02

TABLE II - TABULATION OF SINK-FLOAT TESTS.

 \checkmark Individual $\frac{\%}{\%}$ Ngt . is based on the indivudual 20-gram portion of each screen material, for example:

Individual $\frac{8}{9}$ Wgt. of the Sink = $\frac{9.42}{20.0}$ = 47.1 %.
(minus 65 to plus 100) = $\frac{9.42}{20.0}$ = 47.1 %.

The vertical tabulations are laboratory, and results of indivudual 20-gram portion of sample from each NOTE: screen.

Increased almost proportionally with the degree of grinding. Thus, the iron loss in the fine particles is another factor which governs the economic recovery.

The results of sink-float tests showed that if the ore is ground to minus 270 mesh, it is possible to up-grade the iron content to at least 60.2% and an insoluble content of less than 10\$. But before the pulp of the taconite is put into the flotation circuit, there should be a thorough removal of the fine particles (minus 20 microns). Thus in Fig. **2, if the ore is ground to minus 270 mesh, the percent iron loss in the fine particles will be about 43\$ of the original ore. In other words, only about 57\$ of the original iron content is left in the minus 270-mesh material. From the sink float tests again, it was shown that not all of the 57\$ iron content of this ore is completely liberated; the liberation is only 95.3\$ complete (\$ Fe Distribution in the Sink) 3/.**

37 Although 95.3\$ liberation asindicated by the percent Fe distribution in the sink (Table II) is based on the figures of minus 270 to plus 325-mesh ore, this figure is also approximately right for all minus 270-mesh ore in which the minus 325-to plus 750-mesh materials are included. (All minus 750-mesh or minus 20-micron particles were removed by elutriation during ore preparation for the flotation tests) . To avoid producing an excessive amount of fine particles, stage-grinding in the Denver Laboratory rod mill was employed. Thus, if the ore is all minus 270-mesh to plus 20-micron material, only a small portion of particles between minus 325 to plus 750 mesh are present. The same is true for all other coarser grinds

Therefore, 0.953 of 57\$ is 54.4\$, is the actual amount of iron liberated and available for concentration. It Is also **equivalent to the highest probable recovery, if this minus 270-mesh to plus 20-micron ore is concentrated by flotation and a good grade of concentrate is to be obtained. It is obvious that 54.4\$ recovery In iron ore is not economical b e c a u s e 'of low unit cost of iron and high mining cost of taconites•**

To determine the probable recoveries of all-size grinds, the following complete calculations are shown:

> **For Minus 270-mesh Grind: (43\$ Iron-Loss) 57\$ x 0.953 r 54.3\$ Probable Recovery For Minus 250-mesh Grind: (40\$ Iron-Loss) 60\$ x 0.941 = 56.5\$ Probable Recovery For Minus 200-mesh Grind: (30\$ Iron-Loss) 70\$ x 0.916 = 64.1\$ Probable Recovery For Minus 170-mesh Grind: (25\$ Iron-Loss) 75\$ x 0.894 = 67.0\$ Probable Recovery For Minus 150-mesh, Grind: (22\$ Iron-Loss) 78\$ x 0.881 = 68.7\$ Probable Recovery For Minus 115-mesh Grind: (14\$ Iron-Loss) 86\$ x 0.830 = 71.4\$ Probable Recovery For Minus 100-mesh Grind: (13\$ Iron-Loss) 87\$ x 0.818 * 71.2\$ Probable Recovery For Minus 65-mesh Grind: (10\$ Iron-Loss) 90\$ x 0.739 = 66.5\$ Probable Recovery**

Based on the above calculated figures, it was decided that the sample ore should be ground to minus 115 mesh, in order to prevent excessive production of minus 20-micron particles with the resultant iron loss.

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COMPARATIVE FLOTATION TESTS

Comparative flotation tests with various cationic reagents of the amine-salt types and various starches are the main purpose of this investigation. The process of the experimental work is divided Into two parts. Part I deals with the investigation of six different types of amines as collectors for the siliceous gangue minerals, using a constant quantity of Globe Pearl starch as iron mineral depressant. Part II deals with the Investigation of five different types of starches as depressants, with a predetermined constant quantity of the amine that proved most favorable as a selective collecting agent of silicate gangue In Part I.

Before the details of the experimental work are described, some established facts concerning cationic flotation should be mentioned. The cationic collectors are ionizable organic compounds in which the ion that carries the hydrocarbon and reactive groups is the cation. The. cation reacts with the anion present at the mineral surface, and forms a film in which the organic part is oriented outward from the surface. In amine flotation of quartz, which is a typical cationic flotation, the chemical reaction

involved consists of en exchange between the amine-bearing (aminium) ion and the cation of the mineral, and formation at the mineral surface of an oriented film of amine salt of the mineral anion. The preliminary formation of a silicic acid film at the mineral surfaces, followed by reaction with the amine hydroxide according to the following equation:

 $RNH_8 \cdot OH + H_2SLO_8 \longrightarrow (RNH_8)$ SiO₈ + H₂O. **The reactions were evidenced qualitatively on several occasions at Columbia University (Taggart, 1945, p. 12-14).**

With regard to the starch as depressant, Professor Cooke and his co-workers have shown that starches selectively coat hematite In preference to quartz (Cooke, Schulz, and Llndroos, 1952, p.697). The mechanism by which the starch inhibits a collector coating Is not clearly known. However, because of the presence of large numbers of (OH)" groups in the starch molecule, mineral surface coated with starch should be hydrophilic.

ORE PREPARATION:

A 3-kllogram portion of sample ore (minus 6 mesh) was mixed and split Into three equal portions of about 1 kilogram each. The 1-kilogram portion was accurately weighed to the nearest tenth of a gram. To avoid overgrinding, the minus 100-mesh material was removed by dry screening, and the plus 100-mesh material was ground in a Denver laboratory rod mill

for 5 minutes with about 50\$ water. After 5 minutes of grinding, the minus 100-mesh material was removed by wet screening, and the plus 100-mesh material was returned to the rod mill for regrinding. This was repeated several times, until the ore all passed through the 100-mesh screen. All the minus 100-mesh material, including the material from the dry screening, was placed into a 10-liter glass jar. The pulp was diluted so that the pulp filled a 10-liter glass jar to within 3 inches of the top. Twenty-five milliliters of 10\$ sodium silicate, equivalent to about 5 pounds per ton was added to the pulp, and the pulp was stirred until all the solid material was suspended. The pulp was elutriated to remove the minus 20-micron particles by the beaker decantation method (Dorr, 1950, p. 10). The pulp left in the glass jar was diluted to around 50\$ solids, transferred to a laboratory Fagergren cell, and scrubbed for 5 minutes with 10 milliliters of 10\$ sulphuric acid, equivalent to 3.6 pounds per ton of ore. The pH of the pulp at the start of the acid scrubbing was 2,65: this increased to 4.50 after 5-mlnute period. After acid scrubbing, the pulp was elutriated once more to remove any freshly produced minus 20-micron particles. The resulting pulp was rinsed with distilled water to remove the residual acid, filtered, dried, and weighed; and samples were prepared for assay. The prepared ore was used for the subsequent flotation tests.

For the preparation of the minus 150-mesh and minus **200-mesh elutriated ore, the same laboratory procedure was used. The following results were obtained:**

(1) Ore ground to -100 mesh:

Original wgt. of ore----------------1000.0 grams (28.4\$ Fe)

Final wgt. of ore after acid scrubbing and removal of all minus 20-micron fine particles 803.5 grams (30.6\$ Fe)

Iron loss in minus 20- micron particles-----------------------------13.4\$ by Wgt

(2) Ore ground to -150 mesh:

Original wgt. of ore------------- ---1000.0 grams (28.4\$) Final wgt. of ore after acid scrubbing and removal of all minus 20-micron of air minus zo*micron
fine particles---------------------762.6 grams **(29.6\$ Fe)**

Iron loss in minus 20-micron particles 20.6\$ by Wgt

(3) Ore ground to -200 mesh:

Original wgt. of ore------------- ---1000.0 grams (28.4\$ Fe)

Final wgt. of ore after acid scrubbing and removal of all minus 20-micron fine particles---■ 681.0 grams (29.1\$ Fe)

Iron loss in minus 20- micron particles ------------------------ 31.9% by Wgt.

Sink-float tests on the closely sized fraction of the plus 20-micron material indicated that the best probable recovery could be obtained at a grind to either minus 115 **mesh or minus 100 mesh.**

Therefore for the subsequent flotation tests the ore was stage-ground in a Denver laboratory rod mill until the entire sample passed the 100-mesh screen.

REAGENTS AND THEIR PREPARATION:

Collectors - All the amine collectors used for flotation tests, except Amine 0, were supplied by Armour Chemical Division, Armour and Company, Chicago, 111. The Amine 0 was obtained from Alrose Chemical Company, Providence 1, R. I. All the amines were soluble in hot water. However, the Soy Amine Acetate would not completely dissolve in hot water without the aid of the Waring Blender high-speed agitator. A freshly prepared 0.1\$ solution was used in each series of tests.

Depressants - Five different starches and starch-like products were tested as the depressants for iron minerals. Among them, Globe Pearl Starch No. 144 and No. 152 Canary Dextrine were supplied by Corn Products Refining Company, Argo, 111.; Guartec and Gum-39 were supplied by the Research Laboratory of General Mills, Inc., Minneapolis, Minn.; and rice starch was prepared from California rice.

Globe Pearl Starch No. 144 is a direct product of corn kernel. It is 20\$ soluble in water. The water-insoluble

constituents do not affect the flotation results. (Chang, Cooke, and Huch, 1953, p.1285).

Guartec is a refined endosperm of the guar seed. It is cold-water swelling and will develop its properties without the application of heat. Guar, which had been known botanically as Cyamopsls tetragonoloba, is a drouthresistant legume from India, and is being groomed as a new cash crop for farmers in the southwestern United States. (Esser, 1947, p. 229).

Gum-39 Is a proprietary compound of General Mills, Inc.; it Is a blend of several substances and Is still in the process of undergoing patent application.

No. 152 Canary Dextrine is a decomposition product obtained by dry heating of corn starch. It is 98.9\$ soluble

The rice starch was prepared os follows: The rice grains were ground to a very fine powder by mortar and pestle; then about 20 grams of this fine rice powder were placed into 200 milliliters of distilled water in a beaker, and digested on the hot plate (50 deg. C) until all the water evaporated. The residue was ground to a very fine powder again, and used to prepare 0.1\$ starch solution.

Starch reagents to be added to the flotation cell were all prepared as 0.1\$ solution. Most of the starches are not as readily soluble as amine collectors; they were prepared as below:

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A 0.200-gram portion of starch was weighed into a 400-

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milliliter beaker. A small amount of cold water was added to the beaker to wet the starch, then about 200 milliliters of boiling water was added, and the solution was thoroughly stirred. This hot solution was transferred to the Waring Blender, a high-speed mechanical agitator, and agitated for 5 minutes. It was found that even the Pearl starch, which is only about 20\$ soluble in water, was pretty well dispersed into a colloidal solution by this process. After 5 minutes of vigorous agitation, the starch solution was returned to the beaker, and digested in the hot bath (around 50 deg. C) for about 12 hours. Finally the starch solution was diluted to exactly 200 milliliters before being used as flotation additive.

FLOTATION CELL SET-UP:

The flotation cell, fundamentally a pneumatic type, was similar to that described by Professor Cooke (Chang, Cooke, and Huch, 1953, p. 1283). In addition, a mercury **manometer was installed to regulate the compressed air, and a self-made cotton air filter was used. The cell consisted of a 350-milliliter-capacity Buchner fritted glass funnel with medium porosity frit. Air was admitted through the bottom of the cell and controlled by a diaphragm regulator. Admission of air alone did not give sufficient agita**tion to keep all the material in suspension; hence supple**mental mild agitation, provided by a motor-driven stirrer,**

was used in the cell* To facilitate the collection of the froth, a rubber sleeve was fitted in to the neck of the cell. The complete assemblage is shown in Fig. 3.

Fig. 3 - The flotation cell with its auxiliary equipment. (1) Buchner fritted glass funnel, (2) stirrer with stainless steel rod, (3) rubber sleeve, (4) air inlet, (5) Buchner funnel, (6) solution reservoir, (7) air filter, (8) Vacuum line, (9) air regulator, and (10) Hg manometer.

FLOTATION TEST PROCEDURE:

Exactly 50 grams of prepared ore was added to the cell, together with about 180 milliliters of distilled water and starch or starch product as called for in the test. The mixture was conditioned by agitation for 3 minutes, after which the amine collector was added. This was followed by an additional 1-minute conditioning. Then the air was admitted to the cell, and the froth was removed until it was almost

barren of mineral. The froth flowed from the cell into a Buchner funnel, where the froth was filtered and the filtrate transferred to the solution reservoir by suction. This solution was then available for re-introduction into the cell for the first cleaning, thus eliminating the necessity of adding **water and more reagents during the test. (Clemmer and Clemmons, 1943, p. 72). For the recleaning, the filtrate was reused once more. The removal of the froth required 1 to 5 minutes, depending on the froth characteristics. Natural pH was used throughout the test. The froths contained most of the quartz, and most of the iron minerals remained in the cell. These two products were analyzed for iron and Insoluble contents.**

Before being used in any subsequent tests, the flotation cell was thoroughly washed with hot soapy water and rinsed with distilled water.

The flotation data and metallurgical balances are tabulated in Table III.

DISCUSSION:

Professor Cooke 4/ and his research co-workers indicate

Cooke, S.R.B., Member AIME, is Professor of Metallurgy and Mineral Dressing, University of Minnesota.

that Pearl starch is an effective depressant of iron ore (Chang, Cooke, and Huch, 1953, p. 1286). A few preliminary tests showed that about 0.10 pound per ton of Pearl starch No. 144 should give a fairly distinguishable separation for the nonmagnetic taconite tested. Although there was no

BALANCE TABLE III - FLOTATION DATA AND MATALLURGICAL

$\underline{A} \quad \underline{R} \quad \underline{T}$ \overline{a}

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Part I consisted of comparative tests of different amine collectors, with constant
quantity of 0.10 $#/T$ of Globe Pearl Starch (No. 144) as depressant.

*Total Fe Recovery is based on original head assay (28.4% Fe) and takes into con-
sideration the iron lost in fine particles which are removed by elutriation.
Throughout the Part I, one drop of frother (cresylic acid) was Note:

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<u>Part</u> II consisted of comparative tests of different starches as depressants, with
constant quantity of 0.35 $\#/T$ of Dodecylamine Acetate (ARMAC 12D) as collector.

Throughout the Part II, two drops of frother (cresylic acid) was added to the cell
after conditioning with starch in each flotation test. Note:

Test 11: (Continued) **Test 1 1 : (Continued)**

TABLE IV - Grain Counts on Flotation Products with
 $\#152$ Canary Dextrine, 0.15 $\#/T$;
Dodecylamine Acetate, 0.35 $\#/T$:

 $**TABLE** **IV** - (Continued)$ </u>

* I.M. refers to iron minerals; they are either magnetite, hematite, goethite, or
other iron oxides.
** Gangue materials are mostly quartz or chert.

indication that the specified grade (61% Iron and 8.5% Insoluble) could be obtained with this starch, this quantity of Pearl starch was used to start with on comparative testing with different amine collectors.

To evaluate the selectivity and the effectiveness of both amines and starches tested as iron mineral collectors and depressants respectively, it is necessary to set a specification on both the grade of the Iron concentrate produced and on the iron recovery. Figs. 4 and 6 show such results graphically In condensed form.

In Fig. 4, the iron recovery and percent of Iron content in the concentrates were plotted against the amine collector addition. If the effectiveness of amines as selective collectors is based on equal quantitative additions to bring about the best obtainable concentration, then Dodecylamine Acetate (also see Fig. 5) is the most effective collector tested, followed respectively by CoCo Amine Acetate, Laurylamine Hydrochloride, Soy Amine Acetate, Tallow Amine Acetate, and Amine 0. On the same basis, Dodecylamine Acetate also gave the best recovery among the collectors tested.

In Fig. 6, the iron recovery and the percent iron In the concentrates are plotted against the iron depressants added. On a quantitative basis, Gum-39 is the best depressant for both iron minerals and silica, because It gives the best recovery among the depressants tested. However, the most effective depressant that selectively depressed the iron

minerals only is No* 152 Canary Dextrine (also see Fig. 7), then followed by Globe Pearl starch No. 144, Gum-39, Guartec, and rice starch.

During the flotation tests, intense flocculation of the fine particles occurred after the addition of the collector. It is possible that most of the iron mineral particles carried into the froth were those trapped during flocculation. Microscopic examination of the concentrates showed that most of the quartz particles appeared in the coarsest screen fraction, which apparently failed to respond to the collector. From Table IV, grain counts on the froth product showed that the number of Iron mineral particles rises very sharply as the particles size decreases, indicating that a large part of iron consists of free oxide particles which presumably **are carried mechanically into the froth.**

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SUMMARY AND CONCLUSIONS

1. X-ray and chemical analyses showed that the nonmagnetic taconite under investigation is quite a complex ore. The most serious complication is that it contains more than 5*%* **ferruginous carbonate, or impure siderite, which renders the ore unamenable to the flotation method of concentration.**

2. Sink-float tests indicate that the ore should be ground to minus 115 mesh in order to attain a maximum probable recovery of 71.4# iron content. It also Indicates that satisfactory liberation of iron minerals from the gangue will not be obtained until the ore is ground to minus 270 mesh, but the iron loss in fine particles (minus 20 microns) during the elutriatlon process will be excessive.

3. From the sink-float tests and chemical analysis, the ore should be grouped in that particular class of nonmagnetic taconites which is the most difficult bo treat at present.

4. Part I of the flotation tests showed that, in the order of decreasing metallurgical effectiveness as selective collectors of silica from iron minerals, the amine-salt reagents are : Dodecylamine Acetate, CoCo Amine Acetate, Laurylamine Hydrochloride, Soy Amine Acetate, Tallow Amine

Acetate, and Amine 0.

5. Part II of the flotation tests showed that, in the order **of decreasing metallurgical-effectiveness as selective depressants of iron minerals, the starches or starch products are: No. 152 Canary Dextrine, Globe Pearl starch No. 144, Gum-39, Guartec, and rice starch.**

6. No entirely satisfactory flotation result was obtained. However, the results indicate that the removal of minus 20 micron particles and acid scrubbing of the flotation feed reduces the reagent consumption considerably. The results also indicate that the prepared ore responds readily to the collecting effects of the amine-type collectors and the selective depressing effects of starches.

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