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## SELECTIVE FLOCCULATION OF ZINC CONCENTRATE TO REDUCE SILICA CONTAMINATION

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**Abstract.** Selective flocculation was used to produce superclean zinc concentrate with minimized silica contamination. Research was carried out on a flotation concentrate from the Jersey Miniere Zinc Company (JMZ) in Gordonsville, Tennessee. The material was first dispersed with sodium silicate and then causticized tapioca starch was used to selectively flocculate the sphalerite, leaving the silica in suspension. This technique has been applied commercially to iron ore beneficiation but has never been used for processing zinc. The feed material assayed 0.45% SiO<sub>2</sub>. Use of 0.56 kg/tonne (1.13 lb/st) of sodium silicate and 0.070 kg/tonne (0.141 lb/st) of causticized tapioca starch resulted in 98.2% zinc recovery in a purified zinc concentrate assaying 66.0% Zn and 0.24% silica. The present target level for silica contamination in JMZ zinc concentrates is 0.3% SiO<sub>2</sub>. This work has shown that selective flocculation offers a potential method for cleaning flotation concentrates, particularly where low silica products are required.

#### Introduction

The Jersey Miniere Zinc company mines and mills zinc ore at Gordonsville, Tennessee. The current milling rate is 5443 tonnes/d (6000 stpd); concentrator capacity is 9072 tonnes/d (10 000 stpd). Ore deposit geology, mining, and milling techniques have been described in the literature (Callahan 1967, 1974, 1977; Derry 1973; Fischer and Hoagland 1980; Gratz and Misra 1987; Kyle 1976; Painter 1980a, 1980b; Schultze 1983; White 1979; Winslow and Hill 1973). The ore, grading about 3.36% Zn, occurs in carbonate rocks and contains sphalerite, calcite, and dolomite, with lesser amounts of galena, barite, fluorite, and pyrite. Small amounts of deleterious impurities (SiO<sub>2</sub>, CaO, MgO) are found in the zinc concentrate. Among the most troublesome of these is silica, since it interferes with byproduct recovery.

Germanium is a valuable byproduct which is recovered from the zinc concentrate in the zinc electro-refining process. The zinc concentrate typically assays about 0.049% Ge. During concentrate roasting operations at the zinc refinery, silica combines with some of the germanium in the concentrate to form a compound containing germanium and silica in solid solution. This compound is refractory to leaching, thus rendering the germanium unrecoverable.

The present work was undertaken to reduce the silica level in the zinc concentrate below the target level specified by the company, i.e. 0.3% SiO<sub>2</sub>, and thus mitigate the problems caused by silica in later concentrate processing. This goal was accomplished by dispersing the feed material with sodium silicate, a well known silicate mineral dispersant, and then selectively flocculating the sphalerite with causticized tapioca starch, leaving the impurities in suspension, followed by selective desliming.

This technique was developed at the U.S. Bureau of Mines (Frommer 1964, Colombo and Rule 1969, Colombo and Frommer 1976) and was applied commercially to iron ore beneficiation to separate silicate minerals from hematite at the Tilden mine in Michigan (Villar and Dawe 1975). The experimental variables studied here were sodium silicate and starch addition levels; pH was not studied because of the limited amount of sample, although other researchers have shown that pH has a great effect on the adsorption of starch on quartz and hematite (Schulz and Cooke 1953; Iwasaki and Lai 1965).

#### Experimental Equipment and Materials

1) Two samples of zinc concentrate from the Jersey Miniere Zinc Company concentrator, Gordonsville, Tenn.

The first sample was taken during experimental work carried out by Salt Lake City Research Center personnel in the JMZ mill during 1989; it was a cleaner concentrate from a mobile column flotation machine taken July 23, 1989. The sample was filtered in our laboratory before use in the flocculation tests; moisture content was 11%.

The second sample was taken by JMZ personnel. It was a cleaner concentrate sample and was received as a slurry at the Salt Lake City Research Center on 23 May 1990. Before use in the present testwork, it was filtered to a moisture content of 11% and then blended moist. On a dry basis, the material assayed 0.45% SiO<sub>2</sub>, 0.38% CaO, 0.17% MgO, and 64.9% Zn. This second sample was used to generate all data reported herein. Size and chemical analysis of the concentrate (Table 1) showed that the minus 38- $\mu$ m (minus 400-mesh) fraction contains the bulk of the silica (43.3%) and 29.7% of the zinc. Most of the possible physical beneficiation methods which could be used to remove the silica contaminant are precluded because of the extreme fineness of the material.

2) Denver laboratory flotation machine model D-1<sup>1</sup> with a 3.4 L capacity plastic cell.

<sup>1</sup>Reference to specific trade names does not imply indorsement by the U.S. Bureau of Mines.

3) Brand N sodium silicate (SiO<sub>2</sub>/Na<sub>2</sub>O = 3.22/1) from the Philadelphia Quartz Co.

4) Tapioca starch flour from the Chicago Dietetic Supply House, Inc. The starch was causticized before use. The procedure for preparing a 2% solution of causticized starch in deionized water was as follows:

- a) In 50 cc of cold water, mix 4.0 g tapioca starch.
- b) In 50 cc of cold water, dissolve 1.0 g NaOH.
- c) Mix both samples into a 200 cc volumetric flask.
- d) Dilute to full 200 cc volume.
- e) Heat slowly until the solution clears, but do not boil. Keep at approximately 82°C (180°F) for 30 min while

continuously stirring.

f) Cool to ambient room temperature before use. Add deionized water to full 200 cc volume.

The tapioca starch solution was used within 36 hr after mixing in order to minimize the effects of microbiological decomposition.

#### Procedure

First, 400 g of moist zinc concentrate (11% water; average dry charge wt. 356 g) was slurred with 1.5 L of deionized water in the flotation cell with the impeller at 1025 rpm. The natural pH of this slurry was 6.9 to 8.3. Next, a measured amount of sodium silicate was added as a 10% solution in deionized water and conditioned with the solids for 10 minutes; the pH at this stage ranged from 8.1 to 9.3, depending on the amount of silicate added (pH changes during the tests as a result of reagent additions were monitored, but not controlled). The slurry was diluted by adding another 1.0 L of deionized water. A measured amount of causticized tapioca starch was added as a 2% solution and mixed with the solids for 1 minute; the pH at this point ranged from 8.6 to 9.2.

Agitation was stopped and the solids allowed to settle for 1 minute. This resulted in a visibly distinct layer of settled solids at the bottom of the cell with a dilute, dispersed slurry above.

The supernatant liquid with suspended solids was siphoned off, leaving about 1.3 cm (1/2 inch) of liquid above the "mudline".

The material remaining in the cell was then reslurried with 2.5 L of deionized water and agitated for 1 minute; the pH range for all tests was 7.3 to 9.0 at this point. The flotation cell impeller was then turned off and the solids allowed to settle for another minute. The supernatant liquid-solid mixture was then siphoned off as before.

The two siphoned products were termed "slime reject product" and the material remaining in the cell at the end of the test was called the "purified zinc concentrate". All test products were filtered, dried in an oven overnight, weighed, and assayed.

#### Results and Discussion

Exploratory tests performed during December 1989 and January 1990 on the first sample (taken in July 1989) were promising. Based on these results, more tests were performed on this same sample during July 1990. However, the initial tests could not be duplicated; results from the tests performed in July 1990 were very different from those performed earlier. The differences may have been caused by aging due to chemical reactions occurring on the mineral particle surfaces. It was decided to obtain a fresh sample for further testwork.

The second round of tests (the subject of this report) was carried out in July 1990 on

the sample received in May 1990. The test without added reagents (test R41) was duplicated in October 1990 (tests R 50, 52) without any apparent aging effect, since repeatability was good.

Test results are given in Table 2. The silica rejection in the control tests performed without reagents (tests R41, R50, R52) was very low (4.3-10.4%, averaging 7.3%), indicating that only minimal dispersion took place. The silica content of the treated zinc concentrate (0.35-0.42%) was higher than the company specification of 0.3%. A relatively low amount of the feed (average 1.6 wt. %) was rejected to the slime product and zinc loss in this product was very low (average 1.5%).

Tests in which sodium silicate was added without tapioca starch (tests R31, R40, R46, R47) achieved fairly uniform results. The silica was well dispersed; average silica rejection was 60.7%. The amount of silica in the zinc concentrate was reduced to below the target level (average 0.23% silica). However, because some sphalerite was also dispersed by the sodium silicate, zinc losses were unacceptably high (average 13.1%). A substantial amount of the feed reported to the slime reject product (average 13.7 weight percent).

Use of tapioca starch with the sodium silicate was also effective in reducing the silica contamination in the zinc concentrate. The amount of silica was reduced below the target level (0.3% SiO<sub>2</sub>) in every test which used both reagents; the purified zinc product averaged 0.24% SiO<sub>2</sub> (cf. average of 0.23% SiO<sub>2</sub> with sodium silicate alone), representing an average silica rejection of 49.3%. The advantage of using both reagents vs. sodium silicate alone is that the added tapioca starch decreased the amount of zinc lost; this was accomplished by selectively flocculating the zinc, bringing more of it down into the settled purified zinc product, while leaving the silica relatively undisturbed and dispersed in the supernatant liquid. The average zinc loss using both sodium silicate and tapioca starch was reduced to 5.7%, down from 13.1% using sodium silicate alone. The zinc recovery ranged from 94.5 to 98.2%. One of the better tests, R32, resulted in 98.2% Zn recovery in the purified zinc product assaying 66.0% Zn and 0.24% SiO<sub>2</sub> while rejecting 40.2% of the silica in the slime reject product. Similar metallurgical results were obtained at several different reagent dosages, indicating that the same balance of the dispersing effects of sodium silicate and the flocculating effect of the tapioca starch could be achieved under various conditions.

It may be possible to carry out the selective flocculation operation using only the tapioca starch without sodium silicate. However, this is considered unlikely, because in order for selective flocculation to work, the silica must be dispersed so that it will report to the slime product; very little silica was dispersed and rejected in the control tests and it seems

improbable that the tapioca starch would both disperse silica and flocculate sphalerite. No tests were performed to test this hypothesis because of the limited amount of sample.

Figure 1 shows the effect of reagent dosages on silica rejection. Two general trends are apparent, although some outlier experimental scatter is evident. At any given level of sodium silicate addition, there is a marked difference in silica rejection between tests with no starch and tests with added starch. However, progressive increases in the starch level above 0.018 kg/tonne have no significant effect on the silica rejection, as by shown the horizontal tendency of the curves in Figure 1 as the starch dosage increases above this level. Conversely, at a given level of tapioca starch addition, increasing the amount of sodium silicate added resulted in greater silica rejection in the supernatant slurry through increased dispersion.

Figure 2 shows how reagent dosage affected zinc losses. Trends were similar to those noted for silica rejection. At any given level of sodium silicate dosage, there is again a sharp difference in zinc loss between tests with no starch and tests with added starch. However, progressive increases in the starch level above 0.018-0.035 kg/tonne have no significant effect on the zinc loss, as by shown the flattening tendency of the curves in Figure 2 as the starch dosage increases above this level. Also, zinc loss to the supernatant slurry increases as the sodium silicate level rises from 0.56 to 1.13 kg/tonne. However, present data show that further increase in the sodium silicate dosage does not appear to have a significant effect on zinc loss; this could be due to experimental error. Further testing would likely show that sodium silicate acts as a dispersant on sphalerite as well as the silica impurities in the concentrate.

The silica rejection is shown as a function of zinc loss in Figure 3. Note that the bulk of tests performed with reagents resulted in zinc losses of less than 6% while rejecting 38-56% of the silica. These tests all met the specifications for silica content in the purified zinc product. The control tests performed without added reagents (tests R41, R50, and R52) had very low impurity rejection (averaging only 7.3% of the silica) and the treated concentrate ranged from 0.35-0.42% SiO<sub>2</sub>, averaging 0.38%. Thus, none of the control tests met the specification for silica in the purified zinc product.

Because it contains significant zinc values, the slime reject product would have to be returned to the flotation circuit at a point yet to be determined. Further testwork would have to be carried out to determine the effect of the added sodium silicate and tapioca starch on subsequent zinc flotation.

The company has no immediate plans to

implement the selective flocculation scheme in the concentrator because of other corporate priorities, but may do so at some future date.

#### Conclusions

1) Analysis of the concentrate has shown that the -38  $\mu$ m (-400 mesh) fraction contains the bulk of the silica (43.3%) and 29.7% of the zinc.

2) Selective flocculation using sodium silicate and causticized tapioca starch is effective in reducing the silica contaminant level in Jersey Miniere zinc concentrate below the company specification (0.3% SiO<sub>2</sub>), while maintaining zinc recovery at 94.5-98.2%. One of the better tests resulted in 98.2% zinc recovery in the purified zinc product which assayed 66.0% Zn and 0.24% silica using 0.56 kg/tonne (1.13 lb/st) of sodium silicate and 0.070 kg/tonne (0.141 lb/st) of causticized tapioca starch.

3) Control tests performed without added reagents had very low impurity rejection (averaging only 7.3% of the silica). In these tests, the treated concentrate product contained an average of 0.38% silica. The specification for silica was not met in the purified zinc product in the control tests.

4) The dispersing effect of sodium silicate and the flocculating effect of the causticized tapioca starch must be balanced in order to achieve acceptable levels of both impurity rejection and zinc recovery.

#### Acknowledgements

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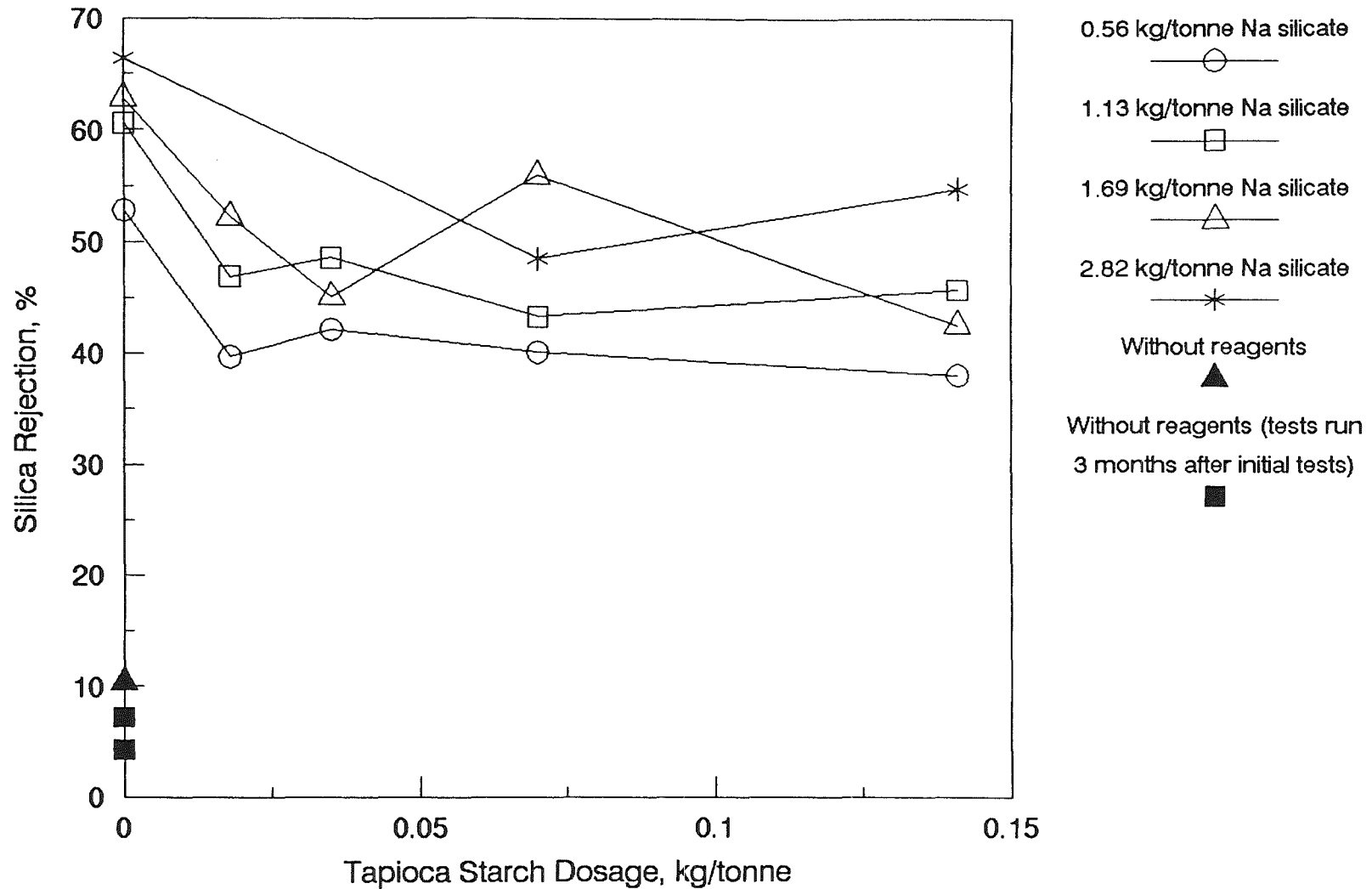
TABLE 1. Size analysis of Jersey Miniere zinc concentrate,  
as received May 23, 1990

Size (Tyler Mesh)	Weight, pct	Assay, pct				Distribution, pct			
		SiO <sub>2</sub>	CaO	MgO	Zn	SiO <sub>2</sub>	CaO	MgO	Zn
+35	1.4	0.64	0.08	0.05	57	2.0	0.4	0.6	1.3
35x48	6.5	0.70	0.10	0.08	59	10.5	2.2	4.4	6.4
48x65	8.4	0.49	0.07	0.08	62	9.6	2.1	5.7	8.7
65x100	10.5	0.32	0.04	0.03	62	7.8	1.5	2.8	10.9
100x150	12.4	0.21	0.03	0.03	58	6.0	1.2	3.3	12.0
150x200	11.3	0.38	0.03	<0.02	61	9.9	1.1	1.5	11.5
200x270	9.0	0.24	0.04	<0.02	61	5.0	1.3	1.2	9.2
270x325	5.7	0.32	0.06	0.03	60	4.2	1.1	1.5	5.7
325x400	4.7	0.14	0.10	0.05	59	1.6	1.6	1.9	4.6
-400	30.1	0.62	0.84	0.32	59	43.4	87.5	77.1	29.7
TOTAL (calc)	100.0	0.43	0.29	0.12	59.9	100.0	100.0	100.0	100.0
HEAD ASSAY		0.45	0.38	0.17	64.9				

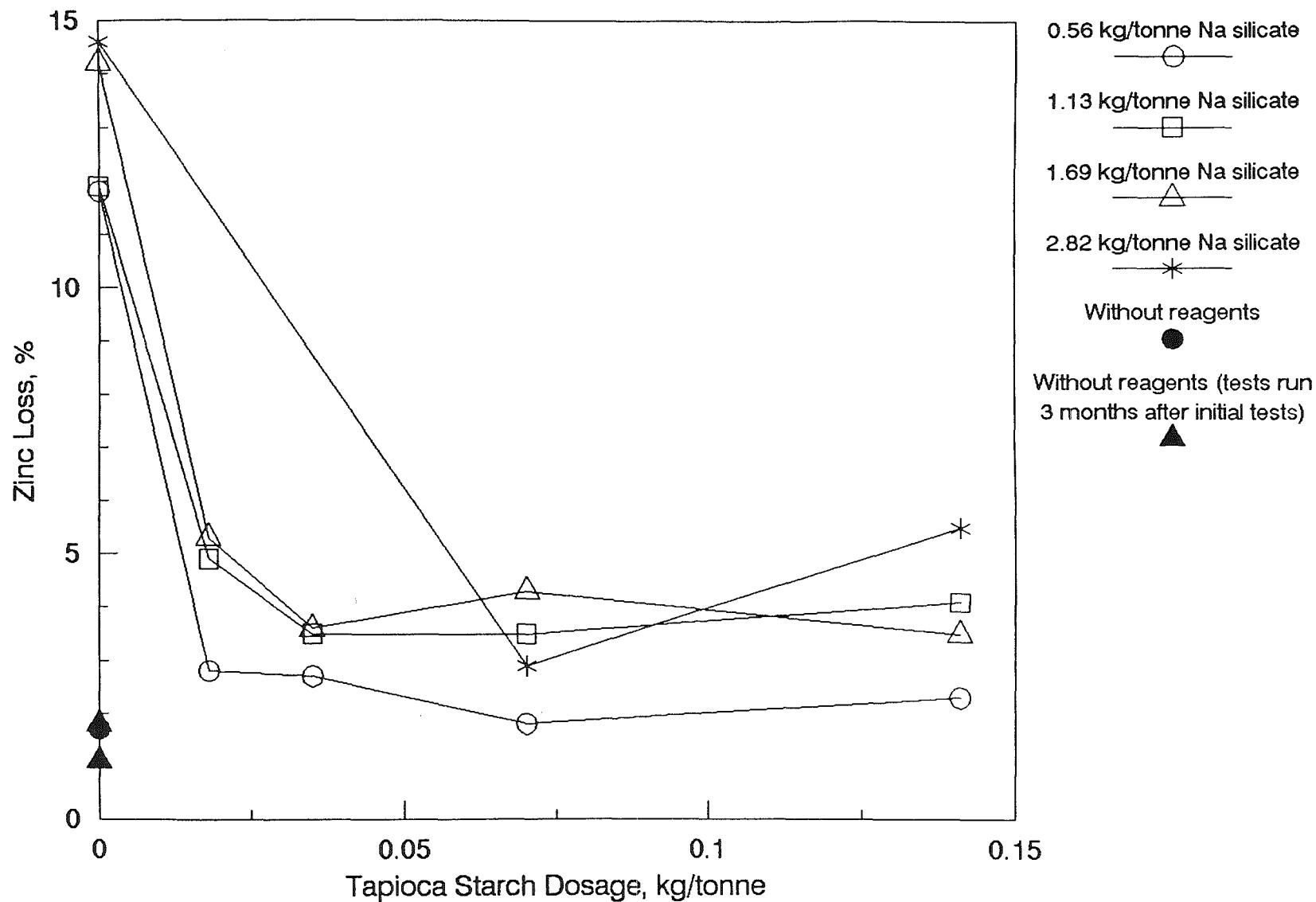
TABLE 2. JERSEY MINIERE ZINC CONCENTRATE SELECTIVE FLOCCULATION TESTS

Material: JMZ Zinc Concentrate, as received May 23, 1990

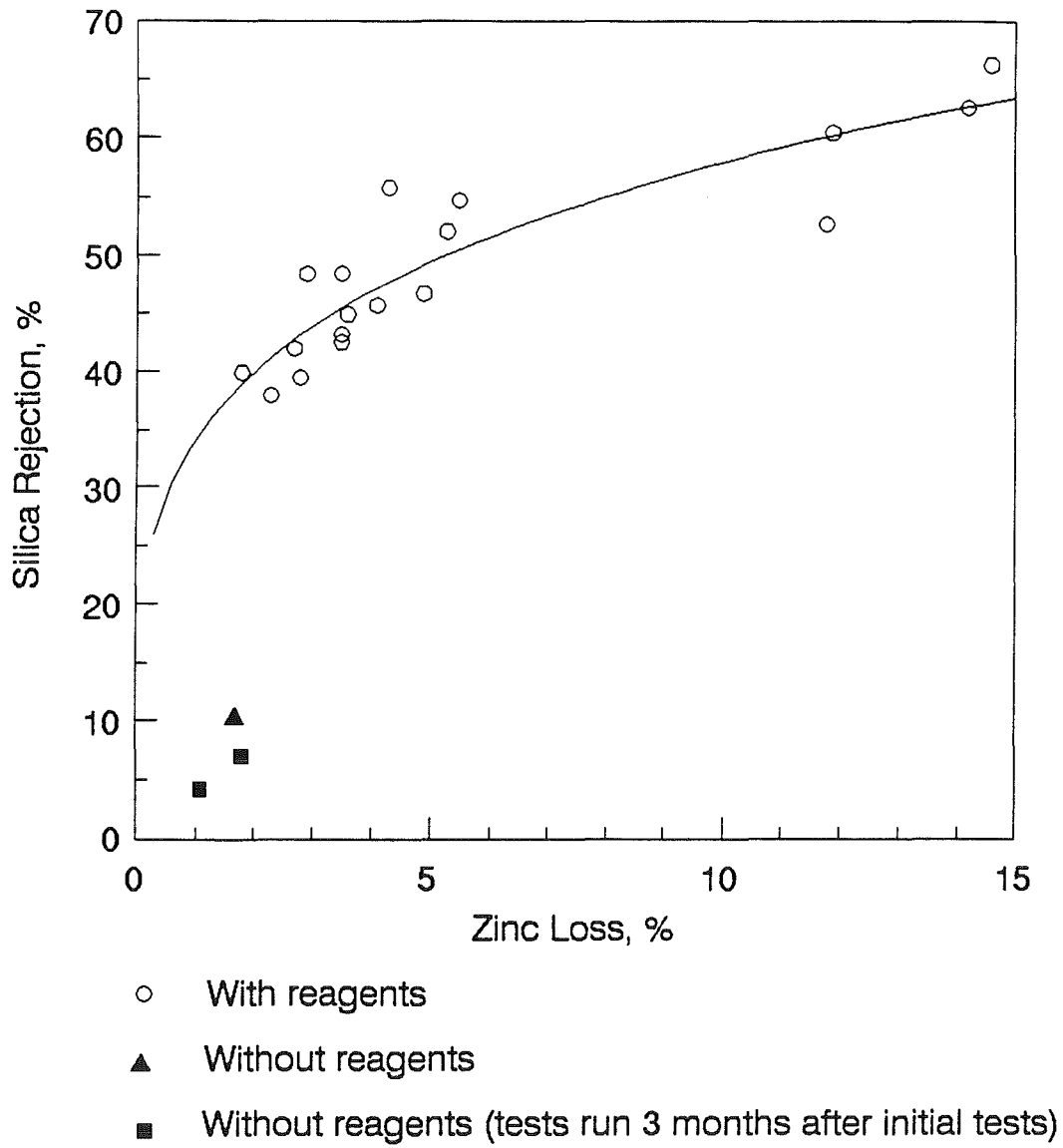
Test No.	Reagents (kg/tonne)		Purified Zinc Concentrate				Slime Reject Product				
	Na <sub>2</sub> SiO <sub>3</sub>	Tapioca starch	Wt.%	SiO <sub>2</sub> %	Zn %	% Zn Recovery	Wt.%	SiO <sub>2</sub> %	Zn %	% Silica Rejection	% Zn Loss
R41	0	0	98.2	0.35	65	98.3	1.8	2.2	62.7	10.4	1.7
R50	0	0	98.8	0.42	62.9	98.9	1.2	1.6	60.6	4.3	1.1
R52	0	0	98.2	0.37	59.6	98.2	1.8	1.6	59.7	7.2	1.8
R31	0.56	0	87.8	0.22	62	88.2	12.2	1.8	59.5	52.8	11.8
R35	"	0.018	96.6	0.23	65	97.2	3.4	4.3	53.8	39.7	2.8
R34	"	0.035	96.8	0.24	63	97.3	3.2	5.3	52.7	42.2	2.7
R32	"	0.070	97.6	0.24	66	98.2	2.4	6.7	49.8	40.2	1.8
R33	"	0.141	97.2	0.24	67	97.7	2.8	5.1	53.3	38.2	2.3
R40	1.13	0	87.1	0.23	65	88.1	12.9	2.4	59.5	60.6	11.9
R38	"	0.018	94.5	0.24	64	95.1	5.5	3.6	56.3	46.9	4.9
R37	"	0.035	95.8	0.22	66	96.5	4.2	4.8	55.1	48.6	3.5
R36	"	0.070	96.1	0.26	59	96.5	3.9	5.0	53.1	43.4	3.5
R39	"	0.141	95.3	0.27	65	95.9	4.7	4.6	55.7	45.9	4.1
R46	1.69	0	86.0	0.25	60	85.8	14.0	2.6	61.5	62.8	14.2
R44	"	0.018	93.9	0.23	60	94.7	6.1	3.9	51.9	52.2	5.3
R43	"	0.035	95.6	0.25	62	96.4	4.4	4.5	51.3	45.1	3.6
R42	"	0.070	94.8	0.25	64	95.7	5.2	5.8	52.9	56.0	4.3
R45	"	0.141	95.7	0.28	61	96.5	4.3	4.7	50.5	42.7	3.5
R47	2.82	0	84.4	0.22	61	85.4	15.6	2.4	56.5	66.4	14.6
R49	"	0.070	96.5	0.23	60	97.1	3.5	6.1	50.7	48.6	2.9
R48	"	0.141	93.7	0.24	60	94.5	6.3	4.3	51.7	54.9	5.5
Average of control tests without reagents			98.4	0.38	62.5	98.5	1.6	1.8	61.0	7.3	1.5
Average of tests with Na <sub>2</sub> SiO <sub>3</sub> only			86.3	0.23	62.0	86.9	13.7	2.3	59.3	60.7	13.1
Average of tests with both Na <sub>2</sub> SiO <sub>3</sub> & tapioca starch			93.7	0.24	62.8	94.3	6.3	4.3	54.2	49.3	5.7



**Fig. 1. EFFECT OF TAPIOCA STARCH DOSAGE ON SILICA REJECTION FOR VARIOUS LEVELS OF SODIUM SILICATE ADDITION**



**Fig. 2. EFFECT OF TAPIOCA STARCH DOSAGE ON ZINC LOSS FOR VARIOUS LEVELS OF SODIUM SILICATE ADDITION**



**Fig. 3. SILICA REJECTION AS A FUNCTION OF ZINC LOSS**