# PROCEEDINGS OF THE

## **FOURTH MINERAL WASTE UTILIZATION**

### **SYMPOSIUM**

INDUSTRIAL WASTES—SCRAP METAL—MINING WASTES—MUNICIPAL REFUSE



EUGENE ALESHIN EDITOR



COSPONSORED BY THE U.S. BUREAU OF MINES AND IIT RESEARCH INSTITUTE

MAY 7 AND 8, 1974 CHICAGO, ILLINOIS

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#### ACKNOWLEDGEMENT

It is a pleasure to acknowledge Linda Widlock and Nancy Spetyla for corrective and special typing. Joan Pawelski provided editorial assistance.

> Eugene Aleshin Editor

#### FOREWORD

Protection and preservation of the environment and our natural resources has been a growing concern for all facets of industry and government. There is an accompanying need for information on all subjects related to recovery and utilization of resources from waste materials. The major topics of this, the Fourth Mineral Waste Utilization Symposium, are mining and mineral processing wastes, industrial wastes, scrap metal and municipal refuse. The important areas of solid waste disposal, reuse and recycling are thoroughly explored.

Solid wastes are a by-product of our standard of living and they are accumulating at a rate more in line with our increase in national income than with our increase in population. A poor nation cannot afford the luxury of waste. Instead its dumps are turned over to scavengers to find uses for these wastes. The United

States must begin to look at its wastes in a similar manner because it can no longer afford this luxury.

Every time we fail to reuse a ton of iron or steel it takes two and one-half tons of iron ore, a ton of coal and 2,000 gallons of water to produce its replacement. We can no longer afford the energy, water and land resources to perform this task. We must consider used materials even when not economical at present prices. It has been stated that this process is like planting oak trees for future generations.

The objective of this meeting is to provide a mechanism for promoting innovative and rational approaches to resource recovery problems. It is hoped that this approach will help reduce the ecological fantasies that have grown up around the entire field of mineral and solid waste disposal.

Seymour A. Bortz Symposium Chairman

#### MY FAVORITE THINGS

Peach stones and dog bones and green-colored glasses;

Kleenex and Kotex and weird-smelling gases, Fish heads and feathers without any wings These are a few of my favorite things.

Road maps and hub caps and windshield wipers; Toothpaste and food waste and discolored diapers;

Sawed-in-half plaster casts, tied up in slings These are a few of my favorite things.

When the bag breaks,
And the sewer plugs-When I'm feeling sad,
I simply remember
My favorite things,
And then I don't feel
so bad!

Robert R. Richter Salt Lake City Metallurgy Research Center U.S. Bureau of Mines

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#### SESSION NO. 1

#### **GENERAL**

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A Paper in the PROCEEDINGS OF THE FOURTH MINERAL WASTE UTILIZATION SYMPOSIUM Jointly sponsored by the U.S. Bureau of Mines and IIT Research Institute Held in Chicago, Illinois on May 7-8, 1974

#### MINERAL WASTE UTILIZATION STUDIES

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#### ABSTRACT

This paper outlines the mineral waste utilization program of the Mineral Processing Division and summarizes studies relative to the recovery of raw material from mineral wastes for various ceramic, construction material, and mineral filler applications. Tailings from several metal mines, notably iron, uranium, and gold, and industrial mineral operations, specifically silica, were studied as source material for dry-pressed and sand-lime brick manufacture. High-strength bricks were made with several of these tailings. A brick of exceptionally high strength was produced by an autoclave process using dicalcium silicate slag from a magnesium metal operation.

Sand suitable for use in the manufacture of lower grade glass was recovered from uranium mill tailings by magnetic separation and froth flotation. Glass samples made with this sand were deemed acceptable from the standpoint of color but exhibited some residual radioactivity. A process for the manufacture of mineral wool from asbestos tailings and high quartz sand was developed. Good quality wool was produced on a laboratory basis. Plaster of paris was successfully produced from waste phosphogypsum by autoclave and kettle processes. Plaster and wallboard test specimens made with this material were of acceptable quality. Filler clay-wood fibre sludge from a paper plant was separated into its constituent components by flocculation and centrifugation. The products are believed to be suitable for re-use in paper manufacture. Two microfillers, fly ash and silica flour, were used as partial replacements for portland cement in concrete. The fly ash, alone, improved compressive strength at 20°C; However, both additives improved heat resistance, especially at temperatures up to 500°C. Expanded, blastfurnace-slag was used as an aggregate to produce good-quality, low-density concrete. Research is in progress on the use of high-quartz and high-carbonate mill tailings, ilmenite tailings, and nepheline syenite rejects, in various construction material and ceramic products.

#### MINERAL WASTE UTILIZATION STUDIES

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#### Introduction

Canada is fortunate in having large resources of most minerals. However, these resources are non-renewable. Minerals once mined and used by man cannot be restored, although their value may be extended to a degree by the recovery and recycling of discarded consumer goods, e.g., metal cans, glass, and paper. The rapid industrial growth of Canada during the present century has depleted many domestic mineral resources and there is concern, in some areas, over supplies for the future. Although the only significant shortages at present are sand and gravel for construction purposes near large centres, other shortages will develop unless corrective measures to ensure the effective use of these resources are initiated. By contrast, solid wastes such as mill tailings, slags, residues and sludges are being accumulated at an increasing rate. The resulting large waste piles or tailing ponds are becoming aesthetically and economically intolerable and, in some cases, are contributing to water pollution. Growing public awareness has focussed attention on the need to reduce the pollution potential of these wastes, but equally important is the possibility of enhancing the economics of a process by developing new products from these materials. These partially processed wastes or "man-made resources", undoubtedly will become increasingly attractive in the future as source material for the recovery of additional minerals and for the fabrication of useful products.

The Mines Branch of the Department of Energy, Mines and Resources, Ottawa, is concerned over the effects of these wastes on the environment and, as part of a departmental program of environmental improvement, is conducting research on their economic recovery and conversion to useful products. This research is concentrated in the Mineral Processing Division. This Division has occasionally investigated wastes of this nature in past years; however, its involvement has been minor and incidental to the main task of developing processes for the economic recovery of metals and minerals from specific ores. A systematic approach to mineral waste utilization is now being made. The Non-Metallic and Waste Minerals Section of the Division has been specifically charged with this responsibility; however, research in this area is also being carried on by the Construction Materials and Ceramic Sections.

#### Mineral Waste Research

#### General

Prior to initiating specific studies, an overall review of the type and variety of mineral wastes produced in Canada was made. These wastes were divided into four general categories as noted in Table I . A study of the information contained in this table showed the first two groups to be large-volume, low-value mixtures of minerals and as such, usually unattractive for further economic exploitation. Such waste might be used as subgrades for roads or as land-fill but, in most instances, the logical solution for wastes of this type is long-term, planned, stabilization and/or landscaping. Groups three and four, on the other hand, include wastes which have been partially processed and are often uniform in character and grain size. These wastes are of interest as possible sources of useful raw materials for the manufacture of construction and ceramic products, for the recovery of chemicals and minerals, and for use as mineral fillers in various applications.

#### Objective and Scope

The objective of research on mineral wastes is two-fold:

- the recovery of additional raw material, and
- the development of useful products from the recovered material.

The materials of chief interest in this program are the solid mineral wastes from mining and mineral processing, metallurgical, chemical, and pulp and paper plants that appear, on the basis of composition, tonnage, location, etc., to have re-use potential. Conservative estimates indicate that these industrial groups generate, respectively, 500 million, 13 million, 5 million, and 2 million tons of mineral wastes annually.

A five-point approach to wastes was adopted, as follows:

- 1. Inventory of wastes.
- 2. Selection of wastes for research projects.
- Examination, including mineralogy, chemical and physical characterization.
- 4. Beneficiation and conversion.
- 5. Process and/or product evaluation.

The mineral waste inventory has been, published in preliminary form 1-5 Although the information is not complete, this inventory pro-

TABLE 1 Classification of	n of Solid Mineral Wastes	•		
		Group and Type	d Type	
	1. Overburden	2. Gangue or waste rock	3. Mine and mill tailings	4. Metallurgical, chemical, 6 pulp and paper residues
Description	Soil, sand, clay, shale, gravel, boulders, etc.	Rock which must be broken and removed to obtain ore; many types, e.g., limestone, granitic and volcanic rocks.	Rock minerals, usually sand to slime sizes but sometimes larger; may include sulphides	Slags, fly ash, cinders, dust, slimes, sludges, etc.
Characteristics	Heterogeneous and unconsolidated.	Broken rock, usually homogeneous, but varying widely in size.	Usually homogeneous and uniform in character and size.	Usually homogeneous and uniform in character and size; sometimes toxic.
Examples	Cover removed from open pit coal, gypsum, and some iron mines.	Broken rock, usually from open pits, but also from underground sources.	Tailings from many diverse operations, e.g., base, ferrous and precious metal mines, and non-metallic mineral operations.	Slags from iron and steel plants, fly ash from power plants, salt from potash recovery operations.
Nature of Problem	Materials handling and storage; little intrinsic value but may be useful as fill, ballast, and in landscaping	age; little intrinsic value ballast, and in landscaping.	Materials handling and storage; may compete for valuable land space; unsightly and possible source of air and water pollutants; potential sources of additional metals and minerals.	e; may compete for valuable sible source of air and water of additional metals and
,		185 1 - 21 2 - 21	er s	

vides a basis for the classification of data and for the selection of waste materials for laboratory study.

#### Laboratory Studies

#### Source of Samples

Samples for laboratory study are obtained from many sources, primarily upon request by the Division, but frequently as a result of industry's awareness of our program and concern over accumulating tonnages of mineral waste.

Tailing samples from a cross section of Canadian metal mining operations in Newfoundland, Nova Scotia, New Brunswick, Quebec, Ontario, Manitoba, and British Columbia were examined during the initial phase of our laboratory study. Various physical characteristics of the tailings, e.g., particle size distribution, bulk density, specific gravity, and pH, were studied, along with data relative to chemical composition and mineralogy. Results indicated that, although there is little current likelihood of the further economic recovery of metals from metal-mine tailings, the recovery of non-metallic materials for re-use by industry appears to be technically feasible in many instances. With this in mind, several of the more promising samples were evaluated for use in certain construction material. ceramic product, and mineral filler applications. Samples from base metal mines, in most instances, were of little or no value for these purposes because of high metallic sulphide contents. Those from uranium and gold mines, on the other hand, generally contained relatively high percentages of quartz and other non-metallic minerals. Several of these latter samples showed promise for use in glass, and in dry-pressed and sand-lime brick manufacture.

Tollowing completion of this preliminary study, samples of tailings from select metal mines and industrial mineral operations, slags from metallurgical plants, chemical residues, and pulp and paper sludges, were requested from industry for detailed laboratory study.

In contrast to metal mines, most nonmetallic or industrial mineral operations do not generate significant tonnages of tailings. Where produced, these tailings are usually of consistent composition and readily responsive to beneficiation by known mineral processing techniques. Wastes from metallurgical operations vary widely in composition. Some are unique, e.g., a calcium silicate residue from the manufacture of magnesium metal by the Pidgeon process. Chemical residues vary in composition and are often complex. One of the more interesting chemical residues investigated was waste gypsum from the phosphate fertilizer industry. This material has considerable potential as a substitute for natural gypsum in gypsum products manufacture 7-11 Some work has been completed on the recovery of wood fibre and clay from pulp and paper sludges. Typical examples of work completed and in progress are given below.

#### Dry-Pressed Brick

Tailings from an iron ore mine in southern Quebec were studied as source material for brick manufacture. These tailings principally consisted of quartz, feldspar and amphibole with minor mica, magnetite and pyrite. They were similar, mineralogically, to copper and gold mill tailings that have been shown by researchers in the United States Bureau of Mines to be suitable for producing dry-pressed brick 12. A similar study was initiated using calcium lignosulfonate, a waste product from the pulp and paper industry, as binder. The tailings were mixed with 1.0 per cent binder and 6 per cent water and pressed into cylinders, cubes, and bricks for testing. The pressed samples were then fired over a 5- to 6-hr period to a maximum temperature of 1200°C.

Preliminary tests results were disappointing because of the formation of hair-line cracks in the specimens on firing. These fractures occurred at about  $600^{\circ}$ C and were believed to be due to quartz which expands on changing from the  $\alpha$  to  $\beta$  variety at 573 °C. Grinding, to 50 per cent minus 200 mesh, prior to pressing overcame this problem. Fired products fabricated from the tailings showed no evidence of bloating or blistering, generally showed strengths in excess of 5000 psi, and withstood exposure to 50 freeze-thaw cycles without damage  $^{13}$ , as required by Canadian Standard Specification (CSA) 82.1 (Burned Clay Brick). A flow diagram for this process is shown in Figure 1.

Mine officials are studying the feasibility of producing brick with these iron tailings to provide continuing employment for mine personnel following the expected exhaustion of ore reserves in another 5 to 6 years.

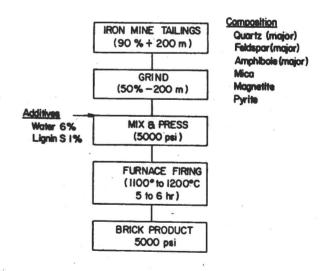


Figure 1 Flow diagram for brick production from iron mine tailings.

#### Sand-Lime Brick

Previous studies 14 have shown that sand-lime brick which met ASTM Specification C 73 (Specification for Calcium Silicate Face Brick) could be produced from high-quartz, gold mine tailings. The manufacture of sand-lime brick requires the availability of substantial quantities of inexpensive, fine, high-quality silica sand that is relatively free from clays, organics, and water soluble compounds. Several of the mine tailing samples examined contained high percentages of silica and were from deposits that were near potential markets. These were evaluated in the laboratory as source material for sand-lime building products manufacture.

The tailings (88 per cent) were blended with reagent-grade calcium hydroxide (12 per cent), moistened with water, and pressed into 1.7 in. diameter by 2 in. high cylinders at 5000 psi. The cylinders were autoclaved under steam at 14 atm (gauge) for 6 hrs. air dried, and tested for com-

Material	SiO <sub>2</sub> Wt %	Compressive Strength psi			
Standard (Buckingham Quartz)	100	7,150			
Gold tailings (Kirkland Lake, Ontario)	36	2,460			
Nickel-copper tailings (Sudbury, Ontario)	53	3,450			
Uranium tailings - fine - - coarse (Elliot Lake, Ontário)	76 88	3,060 5,470			
Silica tailings (southern Ontario)	98	5,000			
ASTM C73		4,500			

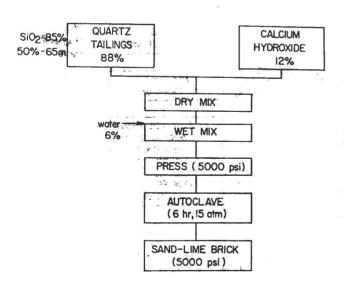


Figure 2 Flow diagram for sand-lime brick production from quartz tailings and calcium hydroxide.

pressive strength. A high grade, minus 14 mesh silica was used as a reference standard. Preliminary results are shown in Table 2. It is evident that only those products fabricated from tailings containing in excess of 85 per cent silica, and the standard, met ASTM specifications. Additional testwork is being carried out in an attempt to improve the quality of calcium silicate bricks produced with these and other mine tailings. A flow diagram for this process is shown in Figure 2.

#### Slag Brick

Waste dicalcium silicate slag (Ca2SiO4) is produced at an Ontario magnesium plant by the vacuum reduction of calcined dolomite with ferrosilicon at 1200°C. The slag dusts, or decrepitates into fine powder, on cooling as a result of the inversion of  $\beta$  to  $\gamma$  dicalcium silicate that occurs below 525°C. This crystalline change is accompanied by a 10 per cent expansion. The slag contains both  $\beta$  and  $\gamma$  dicalcium silicate and minor amounts of MgO. The  $\beta$  phase, which is a constituent of normal portland cement, has few or no hydraulic properties, probably due to deficiency of calcium oxide. The  $\gamma$  phase, however, is hydraulically active at temperatures above 150°C.

The air-cooled slag was screened on a 100-mesh sieve to remove most of the unreacted calcined dolomite. The minus 100-mesh fraction, representing 85 per cent of the total, was then autoclaved at 150°C (50 psig) to hydrate residual magnesium oxide. A filler such as quartz, dolomite or natural sand was added to the mix to aid the pressing operation and to improve product resistance to freeze-thaw. The product was then mixed with 8 per cent water and pressed at 5000 psi. The green bricks were autoclaved for 24 hours at 170° (100 psig). A brick with a smooth, uniform, grey surface was produced. Strengths exceeding 10,000 psi were obtained with sand or silica in 50 per cent additions. Various properties were investigated in accordance with ASTM C62 (Specification

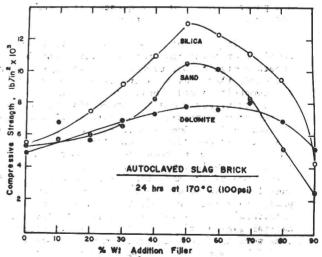


Figure 3 Effect of sand, and crushed silica and dolomite, on strength of slag brick.

for Clay Brick), and ASTM C 67 (Sampling and Testing Brick). Results showed that this sand-slag brick had properties comparable to good clay brick and superior to sand-lime brick. Figure 3 shows the effect on strength of additions of minus 20-mesh sand and crushed silica and dolomite. A flow diagram for this process is shown in Figure 4.

Company officials have expressed considerable interest in this work and are now studying the feasibility of using these tailings in brick manufacture.

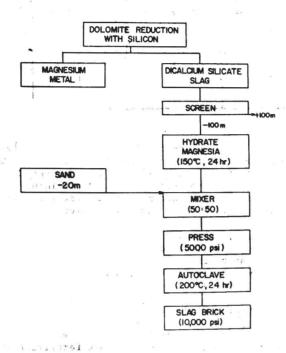


Figure 40 \*Flow diagram for slag brick

\*\*The state of a production from dicalcium

\*\*The state of a state of

#### Glass Sand

respectue or Following an inquiry by a major Canadian glass producer, an investigation was initiated early in 1972 to determine the feasibility of using high-quartz tailings from an Ontario uranium mine for glass manufacture. Mineralogical studies of minus 20-mesh tablings showed the principal constituents to be quartz (80 per cent) with lesser amounts of finely divided muscovite, potashfeldspar, pyrite and gypsum. From the standpoint of glass manufacture, the principal impurity was pyrite (7 to 8 per cent) which mostly occurred in finely divided particles attached to quartz grains. Two beneficiation approaches were tried - the first was essentially dry and included screening followed by magnetic and electrostatic separation; the second was wet and included sizing, magnetic vseparation, and froth flotation. Recovery of minus 28-, plus 150-mesh beneficiated sand was 60 to 65 percent in each case. Both products contained some residual pyrite. Chemical analyses of the products are in Table 3.

These analyses do not meet specifications for top quality sand which are: silica 99 per cent, iron 0.04 per cent (Fe<sub>2</sub>O<sub>3</sub>), and alumina 0.15 per cent. Samples of the as-

received material and of the two beneficiated products were incorporated in glass-batch mixtures which were then melted. The glass formed with the head sample was very dark green to black, and opaque. The glasses formed with the two beneficiated products were each transparent but tinged with green. A flow diagram for this process is shown in Figure 5.

Although this material undoubtedly could be used as a substitute for glass sand in the manufacture of coloured container ware, it is unlikely to find any large scale application for this purpose because of location, and impurity content. One point of particular concern with regard to the use of these tailings in glass is the question of residual radioactivity. This problem is being further studied.

Sample	Wt %								
	S10 <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	A1203	K	s	Total			
Head	85.40	5.13	3.63	0.75	4.28	99.1			
Product, dry process	93.40	0.25	3.78	0.80	0.22	98.4			
Product, wet process	94.60	0,20	2.63	0.57	0.11	98.1			

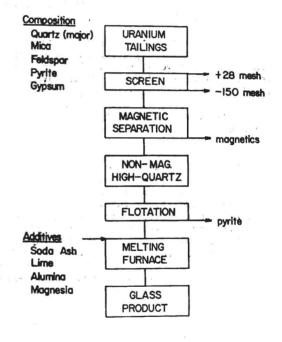


Figure 5 Flow diagram for production of glass from uranium mill tailings.

#### Mineral Wool .

Asbestos has been mined in the Eastern Townships of southern Quebec since 1878. Today's production of chrysotile asbestos from mines in that area is in the order of 1.3 million tons annually. Although asbestos mining continues to be

Sample		Wt %										
Jampie	MgO	SiO <sub>2</sub>	Fe <sub>2</sub> 0 <sub>3</sub>	н <sub>2</sub> о	A1203	CaO	Na <sub>2</sub> O	к <sub>2</sub> 0	N1	LOI	Tota	
Tailings No. 1	41.37	37.50	6.76	0.54	0.30	0.40	0.05	0.05	n.d.	13.29	100.26	
Tailings No. 2	38.85	38.65	8.32	0.54	0.83	0.56	0.06	0.09	n.d.	12.59	100.49	
Tailings No. 3	40.07	37.84	7.65	0.38	0.31	0.20	0.07	0.05	n.d.	13.41	99.98	
Melt No. 3	39.44	53.56	5.74	n.d.	0.85	0.84	n.d.	n.d.	0.08	n.d.	100.51	
(20% sand, 80% tailings)	7 70		1									
											1	

n.d. = not determined

a very vigorous and profitable industry, the fibre recovered represents only 5 to 10 per cent of the mined ore, so vast tonnages of tailings are produced every year. Considerable interest has been expressed by the asbestos industry and by others, including the Mines Branch, in the re-use potential of these tailings. As part of the research program on mineral wastes, the feasibility of producing mineral wool insulation with this material has been studied.

The asbestos tailings are primarily serpentine with minor magnetite and iron-nickel minerals. Samples were obtained from three Quebec mines representative of the producing area. The chemical analyses of these tailings, which are similar, are in Table 4, with that of an experimental melt formed from the tailings and high-purity sand.

Mineral wool was prepared from mixtures of asbestos tailings and a local high-quartz sand. Batches containing 15 to 20 per cent of sand were melted at 1540°C in a laboratory, single phase, two-electrode, 60-kVA arc furnace. As the melt was poured from the furnace it was blasted by a jet of high pressure air which caused it to disperse into small particles. These particles formed tails of mineral wool fibre. The wool was then pulled into a collector bin by a fan. Good quality wool was formed with mixtures of 70 per cent asbestos tailings and 30 per cent sand. 15

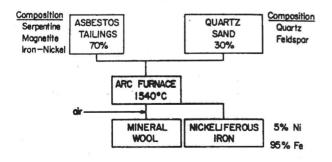


Figure 6 Flow diagram for mineral wool production from asbestos tailings and quartz sand.

An iron-nickel "button", conservatively estimated at 5 per cent nickel, remained in the bottom of the pot. This could be recovered and credited to processing costs. A preliminary study of capital costs and markets indicates a viable operation. A pilot plant study therefore appears warranted to firm up the technical and economic aspects of this process. Further laboratory studies will include the addition of ground glass and clay to the batch to lower melting temperatures. A flow diagram for this process is shown in Figure 6.

#### Gypsum Products

Mines Branch interest in phospho-gypsum, a waste product of the phosphate fertilizer industry, dates back to 1962, when a study of phospho-gypsum from a plant in southern Ontario demonstrated the technical feasibility of utilizing this material in gypsum products manufacture. Phospho-gypsums from three other plants, one in southern Quebec and two in Alberta, were subsequently investigated and found satisfactory for this purpose, 10.

Two approaches were considered in these studies:

- the production of a beneficiated, dry gypsum powder suitable for use in a standard calcining kettle; and
- (2) the production of beneficiated, moist gypsum suitable as feed to an autoclave.

The investigations, which included calcining and product fabrication and testing, were mostly confined to the first process because this appeared to be of greatest interest to the gypsum products industry of Canada.

On the basis of preliminary laboratory studies, a relatively simple treatment was developed for kettle calcining. Processing consisted of the following steps:

- remove, by wet screening, the plus 65-mesh high-impurity fraction;
- (2) water-wash the minus 65-mesh fraction to raise the pH to about 6, filter and dry;

3 ... 7

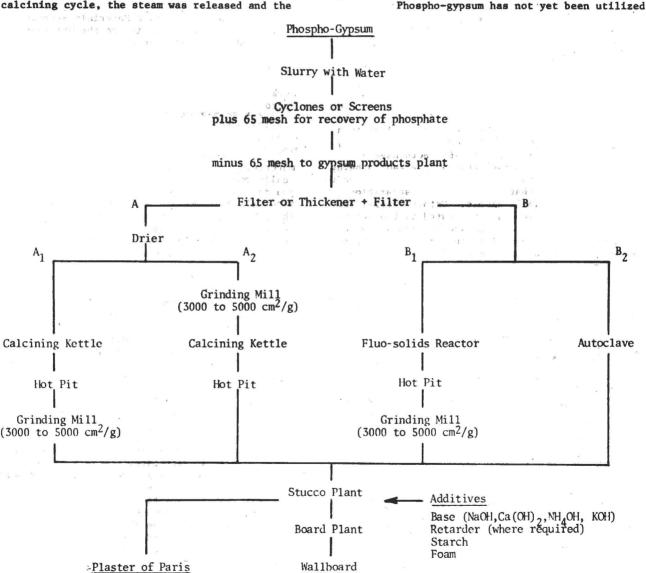
- (3) calcine the washed product to the hemihydrate stage and store the calcined product (stucco) in sealed polyethylene bags:
  - (4) grind to the surface area required for good bond-strength development, e.g., 3000 to 5000 cm<sup>2</sup>/g (Blaine fineness);
  - (5) determine consistency, time of set, strength, bond, etc. by CSA Specification A 82, (Gypsum Materials and Products).

The kettle product was acidic, set very quickly after mixing with water, and bonded rather poorly to paper during the fabrication of wallboard specimens. Various bases were employed to neutralize the acidity, the set was successfully retarded with commercial retarder, and the bond was markedly improved by adding starch, 7,9-11

202 In the autoclave process, the gypsum was washed, filtered, slurried with an equal weight of water, and calcined to the hemi-hydrate stage in a small laboratory reactor. Upon completion of the calcining cycle, the steam was released and the

excess liquor removed by siphon.

The autoclave product thickened very slowly and required several days to set and to develop significant strength, in contrast to the rapid set of stucco produced by calcining at atmospheric pressure. Further study indicated that this set retardation was due, in part, to unreacted or partially reacted phosphate rock. Removal of the plus 65-mesh, high-phosphate fraction before calcining, coupled with sulphuric acid treatment, either during or following calcining, effectively nullified the retarding effect of phosphate. Close control of set was achieved, in particular, by the addition of small quantities of concentrated sulphuric acid (2 per cent or less by weight) to the dewatered, calcined product. The product, so treated, set quickly to form a very strong plaster that bonded well with wallboard paper and, although acidic, did not exhibit any marked detrimental effect. A flow diagram for processing pho A flow diagram for processing phosphogypsum is shown in Figure 7.



Flow diagram showing four possible routes,  $A_1$ ,  $A_2$ ,  $B_1$ , and  $B_2$ , for the manufacture of gypsum products from phospho-gypsum.