STATE OF ARKANSAS

Arkansas Resources and Development Commission
Charles R. Bowers, Executive Director

DIVISION OF GEOLOGY
Norman F. Williams, Director

INFORMATION CIRCULAR 16

CERAMIC EVALUATION OF ARKANSAS NEPHELINE SYENITE

By
William J. Smothers
University of Arkansas Institute of Science and Technology
Norman F. Williams
Arkansas Resources and Development Commission
Harry J. Reynolds
University of Arkansas Institute of Science and Technology

Little Rock

1952

STATE OF ARKANSAS

Sid McMath, Governor

Arkansas Resources and Development Commission

Charles R. Bowers, Executive Director

COMMISSIONERS

Marion L. Crist, Chairman	Little Rock
W. S. Fox, Vice-chairman	Pine Bluff
Clarence Byrns, Secretary	Fort Smith
L. L. Baxter	Fayetteville
L. R. Branting	Bauxite
Robert L. Burton, Sr.	Benton
LeRoy Carter	Leachville
K. A. Engel	Little Rock
Dr. T. W. Hardison	Morrilton
Earl A. Harris	Rogers
Henry Koen	Harrison
W. T. Murphy, Jr.	Texarkana
George Peck	Hope
L. V. Ritter	Marked Tree

DIVISION OF GEOLOGY

Norman F. Williams, Director

Contents

List of Tables

ABSTRACT

Described herein are the results of an investigation to determine the occurrence, beneficiation, and ceramic application of Arkansas nepheline syenite.

The excellent fusion characteristics and the high-strength glassy phase of the Arkansas nepheline syenite make it a desirable constituent for use in the manufacture of colored ceramic products. The presently available Arkansas syenite has its greatest application in the manufacture of structural clay products, abrasive wheels, and low-voltage electrical porcelain and glaze.

FOREWORD

Part I of this study is a review of an investigation conducted by the Geology Division of the Arkansas Resources and Development Commission (Crockett, William E., and Harold B. Foxhall, "Preliminary Report on Arkansas Nepheline Syenite," Bull. Am. Ceram. Soc. *27*, 64-7, 1948). Part 11 contains a description of another investigation which was sponsored by this Commission. W. J. Knapp, at that time assistant professor in the ceramic department at the Missouri School of Mines and Metallurgy, was the principal investigator. Parts III and IV are reports on investigations which were conducted by the University of Arkansas Institute of Science and Technology, in cooperation with the Arkansas Resources and Development Commission. Part V is the summary and conclusions.

The work of the U. S. Bureau of Mines, obtained through the assistance and coöperation of R. G. Knickerbocker (Chief, Minerals Technology Division, Region VI, Rolla, Mo.) is referred to in several parts of this study and it is gratefully acknowledged.

INTRODUCTION

Deposits of nepheline syenite outcrop at scattered localities in the central part of Arkansas. The largest exposure, which covers an area of six square miles, is located immediately south of Little Rock on Fourche, or Granite, Mountain. This outcrop is the dome or boss of a large, buried batholith which was intruded during upper Cretaceous time into the metamorphosed, intensely-folded Paleozoic sediments that comprise the extreme eastern part of the Ouachita Mountains.

The occurrence of these deposits was reported as early as 1835 but it was not until 1890 that they were correctly identified.

Manufacturers of ceramic products in the United States rely solely upon Canada to supply them with nepheline syenite. Approximately 60,000 short tons of Canadian syenite were imported in 1949, primarily for use in the manufacture of glass.

Part One

Preliminary Report on Arkansas Nepheline Syenite

Petrographic and Chemical Analyses

Hornblende nepheline syenite (hereafter referred to as blue syenite) is the predominant rock type which comprises about five sixths of the outcrop on Fourche Mountain near Little Rock. A much smaller, elongate exposure on the northwest limb of the mountain is composed of a biotite nepheline syenite (gray syenite). Results of microscopic examination of thin sections of the blue and gray syenite, including relative percentages of minerals present, are presented in Table 1. The two types, although remarkably similar in chemical composition (Table 2), exhibit distinct physical differences, particularly in coloring. Both the orthoclase and the nepheline in the gray type are more completely altered than they are in the blue type, and the extensive kaolinization of the orthoclase probably causes the gray color. The principal ferromagnesian mineral in the blue type is the amphibole hornblende, whereas the chief ferromagnesians in the gray type are biotite and aegirine.

Ceramic Body Tests

A mixture of 300-mesh blue syenite, 300-mesh gray syenite, and 200-mesh commercial syenite was prepared for a body series test (Table 3). The body was chosen arbitrarily in order to magnify the effect of the flux content. Test specimens, one inch in diameter and six inches long, were prepared by dry mixing, blunging, drying on plaster, extruding, and marking. The specimens were fired at different temperatures in a Remmey Number 2150 test kiln for seven hours. Test data are presented in Table 4. Bodies B-1 and B-2 exhibit unusually early vitrification properties and bloat slightly at approximately Cone 7. These phenomena possibly are due, in part, to the fine division of the component syenites.

Purification by Magnetic Separation

Preliminary tests to recover Fe_2O_3 from the splits were carried out on a triple-rotor machine (Exolon Company). The percentages of Fe_2O_3 recovered are presented in Table 5. Obviously, too much iron is present in the separated products; therefore, it cannot be used in many ceramic applications.

Purification by Flotation

The U. S. Bureau of Mines, Metallurgical Branch, Rolla Division, performed flotation tests on both the blue and the gray nepheline syenite, and the flotation tailing products were separated into magnetic and nonmagnetic fractions in a Frantz ferrofilter. Each sample was stage-ground in a pebble mill to minus 200 mesh, and conditioned with one pound of sodium silicate per ton of ore. Biotite and aegirite were floated from the blue syenite with 0.16 pound of D. P. 243 (E. I. du Pont de Nemours & Company) and 0.32 pound of oleic acid per ton of ore. They were floated from the gray syenite with 0.24 pound of D. P. 243 and 0.48 pound of oleic acid per ton of ore (Table 6).

Purification of Arkansas Syenite by Tabling

A portion of the blue syenite was classified and tabled by Bureau of Mines personnel, and the table tailing was separated into magnetic and nonmagnetic fractions in a high-intensity Frantz ferrofilter to produce a nepheline product which is low in iron content (Table 7).

TABLE 1. PETROGRAPHIC ANALYSIS OF ARKANSAS SYENITES

	Hornblende nepheline syenite (blue syenite)		Biotite nepheline syenite (gray syenite)	
Texture	Granitoid		Granitoid	
Structure	Massive		Slightly tracitoid	
Mineralogy				
Primary minerals: Per cent	60	Orthoclase	50	Orthoclase
	10	Nepheline	15	Nepheline
	10	Hornblende	10	Biotite
	5	Diopside	10	Aegirine and diopside
Accessory minerals: Per cent	15	Titanite Apatite Magnetite Biotite	10	Titanite Apatite Magnetite Fluorite Pyrite
Secondary minerals: Per cent	Trace	Kaolinite Analcite	5	Kaolinite Analcite Cancrinite (?) Chlorite

TABLE 2. CHEMICAL ANALYSES OF CRUDE ORES[1]

	Hornblende nepheline syenite (blue syenite)	Biotite nepheline syenite (gray syenite)
Silica (SiO_2)	60.30	60.75
Alumina (Al_2O_3)	19.93	19.59
Ferric oxide (Fe_2O_3)	4.67	4.26
Titania (TiO_2)	1.10	1.20
Lime (CaO)	1.27	.76
Magnesia (MgO)	1.19	.92
Soda (Na_2O)	6.25	6.68
Potash (K_2O)	5.30	5.90
Ignition loss	.11	1.32

[1] Analyses by T. W. Carney, chemist, Arkansas Resources and Development Commission.

TABLE 3. TEST-BODY COMPOSITIONS

Designation	B-1	B-2	B-3
Blue syenite	30	—	—
Gray syenite	—	30	—
Commercial syenite	—	—	30
Ball clay	25	25	25
China clay	25	25	25
Flint	20	20	20

TABLE 4. PROPERTIES OF UNFIRED AND FIRED TEST BODIES

Unfired Bodies

Body	Water of plasticity	Linear drying shrinkage	Modulus of rupture
Number	*Per cent*	*Per cent*	*Lb./sq. in.*
B-1	23	3.6	262
B-2	28	5.0	268
B-3	22	4.4	241

Fired Bodies

Cone and temperature	Body	Linear firing shrinkage	Total linear shrinkage	Absorption	Fired color
Cone	*Number*	*Per cent*	*Per cent*	*Per cent*	
02 (1095°C)	B-1	8.0	12.1	2.8	Gray
	B-2	7.6	13.0	3.3	Gray
	B-3	5.2	9.1	8.8	White
1 (1125°C)	B-1	8.4	12.8	2.0	Gray
	B-2	8.0	13.4	2.5	Gray
	B-3	5.6	9.8	7.0	White
3 (1145°C)	B-1	9.0	13.0	1.6	Gray
	B-2	9.4	14.6	2.3	Gray
	B-3	6.0	10.0	6.5	White
5 (1180°C)	B-1	9.0	13.2	1.4	Gray
	B-2	9.6	14.6	2.1	Gray
	B-3	8.4	12.6	.6	Light cream
9 (1250°C)	B-1	8.6	12.6	.6	Gray
	B-2	9.6	14.6	.2	Gray
	B-3	8.0	12.8	.2	Cream
11 (1285°C)	B-1	7.6	11.4	2.3	Gray
	B-2	8.0	13.2	3.0	Gray
	B-3	8.4	13.0	.3	Cream

TABLE 5. MAGNETIC SEPARATION TESTS

Split designation	Percentage of total sample	Percentage of Fe_2O_3
Blue syenite (most magnetic portion)	23.4	11.4
Blue syenite (intermediate magnetic portion)	26.8	3.6
Blue syenite (least magnetic portion)	4.2	2.0
Blue syenite (nonmagnetic portion)	45.6	1.1
Gray syenite (most magnetic portion)	33.2	7.2
Gray syenite (intermediate magnetic portion)	13.2	4.4
Gray syenite (least magnetic portion)	18.6	2.7
Gray syenite (nonmagnetic portion)	35.0	1.7

TABLE 6. FLOTATION AND MAGNETIC SEPARATION TESTS

	Blue Syenite					Gray Syenite				
		Analysis					Analysis			
Product	Weight	Al_2O_3	SiO_2	TiO_2	Re_2O_3	Wt.	Al_2O_3	SiO_2	TiO_2	Fe_2O_3
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Biotite-aegirite	21.2	16.64	46.00	3.80	13.90	28.7	15.96	49.48	3.78	10.70
Tailing:										
Magnetic	15.2	20.57	60.40	0.58	2.49	12.6	20.31	62.90	0.37	1.64
Nonmagnetic (nepheline)	63.6	21.48	61.24	.29	.89	58.7	21.05	61.86	.15	1.07
Composite	100.0	20.31	57.88	1.07	3.89	100.0	19.49	58.43	1.21	3.90

TABLE 7. TABLING AND MAGNETIC SEPARATION OF BLUE SYENITE

		Analysis			
Product	Weight	Al_2O_3	SiO_2	TiO_2	Fe_2O_3
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Table concentrate (iron)	10.9	10.74	37.42	5.80	25.20
Table middling	11.7	19.60	60.94	.68	2.16
Slims	13.2	19.80	59.96	.68	2.70
Table Tailing:					
Magnetic (nepheline)	18.0	20.36	60.60	0.41	3.00
Nonmagnetic	46.2	22.11	62.00	.24	.85
Composite	100.0	19.95	58.67	0.98	4.28
Heads	——	19.93	60.30	1.10	4.68

Part Two

Ceramic Tests Using Beneficiated Arkansas Nepheline Syenite

Preliminary Investigations

X-ray spectrographs of the blue and gray Arkansas syenites were prepared to supply additional data on their mineralogical compositions. The X-ray patterns were characteristic of orthoclase with the exception of a few lines which overlapped with those of orthoclase; therefore, it was difficult to effect resolution. However, it appeared from the limited resolution which was effected that the ratio between the orthoclase and the predominate secondary mineral was 4:1 or 5:1.

Processed samples and short chemical analyses were provided by the U. S. Bureau of Mines, Metallurgy Branch (Table 8).

Because of the high orthoclase content of the Arkansas nepheline syenite, fusion block tests were made in which the following materials were tested: Lakefield nepheline syenite, Buckingham potash feldspar, and Arkansas blue and gray nepheline syenite. A heating schedule of 15°C per minute to 1000°C, and 8°C per minute to 1400°C was used. The Lakefield syenite fused completely; the potash feldspar did not start to fuse; and the Arkansas syenites fused between these two limits, the extent of fusion decreasing as the degree of beneficiation was increased. The gray syenite fused more readily than did the blue.

To evaluate the fusion tests further, the particle size distributions of potash feldspar, Lakefield syenite, and beneficiated Arkansas syenites were investigated (Table 9). The larger amount of the fine sizes in the Arkansas syenites would tend to make them fuse more readily.

Another factor upon which fusion temperature and the ultimate value in ceramic application depends is the silica-alumina ratio. Empirical formulae of the blue and gray Arkansas syenites were calculated from the chemical analyses (Part I) without consideration of the iron and the titanium oxide content:

Arkansas blue syenite

0.26 K_2O
0.48 Na_2O 0.92 Al_2O_3 4.72 SiO_2
0.11 CaO
0.15 MgO

Arkansas gray syenite

0.30 K_2O
0.52 Na_2O 0.93 Al_2O_3 4.88 SiO_2
0.07 CaO
0.11 MgO

The silica-alumina ratios of the Lakefield nepheline syenite and Buckingham potash feldspar were calculated from available chemical analyses, as well as from the short chemical analyses of the Arkansas syenites provided by the U. S. Bureau of Mines (Table 10).

The silica-alumina ratio of the Arkansas syenite before beneficiation approached that of the Buckingham feldspar, but this ratio was decreased by beneficiation.

Thus, preliminary investigations of the crude and beneficiated Arkansas nepheline syenites indicated that their action in ceramic compositions would be similar to that of feldspar, except for modification caused by the presence of secondary minerals.

Ceramic Body Tests

A body series of the same formula as that contained in Part I was prepared with the three beneficiated Arkansas syenites and Lakefield syenite used as body constituents (Table 11). Because only a limited quantity of treated syenite was available, the extrusion method of forming could not be employed; therefore, the test pieces were hand-molded, with the water of plasticity approximately equal to that reported in Part I. After the series was fired in a Globar-type kiln, data on fired test pieces were obtained (Table 12).

With the exception of coloring, the four type bodies displayed approximately the same firing changes. The bodies which contained the lower fusion-point Lakefield syenite showed more rapid and extensive changes than did the bodies of beneficiated Arkansas syenite. All four bodies attained minimum volume and maximum bulk specific gravity at Cone 5, and they also developed a vesicular structure above Cone 5. The absorption decreased abruptly between Cones 5 and 7, and approached zero at Cone 11.

The Arkansas syenite which was processed by tabling was still found to contain considerable iron-bearing minerals, and these were not sufficiently disseminated to produce a uniform color. The table tails (Sample 1) might be benefited further by grinding them finer. This would produce greater recovery in the tabling process and would aid in the dissemination of color-producing particles. Further grinding of the table middlings (Sample 3) would result in intensifying the already-excessive discoloration in the fired compositions. The color of fired samples which contain the flotation tails (Sample 2) approaches that of fired samples which contain Lakefield syenite.

Water of plasticity measurements and shrinkage of the samples were as follows:

Body Number	*Water of plasticity (per cent)*	*Linear drying shrinkage (per cent)*
B-1	23.89	5.8
B-2	23.30	5.3
B-3	23.51	5.2
B-4	25.64	5.7

Glass Compositions

The beneficiated Arkansas and Lakefield syenites were incorporated in window glass compositions and in compositions for high-alumina experimental glass (Table 13).

The batches were placed in crucibles made of kyanite clay; they then were heated in a gas-fired pot furnace to 1450-1500°C, and held at maximum temperature for 30 minutes. The resulting glasses were poured on steel and pressed to form a sheet 7/32 of an inch thick, after which the glass was annealed.

The melting qualities of glasses which contained the different syenites were similar. The higher alumina content of the glasses formed by the experimental compositions made them more viscous than those formed by the window glass compositions. The color changes produced by the additions of syenites were:

1. Arkansas blue syenite table tails: more intense discoloration by iron than produced by Lakefield syenite.
2. Arkansas blue syenite flotation tails: color comparable to that produced by Lakefield syenite.
3. Arkansas gray syenite table middlings: most intense iron discoloration of the three samples.

Porcelain Enamel

Antimony-opacified cover-coat frits were made, the alumina for which was furnished either by Buckingham feldspar or by beneficiated Arkansas syenites (Table 14).

The batches were melted in clay crucibles and the melts were quenched in water to produce the frit. Cover-coat batches included the following materials:

Component	*Parts*
Frit	100
"X" clay[1]	7
Opacifier (Opax[2])	6
$MgCO_3$	1¼
Water	40

The batches were milled until 95 per cent passed a 200-mesh screen.

The following ground-coat batch was mixed and milled to the same degree of fineness:

Component	*Parts*
1005 frit[1]	40
1008 frit[1]	30
1009 frit[1]	30
Lakefield nepheline syenite	10
Borax	¼
Water	40

Test pieces of sheet steel were dipped in the ground-coat slip, dried, and fired at 1500-1540°F for 3 to 4 minutes. After the pieces were cool, the specimens were dipped in the cover-coat slip, dried, and fired at 1440-1470°F for experimentally determined periods of time.

A firing time of 1½ to 2 minutes at 1440-1470°F was sufficient to fire the cover-coat enamels which contained the blue syenite flotation tails and the gray syenite table middlings. The firing time for the enamels which contained Arkansas syenite was less than that for the enamel which contained feldspar. The latter required 3 to 4 minutes' exposure at 1450°F. The adherence of all the enamels appeared to be satisfactory.

The opacity, gloss, and color of the enamel coatings which contained Arkansas beneficiated syenites compared favorably with those of the enamels which contained Buckingham feldspar.

---

[1] Chicago Vitreous Products Company.

[2] National Lead Company, Titanium Alloy Manufacturing Division.

TABLE 8. ANALYSIS OF BENEFICIATED ARKANSAS NEPHELINE SYENITES

Sample	Product	Analysis			
		SiO_2	Al_2O_3	Fe_2O_3	TiO_2
Number		*Per cent*	*Per cent*	*Per cent*	*Per cent*
1	Blue nepheline syenite Table tails, nonmagnetic fraction	62.00	22.11	0.85	0.24
2	Blue nepheline syenite Flotation tails, nonmagnetic fraction	61.16	23.77	.85	.05
3	Gray nepheline syenite Table middlings, nonmagnetic fraction	61.92	20.74	1.08	.10

TABLE 9. PARTICLE SIZE DISTRIBUTION OF POTASH FELDSPAR, LAKEFIELD SYENITE, AND BENEFICIATED ARKANSAS SYENITE

U.S.B.S. Sieve number	Sieve opening (mm)	Lakefield syenite	Buckingham potash feldspar	Beneficiated Arkansas syenite (per cent finer)		
				Sample 1	Sample 2	Sample 3
60	0.250	99.92	92.99	99.96	99.99	99.97
80	.177	97.97	99.81	99.92	99.97	99.48
120	.125	35.28	84.21	99.86	99.91	98.58
170	.088	18.38	60.50	99.75	99.75	97.26
230	.062	11.38	50.50	93.50	99.46	70.24
325	.044	7.92	26.00	75.60	87.33	46.22

TABLE 10. SILICA-ALUMINA RATIOS OF FELDSPAR AND NEPHELINE SYENITES

Material	Silica-alumina ratio
	Alumina=unity
Buckingham potash feldspar	5.8
Lakefield nepheline syenite	4.0
Arkansas blue nepheline syenite:	
Before beneficiation	5.1
Table tails, nonmagnetic	4.8
Flotation tails, nonmagnetic	4.4
Arkansas gray nepheline syenite:	
Before beneficiation	5.3
Table middlings, nonmagnetic	5.1

TABLE 11. BODY COMPOSITION FOR EVALUATING BENEFICIATED ARKANSAS SYENITES

Constituents	Body designation 1	2	3	4
	Per cent	*Per cent*	*Per cent*	*Per cent*
Blue nepheline, table tails, nonmagnetic fraction	30	—	—	—
Blue nepheline, flotation tails, nonmagnetic fraction	—	30	—	—
Gray nepheline, table middlings, nonmagnetic fraction	—	—	30	—
Lakefield nepheline	—	—	—	30
Light, Kentucky ball clay	25	25	25	25
North Carolina kaolin	25	25	25	25
Potter's flint	20	20	20	20

TABLE 12. PROPERTIES OF BODIES FOR EVALUATING BENEFICIATED ARKANSAS SYENITES

Cone and temperature	Body designation	Linear firing shrinkage	Total linear shrinkage	Absorption	Bulk specific gravity	Color
Cone		*Per cent*	*Per cent*	*Per cent*	*gm/cc*	
02 (1095°C)	1	2.5	8.7	9.9	1.84	Light cream, speckled
	2	2.5	8.1	9.0	1.81	White
	3	2.2	7.7	14.1	1.80	Very light gray, speckled
	4	2.6	8.7	12.3	1.84	White
1 (1125°C)	1	3.6	9.8	9.4	1.96	Light cream, speckled
	2	3.1	8.8	8.7	1.94	White
	3	2.6	7.8	9.1	1.95	Very light gray, speckled
	4	3.9	10.0	9.4	1.96	White
3 (1145°C)	1	4.3	10.5	9.0	1.97	Light cream, speckled
	2	4.0	9.7	8.5	1.99	Light cream
	3	3.3	8.9	8.3	1.99	Very light gray, speckled
	4	4.8	10.9	8.2	2.00	White
5 (1180°C)	1	9.0	15.5	8.5	2.32	Light cream, speckled
	2	9.4	15.3	8.2	2.33	Light cream
	3	9.4	15.2	7.8	2.35	Light gray, speckled
	4	11.1	17.5	7.0	2.42	Cream
7 (1210°C)	1	6.3	12.6	4.0	2.16	Very light gray, speckled
	2	6.8	12.6	3.5	2.18	Light cream
	3	7.0	12.7	3.2	2.20	Light gray, speckled
	4	8.7	15.0	2.8	2.30	Cream
9 (1250°C)	1	7.1	13.5	2.3	2.20	Very light gray, speckled
	2	7.3	13.1	2.3	2.18	Very light gray
	3	7.6	13.3	1.9	2.21	Light gray, speckled
	4	9.2	15.5	1.1	2.29	Very light gray
11 (1285°C)	1	7.2	13.6	.2	2.21	Light gray, speckled
	2	7.6	13.4	.3	2.23	Very light gray
	3	7.7	13.5	.3	2.22	Light gray, speckled
	4	9.1	15.4	.15	2.29	Very light gray

TABLE 13. BATCH AND MELTED COMPOSITIONS OF GLASSES CONTAINING NEPHELINE SYENITE

Raw material	Batch		Oxide	Melted	
	Window glass composition	Experimental glass composition		Window glass composition	Experimental glass composition
	Per cent	*Per cent*		*Per cent*	*Per cent*
Silica	62.0	48.2	SiO_2	71.2	69.0
Soda ash	20.6	16.8	Al_2O_3	1.6	5.3
Whiting	14.3	13.2	Fe_2O_3	.1	.2
$MgCO_3$	5.7	.0	CaO	8.8	8.7
Salt cake	.5	1.4	MgO	3.0	.2
Cryolite	1.0	1.0	Na_2O	14.2	13.8
Nepheline syenite	5.9	19.6	K_2O	.3	1.3
			SO_3	.3	.9
			F	.5	.6

TABLE 14. BATCH AND MELTED COMPOSITIONS OF COVER-COAT PORCELAIN ENAMEL FRITS

Raw material	Batch		Oxide	Melted	
	Frit A	Frit B		Frit A	Frit B
	Per cent	*Per cent*		*Per cent*	*Per cent*
Buckingham feldspar	24.9	0.0	SiO_2	44.4	44.4
Arkansas beneficiated syenite	.0	20.4	Al_2O_3	8.0	8.0
Borax	23.2	22.6	B_2O_3	9.6	9.6
Flint (silica)	22.6	25.6	Na_2O	14.9	14.9
Soda ash	3.8	5.6	K_2O	4.8	4.8
Fluorspar	4.1	3.9	CaO	3.4	3.2
Soda nitre	5.6	.0	F	8.5	8.5
Cryolite	10.2	10.0	MgO	.0	.2
Antimony oxide	5.6	5.5	Sb_2O_3	6.4	6.4
Potassium nitrate	.0	6.4			

Part Three

Beneficiation of Processed Arkansas Nepheline Syenite

Commercial Mining of Arkansas Syenite

The Minnesota Mining and Manufacturing Company established a plant for the manufacture of roofing granules near Little Rock in 1947. In it nepheline syenite is crushed, screened, and colored to produce the granules. The particle size range for this product is comparatively narrow. Half of the crushed and ground rock is too fine for granule production. This portion, as well as the dust collected in the Cottrell precipitators, is discarded.

Preliminary Investigations

The particle size of the product of the precipitators approached the size required for ceramic bodies. Although Fe_2O_3 constitutes 4 per cent of the quarried syenite, it is only 2.3 per cent in the Cottrell-separated dust. Crude, dry separation of magnetic particles by means of a bar magnet produced a nonmagnetic fraction which contained 1.1 per cent of Fe_2O_3.

A wet sieve analysis of the dust was run and the iron oxide content of each fraction was determined.

The head sample contained 2.68 per cent of Fe_2O_3 and the composite calculation showed 2.65 per cent of Fe_2O_3. Although there apparently was some separation of iron-bearing minerals from the quarried syenite in processing and precipitation, the concentration of these minerals apparently increases as the particle size of the dust decreases (Table 15).

The precipitated syenite dust was separated by means of a Roller Air Analyzer into two samples, one of which had a particle size less than 10 microns; the other had a particle size of 10 or more microns. Petrographic examinations of the original sample and the two size separations were made (Table 16).[1]

The mineralogical examination showed that these samples consisted essentially of orthoclase feldspar, nepheline, altered feldspar and nepheline, appreciable amounts of biotite, augite, aegerite, arfvedsonite, and small amounts of magnetite and fluorite. Some extraneous metallic iron particles were present also. Some iron-bearing minerals occurred as inclusions in the feldspar and nepheline in sizes down to 10 or 15 microns.

Wet Magnetic Separation of Cottrell-precipitated Syenite

A wet magnetic separation of precipitated syenite was made at Bauxite, Arkansas, with the cooperation of the U. S. Bureau of Mines

---

[1] Examination made by U. S. Bureau of Mines, Minerals Technology Division, Rolla, Missouri.

on an 8-inch Roche magnetic separator. This separator is essentially an inclined trough-type rubber conveyor belt under which the magnetic poles are placed. The direction of travel is upward. The magnetic particles cling to the belt and are removed at the upper end by a water spray. The nonmagnetic particles flow down the inclined belt to the other end of the separator. Water is added at the top and it helps to wash the nonmagnetic particles free of the magnetic ones.

The sample of precipitated syenite was mixed with water to form a pulp which contained 20 per cent solids. The pulp was treated at the rate of 160 pounds per hour, and the separated products were analyzed for iron oxide content (Table 17).

It has been suggested that roasting the sample of syenite dust would reduce the ferric iron to ferrous iron and make it more susceptible to magnetic separation. Thus, the Fe_2O_3 content might be reduced below 1.86 per cent. The economic feasibility of such an operation has not been determined.

High-tension Separation Tests

High-tension separation is an outgrowth of electrostatic separation, and the process makes use of a high rate of discharge, in addition to electron flow and gaseous ionization. A mineral mixture under this process receives a spray discharge of electricity, and some minerals gain a high surface charge and are pinned to a grounded rotor. The attraction of particles of one charge toward an electrode of the opposite charge is referred to as the lifting effect. High tension separation makes use of a strong pinning effect, and frequently combines a pinning effect with lifting effects.

The Carpco Engineering Corporation of Jacksonville, Florida, which has developed several types of high-tension separators, offered to make tests on the Arkansas nepheline syenite, but samples of the precipitated dust proved to be too fine, because the Carpco method requires that particles be between 20 mesh and 250 mesh in size.

Another syenite sample was prepared by screening the waste product of the roofing-granule plant. This sample had a particle-size range of 28 mesh to 40 mesh. Tests demonstrated that all of the minerals could be pinned to the roll readily, and that increasing the roll speed threw off all the material without any separation. No separation could be obtained in tests which employed an electrostatic field with both positive and negative polarities. A laboratory model of a high-intensity induced-roll magnetic separator also was used, but no worthwhile separation resulted.

Possibilities of Cyclone Separation of Nepheline Syenite

The cyclone was developed by the Dutch State Mines of Heerlen, The Netherlands, and was used originally in coal washing plants at Limburg. The operating principle is similar to that of a cyclone dust collector. In the Dutch process, however, a fluid pulp is pumped through the cone. A swirling action is imparted and centrifugal force acts on the mineral particles in proportion to their specific gravity. Dilute, light slurry comes out of the top of the cone and the thickened slurry and higher gravity solids discharge through an orifice in the bottom. Feed pressure, orifice openings, and feed rate are used to produce the degree of separation desired.

Correspondence with the Dorr Company resulted in the information that the use of the cyclone would produce only the separation obtained by straight classification, and that it could not produce a fraction which contained less than 1.32 per cent Fe_2O_3.

TABLE 15. WET SIEVE ANALYSIS AND IRON OXIDE CONTENT OF SEPARATED FRACTIONS OF COTTRELL-PRECIPITATED SYENITE

Sieve number (Tyler)	Fraction retained	Fraction through sieve	Fe_2O_3 content
	Per cent	*Per cent*	*Per cent*
100	1.97	—	1.32
150	1.65	—	1.45
200	4.17	—	1.79
325	10.47	—	2.18
325	—	80.47	2.82

TABLE 16. PETROGRAPHIC DATA, COTTRELL-SEPARATED NEPHELINE SYENITE

Syenite sample size	Feldspar and nepheline, free of iron-bearing minerals	Other transparent minerals, free of feldspar and nepheline[1]	Opaque minerals	Interlocked iron-bearing minerals, nepheline and feldspar
	Approximate volume	Approximate volume	Approximate volume	Approximate volume
	Per cent	*Per cent*	*Per cent*	*Per cent*
As received	77	19	2	2
Plus 10 microns	70	26	2	2
Minus 10 microns	71	27	2	0

[1] Includes transparent iron-bearing minerals and fluorite.

TABLE 17. WET MAGNETIC SEPARATION PERCENTAGES OF COTTRELL-PRECIPITATED NEPHELINE SYENITE, AND Fe_2O_3 CONTENT OF PRODUCTS

Syenite sample	Amount separated	Fe_2O_3 content[1]
	Weight, per cent	*Per cent*
As received	—	2.73
Nonmagnetic portion	93.0	1.86
Magnetic portion	7.0	14.3

[1] Analyses by T. W. Carney, chemist, Arkansas Resources and Development Commission.

Part Four

Ceramic Tests with Cottrell-Precipitated Arkansas Nepheline Syenite

Preliminary Investigation

The Fe_2O_3 content of the precipitated syenite dust and that which was subjected to wet magnetic separation was less than the iron oxide content of the quarried rock. Particle sizes of the dust also are suitable for many ceramic uses. It is possible that it may be beneficiated and the iron content further reduced.

The "as received" precipitate used in the ceramic tests contained 2.30 per cent iron oxide and the wet-separated product, 1.86 per cent.

Single-Fire Porcelain Enamel*

Standard compositions of a titania-opacified enamel which contained potash feldspar or precipitated syenite were fritted and then applied to sheet steel specimens. Preliminary tests showed that the Cottrell-fraction syenite produced a single-fire enamel equal to that produced by feldspar, except that it was slightly off color. The syenite enamel was gray-white; the feldspar enamel was white. The index of refraction of the syenite frit was increased to 1.512; the index of the feldspar frit was increased to 1.507.

Floor Tile

The following formula was used in preparing floor tile specimens:

Constituent	*Per cent*
Soda spar Potash spar } or syenite	31.0
China clay	19.0
Florida kaolin	14.0
Flint	5.0

Water was added to the materials as they were mixed in a small Lancaster mixer until a moisture content of 10 per cent was obtained. The damp material then was forced through a 14-mesh screen and shaped under pressure at 500 pounds per square inch into test bars 3 7/16 inches x 13/16 inch x 5/16 inch.

Bodies which contained feldspar were white after firing, and those which contained syenite ranged from tan to chocolate brown, the shade varying with firing temperature. The syenite bodies matured at lower temperatures than did the feldspar bodies, and they were stronger. The bodies which contained the beneficiated, precipitated syenite were slightly higher in strength, absorbed less water, and their shrinkage was less than that of the bodies which contained the untreated syenite dust.

---

* Tests by Theodore Dziemianowicz.

Sanitary Ware

The following formula was used to prepare a slip for casting test bars:

Constituent	*Per cent*
Soda spar Potash spar } or syenite	16.0
Kaolin	28.0
Ball clay	20.0
Flint	20.0

The deflocculant was 0.5 per cent Calgon. After water was added and the mix was blunged for 3 hours, the specific gravity was adjusted to 1.92 to 1.98. Test bars 1 inch x 1 inch x 6 inches and sag test bars 1 inch x 3 inches x 7 inches were cast in plaster molds, the surfaces of which had been dusted with talc to reduce sticking. Data on test bars were obtained after the bars had been fired at specific temperatures ranging from Cone 7 to Cone 11 (Table 18).

The sag bars were supported on 5-inch spans and fired to Cones 7, 8, 9, 10, and 11. Those bars which contained separated syenite sagged 60 per cent more than did those which contained feldspar.

Optimum values of strength, shrinkage, and absorption were shown for bars containing precipitated syenite and fired at Cone 8; tests on bars which contained feldspar failed to show optimum values at Cone 11. The undesirable buff color of bodies which contain syenite discourage their commercial use in sanitary ware.

Hotel China

Test samples of hotel china were prepared with the following:

Constituent	*Per cent*
Feldspar or precipitated syenite	18.0
Whiting	1.5
China clay	37.0
Ball clay	7.5
Flint	36.0

Bars 1 inch x 1 inch x 6 inches were cast in plaster molds. After the samples were fired in the range of Cones 8 to 11, the data showed that with equal shrinkage and water absorption, the syenite bodies were higher in strength than were those which contained feldspar. Their buff color might be desirable in the manufacture of stoneware, but not in the manufacture of hotel china.

Structural Clay Products

A clay which is used in the manufacture of face brick was employed as the raw material for this series of tests. Collected syenite dust to the extent of 3 and 5 per cent was added to the clay. The materials were mixed in a small Simpson mixer, and a 1 inch x 1 inch column was extruded from a laboratory deairing machine. Test bars 6 inches long were fired and tested (Table 19).

The addition of the syenite dust to the commercial brick clay improved the strength of the fired samples, increased shrinkage, lowered absorption, and enriched the color. The strength of the fired samples containing 5 per cent syenite was increased approximately 20 per cent. X-ray patterns of all fired samples were similar. It is possible that a very small amount of mullite was formed in the samples which contained syenite and which were fired to Cone 2.

The increase in strength produced by the syenite may be of interest to manufacturers of drain tile and similar shapes in which greater stresses may be developed.

Electrical Porcelain

The following composition was used:

Constituent	*Per cent*
Nepheline syenite	24.0
Ball clay (Victoria)	35.0
Kaolin (Layton)	18.0
Ottawa silica	21.0
Calcium carbonate	2.0

The components were mixed in a small Lancaster mixer and approximately 10 per cent water was added. The mix was pressed at 1,000 pounds per square inch to form discs 2 inches in diameter and ¼ inch thick. These were fired, and water absorption tests of the discs which had been fired to specific temperatures from Cone 7 to Cone 10 showed that the bodies which contained beneficiated syenite dust matured at the lowest temperatures. Those which contained syenite alone matured at the medium temperatures, and those which contained Lakefield syenite at the highest temperatures.

Dielectric constant measurements[1] of the fired samples showed values of 6.2 for bodies which contained precipitated syenites, 7.0 for bodies which contained Lakefield syenite, and 7.1 for bodies which contained beneficiated syenite dust. Further tests showed that samples which contained beneficiated syenite dust possessed dielectric strengths slightly higher than those of samples which contained Lakefield syenite, but 12 per cent higher than those of samples which contained precipitated syenite.

The color of the body which contained Lakefield syenite was white after firing, but the bodies which contained syenite dust or beneficiated syenite dust were gray. In the manufacture of electrical porcelain for low voltage applications, a ceramic stain is added to produce a gray color in the finished product, of a shade similar to the color of the samples which contained Arkansas syenite.

Glaze

In an attempt to utilize the fluxing ability and iron content of the precipitated syenite, tests were made to develop a brown glaze, similar to Albany slip glaze, for application to the electrical porcelain body which was being evaluated. An effective glaze was developed. It contained precipitated syenite, a brick clay which was red when fired, and Arkansas crude manganese ore, in a typical glaze composition.

Vitrified Grinding Body

Some 75 per cent of all grinding wheels used in industry are composed of alumina or silicon carbide grains held together with a glassy or porcelain-type bond.

Vitrified wheels vary in many respects:

A. *Type of grit.* The types of grit most often used are alumina (Al_2O_3) and silicon carbide (SiC). The former is employed where toughness of grain is important; the latter, when a friable grain is desired. Exolon RW alumina and Carbolon RW silicon carbide were used in this group of tests.

B. *Grain size.* Each type of grit is produced in sizes from Number 10 to Number 240. Number

---

[1] Tests made by Z. V. Harvalik, Institute of Science and Technology, University of Arkansas, with equipment of his own design.

60 was used in these experiments.

C. *Grade.* The grade denotes the tenacity with which the bond holds the grains. Hard grades are used when soft metals are ground, because the grains do not dull quickly and are held in place for maximum use. Conversely, soft grades are used with hard metals. The grade is varied by the addition of differing amounts of bond and by the use of different ingredients. Soft, medium, and hard grades, in which the bond content was 10, 20, and 30 per cent, respectively, were studied in these experiments.

D. *Density.* The combination of varying proportions of grain sizes of grit determines the density. The hardness of the material to be ground and the finish desired govern the density used on a given job. The use of Number 60 grit in the current experiments resulted in an "open" classification.

E. *Type of bond.* The bonding compositions used in industry normally are trade secrets. The type of bond which determines the elasticity and hardness of the grinding wheel is important to the user. The composition of the bond used in these experiments was:

Soda spar or beneficiated syenite dust	55%
Edgar plastic Florida kaolin	45%

After the dry materials had been mixed, sufficient water (which contained 0.25 per cent of gum arabic) was added and two types of test bars were cast. One type was used to test modulus of rupture, and the other to test tensile strength. After the test bars were fired in the range of Cone 7 to Cone 10, physical tests were made on the specimens (Tables 20 and 21).

The tests showed that the bodies which contained beneficiated syenite dust had a greater modulus of rupture and greater tensile strength, exhibited less shrinkage, and were harder than those which contained soda spar. The shrinkage upon drying was approximately the same for both.

TABLE 18. PROPERTIES OF FIRED SANITARY WARE COMPOSITIONS CONTAINING FELDSPAR OR COTTRELL-SEPARATED NEPHELINE SYENITE

Type of flux	Cone of firing	Firing shrinkage	Water absorption	Modulus of rupture
		Per cent	*Per cent*	*Lbs./sq. in.*
Feldspar	7	7.3	3.39	3,953
Separated syenite	7	10.0	1.55	3,888
Feldspar	8	8.7	1.62	3,705
Separated syenite	8	9.9	.79	4,630
Feldspar	11	9.0	1.05	4,123
Separated syenite	11	8.8	1.02	3,280

TABLE 19. PROPERTIES OF A FIRED BRICK CLAY CONTAINING PRECIPITATED SYENITE DUST

Amount of syenite added	Cone of firing	Firing shrinkage	Water absorption	Modulus of rupture
Per cent		*Per cent*	*Per cent*	*Lbs./sq. in.*
0	08-07	1.5	14.6	2,525
3	08-07	1.9	14.6	2,545
5	08-07	2.8	12.0	2,965
0	1[1]	3.4	10.8	2,560
3	1[1]	2.5	12.9	2,725
5	1[1]	3.7	9.1	3,070
0	2	3.4	11.7	2,725
3	2	4.0	9.4	2,810
5	2	5.3	8.4	3,295

TABLE 20. PROPERTIES, AFTER FIRING, OF MODULUS-OF-RUPTURE TEST BARS CONTAINING SILICON CARBIDE ABRASIVE GRAIN AND TEST BARS CONTAINING ALUMINA ABRASIVE GRAIN

Type of bond	Type of flux							
	Beneficiated syenite dust				Soda spar			
	Cone of firing	Porosity	Modulus of rupture	Rockwell hardness	Cone of firing	Porosity	Modulus of rupture	Rockwell hardness
	Number	*Per cent*	*Lb./sq. in.*	*Number*	*Number*	*Per cent*	*Lb./sq. in.*	*Number*
Test Bars Containing Silicon Carbide Abrasive Grain								
Soft	8	33.4	2,342	90	7	29.5	694	86
Medium	10	15.9	4,728	104	8	17.8	2,466	92
Hard	10	10.8	4,582	104	8	17.6	3,005	97
Test Bars Containing Alumina Abrasive Grain								
Soft	8	34.3	3,469	92	7	32.6	1,442	91
Medium	10	25.2	3,126	103	8	19.8	2,855	93
Hard	10	15.5	3,711	108	8	16.6	4,132	102

TABLE 21. PROPERTIES, AFTER FIRING, OF TENSILE-STRENGTH TEST BARS CONTAINING SILICON CARBIDE ABRASIVE GRAIN AND TEST BARS CONTAINING ALUMINA ABRASIVE GRAIN

Type of bond	Type of flux							
	Beneficiated syenite dust				Soda spar			
	Cone of firing	Porosity	Tensile strength	Rockwell hardness	Cone of firing	Porosity	Tensile strength	Rockwell hardness
	Number	*Per cent*	*Lb./sq. in.*	*Number*	*Number*	*Per cent*	*Lb./sq. in.*	*Number*
Test Bars Containing Silicon Carbide Abrasive Grain								
Soft	8	33.0	909	89	7	25.8	480	87
Medium	10	15.9	1,412	98	8	16.6	405	93
Hard	10	10.0	1,410	102	8	16.2	584	97
Test Bars Containing Alumina Abrasive Grain								
Soft	8	29.5	1,004	92	7	25.9	772	92
Medium	10	21.0	1,868	92	8	17.6	698	93
Hard	10	12.9	1,968	105	8	16.0	878	98

Part Five

Summary and Conclusions

Relatively large amounts of nepheline syenite have been found in Arkansas. Deposits are scattered but outcrops show them to cover approximately 15 square miles. Tests of material from a large exposure near Little Rock show that the nepheline syenite has a fusion point between that of potash feldspar and nepheline syenite found in Canada. The content of iron oxide is 4 to 5 per cent, which makes the Arkansas syenite unsuitable for certain ceramic applications. The Fe_2O_3 content was reduced to 1.1 per cent by means of magnetic separation of the oxide in a triple-rotor machine. A whiteware-type body was used to evaluate the syenite after its iron content had been reduced.

By means of a combination of flotation, tabling, and magnetic separation, the iron oxide content of a sample was reduced to 0.85 per cent. This beneficiated syenite was incorporated in compositions for the making of whiteware, glass, and porcelain enamel cover coat.

A finely divided, precipitated dust product from a commercial plant which makes roofing granules from nepheline syenite, was found to contain 2.3 per cent Fe_2O_3. Attempts to reduce the iron oxide content of this material by use of an electrostatic-type, high-tension machine were unsuccessful. By use of the wet magnetic separation process the Fe_2O_3 content was reduced to 1.86 per cent.

Syenite dust and beneficiated syenite dust were used in compositions for single-fire porcelain enamel, floor tile, sanitary ware, hotel china, structural clay products, electrical porcelain, electrical porcelain glaze, and grinding wheels.

No method of processing and beneficiating Arkansas nepheline syenite which produces a white ceramic composition after firing has been devised. However, desirable fusion characteristics and glassy phase of high strength make Arkansas syenite a desirable constituent in colored ceramic products. The Arkansas deposits apparently would have greatest application in structural clay products, low voltage electrical porcelain and glaze, and in grinding wheels.