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## Roofing granules

Morris Harold Grober

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ROOFING GRANULES

BY

MORRIS H. GROBER

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A

THESIS

submitted to the faculty of the  
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI  
in partial fulfillment of the work required for the

Degree of  
BACHELOR OF SCIENCE IN CERAMIC ENGINEERING

Rolla, Mo.

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Approved by .....

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## INTRODUCTION

An abundant amount of chats and clays of various kinds is available in Missouri. In the mining districts of south-eastern and south-western Missouri enormous piles of gang known as chats have accumulated. The mining companies desire to rid themselves of this accumulation and would dispose of the chats at a very low price.

In northern Missouri and in the diaspora district of central Missouri suitable clays could be found which may be had for a small royalty, mining and transportation costs.

Some of the requirements for roofing granules are that they have (1) a size limited by 10 and 30 mesh Tyler screens; (2) permanent and uniform color; (3) low reflectivity and low absorption; (4) resistance to freezing and thawing; (5) ability to be wetted and embed well in hot asphalt; (6) such a composition that it will not effloresce due to soluble salt content in moist climates and ; (7) especially it should have a low production cost.

#### PURPOSE OF INVESTIGATION

Since a definite demand for roofing granules exists and so much cheap material is available in Missouri, an investigation to determine the suitability of these materials and a method of production is proposed.

#### METHOD OF INVESTIGATION

Flint clay from the Forbes pit one mile north of Rolla on the old Vienna road was obtained.

Chat from south-western Missouri was obtained from the Bureau of Mines.

The flint clay and chat were screened so that the particles passed ten-mesh and were retained on thirty-mesh.

Soluble Salt Solutions

From the following soluble metal salts, ten-percent solutions were prepared.

Nickle Nitrate	$\text{NiNO}_3$	Cadmium Oxide	$\text{CdO}$
Nickel Carbonate	$\text{Ni}(\text{CO}_3)_3$	Strontium Carbonate	$\text{SrCO}_3$
Nickel Chloride	$\text{NiCl}_3$	Chromium Chloride	$\text{CrCl}_3$
Rutile	$\text{TiO}_2$	Sodium Chromate	$\text{Na}_2\text{CrO}_4$
Iron Nitrate	$\text{Fe}(\text{NO}_3)$	Potassium Chromate	$\text{K}_2\text{CrO}_4$
Iron Sulphate	$\text{Fe}_2(\text{SO}_4)_3$	Chromium Nitrate	$\text{Cr}(\text{NO}_3)_3$
Copper Nitrate	$\text{Cu}(\text{NO}_3)_2$	Chromous Chloride	$\text{CrCl}_2$
Copper Sulphate	$\text{CuSO}_4$	Chromium Oxide	$\text{Cr}_2\text{O}_3$
Copper Chloride	$\text{CuCl}_2$	Chromium Sulphate	$\text{CrSO}_4$
Uranium Oxide	$\text{UO}_2$	Manganese Dioxide	$\text{MnO}_2$
Cobalt Nitrate	$\text{Co}(\text{NO}_3)_3$	Manganese Chloride	$\text{MnCl}_2$
Iron Chloride	$\text{FeCl}_2$	Manganese Sulphate	$\text{MnSO}_4$
Potassium Dichromate	$\text{K}_2\text{Cr}_2\text{O}_7$	Manganese Carbonate	$\text{MnCO}_3$

These were applied to the clay and chat granules by soaking the granules in the various solutions. After soaking for a period of five minutes some of the granules were placed under a vacuum equal to a twenty-six inch column of mercury in order to thoroughly saturate them. After saturation they were placed and dried in a steam heated drier and fired to cone one in an oil-fired-muffle kiln. Half of those which were vacuum treated and half of the ordinarily treated granules were fired in the wet state.

The best colors resulting from the firing were selected and a one-percent solution of the soluble salts from which they were made was prepared. The clay and chat granules were soaked in these solutions for five minutes and fired wet to cone one in the oil-fired-muffle kiln.

Vitreous Slips

Vitreous slips were selected for the end members of a twenty-one member triaxial system as shown in figure 1.

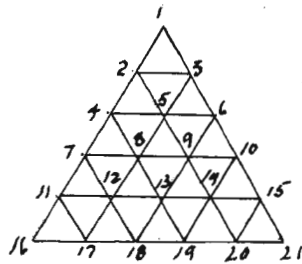


Figure 1.

The batch weight composition of the end members 1, 16, and 21 is presented in table I.

Table I

## Member 1

XX Baraby Sagger Clay	20%
Georgia Kaolin	30%
Canadian Feldspar	15%
Sodium Carbonate	15%
Borax	5%
Flint	15%



## Member 16

XX Baraby Sagger Clay	20%
Georgia Kaolin	30%
Canadian Feldspar	15%
Sodium Carbonate	10%
Borax	5%
Chromium Oxide	8%
Flint	12%

## Member 21

XX Baraby Sagger Clay	20%
Georgia Kaolin	30%
Canadian Feldspar	15%
Sodium Carbonate	12.5%
Borax	7.5%
Chromium Oxide	10%
Flint	5%

Batches of one-thousand grams of the vitreous slips listed in table I were weighed out and placed in ball mills with seven-hundred and fifty cubic centimeters of water and ground for a period of three hours. After grinding they were washed through a hundred-mesh Tyler screen and adjusted to 1.48 specific gravity. The slips were applied to the chat granules by dipping. They were dried in a steam heated drier, placed in a four inch crucible and then fired to nine-hundred degrees centigrade in an oil-fired pot furnace.

Member number one was the best vitreous slip resulting from the study designated in table I. This was used as a basis for a low maturing vitreous slip, which is presented in table II.

Table II

XX Baraby Sagger Clay	15%
Georgia Kaolin	25%
Canadian Feldspar	15%
Sodium Carbonate	15%
Borax	5%
Red Lead	10%
Potassium Dichromate	4%
Flint	11%

This slip was applied to the chat at 1.48 specific gravity and then allowed to dry in the steam heated drier. The chat was fired in four inch crucibles to nine-hundred degrees centigrade in an electric-muffle kiln.

Running water was poured on a portion of the hot granules taken from the hot furnace until steaming ceased, while another portion was allowed to cool to room temperature. Both batches were examined for spall action.

Absorption was determined by weighing dry and saturated. The saturated weight minus the dry weight divided by the dry weight, times one-hundred gave the percentage absorption.

That portion of the granules which was subjected to the water cooling remained in a flow of water for one week. This test showed whether or not the granules contained permanent color.

After the water treatment they were placed in an electric refrigerator and cooled to the point where ice was formed in and around the granules. The granules were then allowed to thaw in the sun and placed in the refrigerator again. This was repeated twenty times before the test on the granules was considered successful.



Fig. 2  
Untreated Flint Clay



Fig. 3  
Flint clay treated with solution (10%) Na<sub>2</sub>CrO<sub>4</sub>



Fig. 4

Flint clay treated with solution (1%)  $\text{FeCl}_2$



Fig. 5

Flint Clay treated with solution (1%)  $\text{CrCl}_3$



Fig. 6

Flint clay treated with solution (1%)  $\text{Co}(\text{NO}_3)_2$



Fig. 7

Flint clay treated with solution (1%)  $\text{NiCl}_2$



Fig. 8

Flint clay treated with solution (1%)  $\text{Fe}_2(\text{SO}_4)_3$



Fig. 9

Flint clay treated with solution (1%)  $\text{Cr}(\text{NO}_3)_3$





Fig. 10

Flint clay treated with solution (1%)  $\text{NiNO}_3$

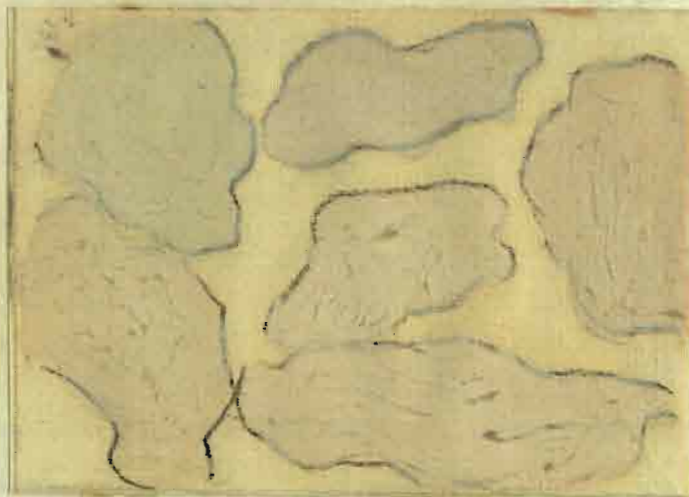


Fig. 11

Flint clay treated with solution (1%)  $(\text{NH}_4)_2\text{CrO}_4$





Fig. 12

Chats treated with solution (1%)  $K_2CrO_4$



Fig. 13

Chats treated with vitreous slip No. 11

## DISCUSSION OF RESULTS

Flint Clay Granules

The flint clay granules which were soaked in the ten-percent solutions of the various soluble salts gave good results. Some of the colors did not penetrate the granules because they were not highly soluble. The highly soluble salts penetrated the granules and made a very acceptable roofing granule.

The granules treated with one-percent solutions appeared to be the same as those treated with ten-percent solutions. The resulting colors are shown in figure four to figure twelve.

The granules which were placed in the muffle while wet fired to the same color as those on which the solution was dried before firing. This indicated that the color was not affected when the wet granules were placed in a hot muffle. See figures three to seven for wet fired granules and figures eight to twelve for the dry fired granules.

No soluble salts were present when the granules were subjected to a constant flow of water. There was no disappearance of color while the water ran over the granules.

The granules resisted freezing and thawing when put in the refrigerator.

They did not reflect light, although some had a glassy appearance.

None of the granules absorbed water when subjected to the absorption test. The color seemed to penetrate the pores of the clay particles, thus making them non-absorbent.

#### Chat Granules

The slip coated chat granules were not as well coated as the clay granules. They had a tendency to spall when taken from the hot furnace and dipped into cold water. At eight-hundred and seventy-five degrees centigrade the chert which comprises them inverts from alpha to beta quartz, causing the granules to break down into a fine powder.

Many colors were obtained and all of the well colored granules were put to the above tests. All of the clay granules survived the above tests and showed that they had the properties of an acceptable roofing granule.

The chats were not considered as having the possibilities of a roofing granule because of the negative results obtained with them.

## CONCLUSIONS

The foregoing investigation leads to conclusions as follows:

1. Flint clay makes a good roofing granule because it is porous and therefore will absorb solutions of soluble metallic salts which may be used as colorants.

2. The flint clay granules can be put in a hot furnace while wet, and still have the same color as when fired dry.

3. They can be taken from a hot furnace and no spall action takes place when they are put in cold water.

4. The rate of heating does not affect color.

5. The clay granules can be sufficiently colored by soaking in a solution without being subjected to a vacuum.

6. Ten-percent solutions of coloring oxide apparently give no better color effect than one-percent solutions on those granules investigated.

7. The flint clay granules have all the properties which are required of a roofing granule, and it should be cheap to produce.

8. The chat would make a suitable roofing granule if it could be permanently colored or completely colored with a vitreous slip at temperatures below eight-hundred and seventy-five degrees centigrade.

## RECOMMENDATIONS FOR FURTHER RESEARCH

For further study of roofing granules, it would be advisable to study more thoroughly the composition of a vitreous slip which will mature below 875°C.

A study should also be made of some agent which will cause the slip to adhere to the chat particles, thereby making a better coated particle.

Further investigation of this problem would aid the mine operators of Missouri in cleaning up and finding a use for the chat piles.

## APPLICATION TO THE INDUSTRY

This should be an ideal product to manufacture due to the low cost of production. The granules could be placed in a rotary kiln after they are soaked in a coloring solution, thus making the method of manufacture a continuous process. No time would be lost between the drying and firing stages of the process.

ACKNOWLEDGMENT

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