Selection of base rock for roofing granules production

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Abstract. Base rock selection is one of the major stages in the roofing granule process that determines profitability of the business and quality of the product. Detailed geological exploration, laboratory evaluations, and marketing considerations take place before the plant location receives an approval. A number of recommendations for choosing the proper base rock, based mainly on experience, have been developed by manufacturers over the last 50 years. The roofing granule industry developed the base rock tests to control quality of rock. However, the available information don’t conform to some significant aspects of the rock specification. Additional research has to be done prior to geologic investigation in order to reduce the number of samples to be analyzed, time and cost of the deposits exploration.

1. Base rock requirements

Roofing granules cover over 97% of the surface of a typical asphalt-soaked fiberglass shingle and roofing membrane. They are typically produced by mixing mineral particles with coating materials in a drum or pan coater prior to drying or curing of the color coating in a kiln with extremely high temperature. \(^1\) Granules are applied to roofing materials for several reasons, including UV protection, coloration, ballasting, impact resistance and fire resistance. Suitable base rocks include basalt, andesite, diabase, nepheline syenite and many more.\(^2\) The essential characteristics of base rock include: opacity to ultraviolet damage; chemical and physical inertness to provide resistance to acid rain and leaching, freeze/thaw and wet/dry cycling, oxidation and rusting; low porosity to improve physical strength, binding between coating and rock, and efficiency with which the pigment coating covers the surface; and tolerance of high firing temperatures.\(^3\) Other necessary characteristics include moderate hardness, to remain intact during the granule colouring process; moderate density, to weigh the shingle against wind lift; uniformity; and cubicle crushing to prevent directional embedment in the shingle manufacturing process, which changes shingle appearance.

Wrongly selected base rock does not provide protection from impact and UV degradation so the roofing material can collapse and stop performing as a protective material. Loss of granules reduces the roof durability, since it is associated with acceleration of photodegradation of the asphalt. In
addition, the aesthetics of the roofing system may be compromised if granules are lost.[4,5] The stains on the roof caused by the base rock rust are one of the reasons for most serious customer complaints. The rust can appear during the curing process or throughout shingle lifetime weathering.

2. **Recommended base rock chemistry**
Twenty-five or more years ago, the specifications for roofing granules were much looser than they are today. No single type or family of rock or minerals can be considered as a class to be a good and acceptable granule base, but rather a mineral deposit must be selected which meets rigid physical and chemical specifications. In recent years, these specifications have been so tightened by advanced technology and competition that only a few rocks can be processed acceptably and economically. The values below are based on rock selections that have been used successfully in producing high temperature roofing granules. [1]

Rock Selection

| % Ranges |
|----------|
| SiO2 45 - 72 |
| Al2O3 12 - 21 |
| Fe2O3 2.5 – 15 |
| MgO 0.25 - 6 |
| CaO 0.55 – 11 |

3. **Base rock industrial test methods**
The roofing granule industry has broken the base rock tests into three categories. The first and most important are “key” base rock tests. The key base rock tests are in no particular order: translucency, wet/dry disintegration, oil absorption, hardness test, rust test and particle shape index. It is very important for the base rock to pass all of the key base rock tests. The translucency test is a photographic method of measuring the actinic light transparency (translucency) of roofing granules. Test deck exposure studies have shown that the degree of actinic light transparency correlates with the extent of granule shedding expected during long term weathering. The wet/dry disintegration test was developed to examine base rocks so as to determine if there is the potential to break down when exposed as roofing granules on asphalt surfacing as a result of repeated wet and dry cycles. The oil absorption test was developed to indicate the relative surface porosity of a rock and/or natural granules and the coating porosity of unoiled coloured granules. It can provide some insight into the cost of coating the base rock with paint as compared to a known control sample. Typically the higher the oil absorption, the more paint is required to coat the granules resulting in higher overall production costs. The hardness test was developed to give an indication of the amount of potential granule breakdown during handling and processing. The rust test was developed to give an indication of the potential for roofing granules to exhibit rust spots when exposed to weathering. The natural granules are tested both uncalcined and calcined at 260°C and 537°C as calcining can often increase rust potential. The particle shape index test was developed to determine the shape of the roofing granules. Roofing granules should be round and cubical and not flat and elongated. Round and cubical granules reduce or eliminate shading effects on shingles whereas flat and elongated granules are more prone to exhibiting shading effects on shingles. Cubical granules give a higher granule loading on shingles than flat and elongated granules. These round out the key base rock tests.
The second group of base rock tests are considered “semi key” tests. These are certainly not as important as the key tests, but they are more important than supportive tests. The semi key tests include calcite, weight per cubic foot, asphalt adhesion, oil back off and grain size. The calcite test was developed to determine the amount of calcite or carbonate present in coloured roofing granules or base rock granules. The test determines the volume of CO2 produced by the reaction of the granules with 3 Molar Hydrochloric Acid. Calcite is an undesirable secondary sedimentary type mineral that may occur in seams and veins within a quarry. It is very different from the hard igneous rocks typically used for roofing granules. Calcite is soft with a Moh hardness of 3 compared to 5 to 6 for hard igneous rocks.

Calcite is affected by acid rain, has 100% translucency and can cause colouring problems if present in small amounts. The weight per cubic foot procedure was developed to be applicable to all types of roofing granules and other mineral surfacings and is intended to determine the weight per cubic foot of the materials as loose bulk density by the free fall method. The asphalt adhesion test was developed to measure the adhesion of granules to roofing asphalt. This test applies to natural granules and to oiled or unoiled coloured granules. Post treatments applied to granules may improve the asphalt to granule adhesion. The oil back off procedure was developed to determine the colour changes that occur to natural granules after wetting with water, after calcining at 260°C and 537°C and after oiling with process oil. Colours of natural granules can vary slightly or drastically from dry to wet, unoiled to oiled or uncalcined to calcined. Therefore, this test can be used to predict process oil requirements of natural granules and coloured granules. Fresh oiled natural and coloured granules will turn lighter after oiling as the oil is being absorbed into the rock. The grain size procedure was developed to make general visual observations of base rocks (7.6cm to 12.7 cm in diameter) before they are further processed into granules and to determine the general particle shape of individual granules as cubical, spherical, flat or elongated after crushing.

The last group of base rock tests are considered “support” tests. These tests are good to complete once a base rock has passed the key and semi key tests, but failing the support tests rarely fail a rock for roofing granules. These support tests include; specific gravity, magnet attraction, headlap rating, colour values, oiling rates, granule yield, discoulouration of white shingles, alkalinity titration of granules, relative surface weight of granules, reaction with 1 N HCl, percent loss on ignition, Moh hardness, PH, odour from cooling water, sieve analysis % retained. A lot of these tests help with the final product rather than determine if the rock passes as an approved base rock.

4. Discussion

During the geological survey conducted to locate a mineral deposit to be a source for the new granule plant it has been found that the existing rock specifications do not cover all aspects of base rock selection. The following discrepancies have been encountered in the process of rock quality assessment:

4.1. The declared list of the rocks that can be used as a base rock include basalt, andesite, diabase, nepheline syenite and many more.[2,6] However, experience shows that many of these rocks don’t meet the granule requirements. For instance, nepheline syenite is usually a transparent stone and gabbro often comprise olivine and quartz, which are also transparent minerals.

4.2. The general guideline on chemical composition poses more questions. It was observed that limiting content of MgO and CaO is controversial because the elements can be part of both alumosilicate and carbonate. More over, just 1% of Ca contained in carbonate could cause more problems than unlimited amount of the element in Pyroxene.
4.3. It has been experimentally determined that overall iron content is not directly associated with rust potential as it was claimed in the guidelines provided by the roofing granules industry.[4] When Fe is a part of oxides or silicates, its amount works for the rock opacity. Otherwise, it is terminate damage for the rock to have more than 0.5% Fe in sulfides.

4.4. The silica content also doesn’t reveal rock properties. Most critical is content of the pure mineral quartz which is transparent and such minerals as opal and chalcedony, which are not only transparent but are soluble in alkaline.

4.5. The current industrial methodology of rock assessment doesn’t include anything about the sulphur content. Recent studies show that sulphur content of more than 0.1% can cause critical rust appearance and deterioration of the rock.

5. Additional procedures
Research has been done by comparison of tests results. Three sets of rock samples were underwent standard roofing industrial tests. Upon comparing preliminary analyses and industrial test results, a data relationship pattern was found which has reduced the number of test samples for each subsequent round of tests. In additional research, data of chemical and petrography analysis of the rock samples were compared with color readings of the rocks before and after calcining process. Several rock samples were used in the granule process were selected for the test. The rock samples were preliminary calcined at operation temperature of 537°C and the color change readings were measured.

Colour measurement procedure was conducted using the LabScan XE Spectrophotometer with 0°/45° optical geometry that measures colour the way that you see colour, ignoring the glare, and including the effect of gloss or texture. Its large measurement port of 50 mm reduces the need to average and the variable illumination option has an automated zoom lens to measure samples as small as 5 mm.

Petrographic analysis was performed using CARL ZEISS JENA Amplival microscope and more detailed mineral analysis was conducted with electronic microscope Tescan VEGA-3 by scanning electron microscopy.

Bruker S4 Explorer has been used for the chemical analysis that has been done by x-ray fluorescence spectroscopy (RFS) method.

For this research we took rock samples from the deposits used for roofing granule production by one of the leading roofing material manufacturers – TechnoNICOL company as well as granule samples from the operating quarries of construction stone in the Ural region. As a control sample we took raw granules from the Havelock deposit in Canada that were used by 3M – the leading manufacturer of roofing granules for asphalt shingle industry.

Rock samples were selected directly from quarries. Specimens, most widespread throughout the deposits were picked.
Iron hydroxide, calcite and sulphur in rock microstructure

Uncalcined and calcined granules: a- Control sample; b- Sangalyk deposit; c- Gudronovskoe deposit.
6. Results
The study revealed that the test samples are extrusive igneous rocks formed by volcanic activity on the surface of the Earth. Several minerals that change their colour and collapse when heated at operational temperature were detected. Some of the minerals display rust, which is iron hydroxide.

Table 1. Tests data correlation

| Rock samples              | Petrography           | Minerals that cause rust when heated to 500 °C, % | Chemical Composition, % | Colour change | Bars test results |
|---------------------------|-----------------------|--------------------------------------------------|--------------------------|--------------|------------------|
|                           |                       | CHL | Grt | Palt | Akt | Si | Mgt | SiO₂ | FeO | CaO | MgO | S  | ΔE  | ΔL  |                       |
| Havelock (Canada)         | Dolerite Basalt       | 9   | 0   | 1    | 11  | 0.7 | 12  | 48.6 | 14.5 | 8.3 | 6.6 | 0.019 | 6.1 | -2 | 2            |
| Abražově                  | Andesite porphyrite   | 12  | 2   | 2    | 1   | 9   | 0.5 | 46.8 | 10.5 | 8.8 | 6.8 | 0.022 | 12.7 | 7.3 | 4            |
| Gudronovskoe              | Andesite              | 12  | 4   | 0.5  | 4   | 1   | 2.5 | 66.1 | 7.1  | 3.3 | 2.8 | 0.042 | 12.5 | 7.4 | 18           |
| Krutorogino               | Dolerite porphyrite   | 11  | 1   | 0    | 0   | 1   | 9   | 46.9 | 14.3 | 5.6 | 5.8 | 0.088 | 11.1 | 3.4 | 21           |
| Sangalyk                  | Greenstone            | 17  | 1   | 0.6  | 9   | 2   | 8   | 49.2 | 11.9 | 6.7 | 7.9 | 0.128 | 15   | 10.8 | 56           |

The mineral symbols: Chl - chlorite, Mgt – magnetite, Akt – actinolite, Gt – goethite, Palt – palagonite, Sf – sulphure

Table 2. Rock colour readings

| Rock samples | L   | a   | b   | ∆L  | ∆a  | ∆b  | ∆E  | Havelock | Abražově | Gudronovskoe | Krutorogino | Sangalyk |
|--------------|-----|-----|-----|-----|-----|-----|-----|----------|----------|-------------|-------------|----------|
|              |     |     |     |     |     |     |     | 42.46    | -1.47    | 2.64        | 40.46       | 1.25     |
|              |     |     |     |     |     |     |     | 44.8     | -0.2     | 1.25        | 39.52       | 3.02     |
|              |     |     |     |     |     |     |     | 46.8     | -0.5     | 4           | 39.39       | 3.47     |
|              |     |     |     |     |     |     |     | 48.8     | -2.82    | 2.72        | 39.34       | 10.61    |
|              |     |     |     |     |     |     |     | 49.76    | -2.19    | 2.94        | 38.97       | 2.69     |
|              |     |     |     |     |     |     |     | 42.46    | -1.47    | 2.64        | 40.46       | 1.25     |
|              |     |     |     |     |     |     |     | 44.8     | -0.2     | 1.25        | 39.52       | 3.02     |
|              |     |     |     |     |     |     |     | 46.8     | -0.5     | 4           | 39.39       | 3.47     |
|              |     |     |     |     |     |     |     | 48.8     | -2.82    | 2.72        | 39.34       | 10.61    |
|              |     |     |     |     |     |     |     | 49.76    | -2.19    | 2.94        | 38.97       | 2.69     |

As a result of the study we can make a statement that the general iron content doesn’t define the rust potential of rocks. It is necessary to know the state of iron in the minerals to determine the rust potential of the rock. The weakest minerals that collapse at operation temperature of the granule process and create the iron hydroxides that can freely leak out of rock body are sulfides.

Current national standards of the rock properties measurements for hardness, strength and rheology apply only to aggregates larger than roofing granules. There are no national standards for translucency or rust potential determination. To make a successful rock assessment in addition to the standard industrial tests a number of research such as petrography and chemical analysis has to be done prior to geologic investigation. As soon as opaque and weak minerals are identified visually or by using a petrography analysis, the most relevant problem of the local Ural deposits becomes the rust potential of rock. To simplify determining rust potential it is recommended to first measure the sulphur content.

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