



Evaluation of the best suitable purification method to purify Calcite micro powder and determine suitable method to enhance the CaCO₃ percentage

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ABSTRACT: Calcite is an important natural resource and also a stable form of the widely distributed mineral. Calcite mineral ores are very common in Rathnapura District in Sri Lanka, especially in Balangoda – Kalthota region.

Several types of physical and chemical methods were evaluated to find out the best method to purify the calcite micro powder. Electromagnetic separation method and Precipitated Calcium Carbonate method are identified as the best available methods to enhance the quality to a high level. But these processes are very costly at present.

Therefore the micro powder grinding process was examined and sampling was done with several rotating speed configurations such as 300rpm, 500rpm, 700rpm and 1000rpm. The ground samples were sieved using micro sieves of size 37, 53 and 100 Microns. Each of the sixteen samples was subjected to chemical and physical tests and it was found that the sample of size less than 37 Microns obtained from 500rpm rotation speed shows the lowest impurity percentage. Therefore, this physical separation method can be applied to local industries to enhance the quality of calcite raw mineral powder.

1.1 Introduction

Calcite is an important natural resource and also a stable form of the widely distributed mineral. It is a carbonate mineral and the primary mineral component of limestone and its metamorphic form, marble. It is very soluble in groundwater and is common in the shells of invertebrates like clams and oysters. A large percentage of calcite rocks has deposited in sedimentary environments of both chemical (limestone) and biochemical (chalk) origin. Calcite rocks consist of transported fragments from previously deposited, biogenic materials. A small amount of calcite is of igneous origin and is the primary component of the rare rock called Calcite.

When we consider about our country this calcite mineral is widely distributed in many parts of the areas, mainly in Balangoda-Kalthota could consist of many deposits with lower impurities, which are considered as calcite vein deposit. These calcite mineral fragments are ground in to micro size by micro powder grinder and supplied to many industries. These have of many types of impurities in various percentages in each ore so that final

product quality, durability and strength are decreased then the price is also decreased. If we can enhance the CaCO₃ in Calcite powder, then we can easily enhance the above qualities and the value addition process steps will also succeed. Previously a large percentage of this raw material was imported to our country but now it has reduced much because micro powder calcite is available in our country. There are many impurities in this micro powder such as Mg²⁺ ions as Dolomite particles and Fe²⁺, Fe³⁺, silicate minerals and small quantities of many other type of minerals.

Even though this calcite mineral is separated manually in to grades as 1st grade, 2nd grade, and 3rd grade, this 1st grade minerals also consist of considerable amount of impurities. These impurities are a big problem for many industries. If there is any method to enhance the purity of CaCO₃ up to 100%, this can be supplied as available raw material for pharmaceutical industry and also for many other industries.

Under these circumstances, it is of utmost importance to identify the all impurities and



identification of suitable method for purification of CaCO_3 .

2. Study area

Calcite vein deposits are occurred from south-East to north-west direction in Kalthota area.

This valuable mineral is mined by many people and transported to calcite processing factories such as Wall tile factory-Balangoda, Calcite factory-Avissavella and Inline Calcite factory-Balangoda. Comparatively calcite is good economic mineral so that we have to do many researches to purify, separate, precipitate and enhance of CaCO_3 . Inmine calcite (Pvt) Ltd is one of the leading companies regarding calcite mining & processing of Micro powder, calcite powder, and super filler. They possess the best of processing units and systems, which has enabled them to become a forerunner in this sector. Impeccable quality standards, yearning for continuous growth and the spirit of optimum productivity have led to their enormous success, inculcating within a one and half of year pride. The company has entered into the market in a well-planned manner to leverage its sale with best of the processes.

3. Industrial Usage of Calcite

Calcite Powder is a natural form of Calcium Carbonate with extremely high whiteness, purity and free flowing in nature. White Calcite Powder is widely used in plastics as filler, due to its high dispersibility, low oil absorption, high impact resistance, smooth surface finish, easy processing, compatibility with polymers and other organic additives and excellent dimensional stability.

➤ Applications;

- Oil Exploration
- Drilling industry
- Plastics
- Rigid PVC Pipes
- LDPE Master Batches
- Paints, Inks, Powder Coating and Ceramic
- Welding Rods
- Automotive and Furniture Compounds.
- Rubber Industry
- Leather Cloth Industry

As a flux in Glass Industry

4. Mineral Separation methods associated with Calcite

All the mineral separation methods are based on chemical or physical property of each mineral. If we have physical method to separate Calcite, it is very suitable for the industrial process because it is cost effective, not spend much time for separation. When we consider about chemical separation method it is not suitable to industrial application because it is not cost effective and time effective. For the purpose of removing dolomite or separating Calcite by physical method I carefully observed each physical property of each mineral.

Magnetic property

Both Dolomite and Calcite mineral are not magnetic minerals so that we cannot use this property for separation

Hardness or density

Hardness of each mineral is also nearly same in each other

Dolomite	2.84
Calcite	2.71

Electro static behavior

These minerals are charged in different voltages

Dolomite	8268 v
Calcite	10920 v

There are many types of methods to separate minerals, and those are based on some physical or chemical factor of mineral

- I. Hydro cyclones -based on density of mineral
- II. Electrostatic separation -based on charging voltage of mineral
- III. Gravity separation -based on density of mineral
- IV. Density separation -based on density of mineral
- V. Magnetic separation -based on magnetic property of mineral



According to above information Electrostatic separation method can be used to separate and purify Calcite mineral in effective and efficient manner.

4.1 Calcite separation by Electrostatic separation method

Electro static separation is defined as "the selective sorting of solid species by means of utilizing forces acting on charged or polarized bodies in an electric field. Separation is effected by adjusting the electric and coating forces, such as gravity or centrifugal force, and the different trajectories at some predetermined time.

Electrostatic separation is important in the production of minerals, also in the reclamation of other valuable materials.

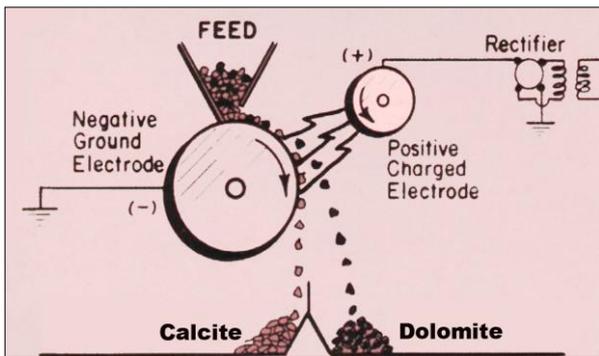


Fig 4.1 Typical diagram of a electrostatic separation of Calcite and Dolomite

The particle is charged by the electric field and attracted towards electrode .Then separate minerals in to two fragments. A very simple demonstration of electrostatic separation can be made by taking a handful of salted peanuts, rubbing the skins off, then taking a comb, rubbing it on fur or the coat sleeve until a static charge has been collected on the comb, and passing it over the peanuts and skins. This can be controlled to a great extent by potential, polarity, temperature, and conditioning of the surface of the particles.

Minerals and non mineral materials are separated electro statically through the difference in degree of electrical conductivity of the particles, however many minerals, metallic and non-metallic, and

other materials, accumulate a surface frictional charge through rubbing together in transit conveying or through coming into contact with various metals and other surfaces while in motion. This is well demonstrated when dry screening many powders.

These Calcite and Dolomite minerals are charged in different voltages as follows

Dolomite	8268 v
Calcite	10920 v

By doing chemical analysis I could determine the sample with highest percentage of CaCO₃, or sample with lowest percentage of dolomite. So that this sample can be enter as the row material for this separation process.

According to that dolomite powder firstly charged when the voltages reach up to 9000 v but calcite doesn't charge in that voltages hence Dolomite powder easily stitch or attach to the electrode (according to above diagram) so that calcite mineral powder can be easily separated.

4.2 Chemical Precipitation Method

Lime stone is screened to remove undersize & washed in rotary screen by using water to remove impurities like dust, dirt, mud etc. These impurities are separated out in a settler and clear water is reused for washing.

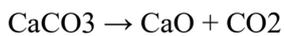
The washed Lime Kiln is then charged to the vertical lime kiln from top through the winch arrangement. In this zone, lime stone is preheated up to a certain temperature by using heat from hot exhaust gases (CO₂) of lime kiln.

Lime stone is then entered in to a calcining / reaction zone of lime kiln. Here, the temperature is controlled near about 1100-1200°C by burning the fuel (Natural gas/fuel oil) & combustion air mixture in a controlled ratio. The total air requirement is fulfilled by two stage air feeding system, primary air for fuel burning in burner and secondary air for recirculation inside chamber.

To gain a better efficiency of lime kiln, the re-circulation air is preheated up to a certain

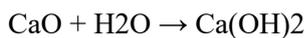
temperature in a heat recovery system. Here secondary air is heated up by the heat of hot air, obtained during cooling of burnt/quick lime from lime kiln bottom. The cooled quick lime obtained from lime kiln is then transferred to storage area.

When the Limestone is burned produce the desired quality to quick lime & CO₂ gas.



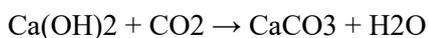
Quick lime of desired lump size is cooled and then transferred in to a hammer mill for crushing.

After crushing, quicklime is hydrated in the slacker/ reactor by adding a controlled quantity of water to yield the hydrated lime with desired purity. Hydration of quick lime to give hydrated lime.



In the burning process CO₂ is generated, it is valuable product for the precipitation step therefor CO₂ is cooled and washed to remove unwanted gases.

Then the produced CaO further react with the CO₂ then produce Calcite solid particles. In this step the required conditions are maintained in reactor to obtain different grade of precipitated calcium carbonate. These solid particles are further washed, filtered and dried to get fine powder of precipitated calcium carbonate.



5. Methodology

5.1 Mineral grading and collection of samples

Calcite minerals are graded in to three groups such as 1st grade, 2nd grade and 3rd grade and Dolomite. 1st grade calcite contain highest percentage of CaCO₃ and the 3rd grade Calcite contain lowest percentage of CaCO₃. The 1st grade calcite was selected for samplings.

Sampling was done with several rotating speed configurations such as 300rpm, 500rpm, 700rpm

and 1000rpm. The ground samples were sieved using micro sieves of size 37, 53 and 100 Microns.

Each of the sixteen samples (Samples were numbered as shown in figure 4.1) were subjected to chemical and physical tests.



Figure 5.1 Samples prepared by several rotation speeds (300, 500, 700 and 1000 rpm) and sieved using sieves of size 37, 53 and 100 microns

5.2 Determination bulk density

Bulk densities were determined as per the BS test method in each sample (300 rpm, 500rpm, 700rpm, 1000rpm).

5.3 Optical identification of impurities

Representative calcite sample was observed through the polarizing microscope..

Representative sample was placed on a slide then suitable objective was selected (4x, 10x, 20x, 40x) by rotating of each strain-free objects. Each type of mineral and impurities were identified, and then rough percentages of each mineral were determined.

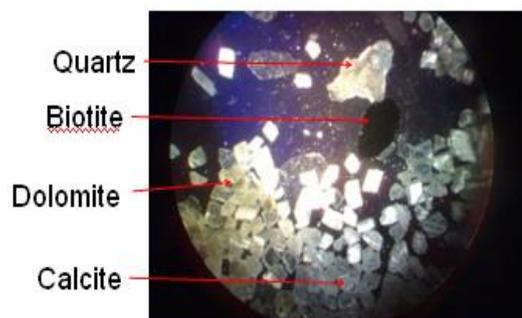


Figure 5.2 Observation through the polarizing microscope using 40 x objects

5.4 Particle Analyses

Particle analysis test was done by particle analyser for the each samples collected from several

rotating speed configurations such as 300rpm, 500rpm, 700rpm and 1000rpm.

5.5 Chemical Tests

Ca²⁺ and Mg²⁺ ions were determined by EDTA analysis method, Fe²⁺ and Fe³⁺ ion were analysed by Colorimetric method.

6. Conclusion and recommendations

The prepared samples were subjected to both chemical and physical tests and the following observations were made.

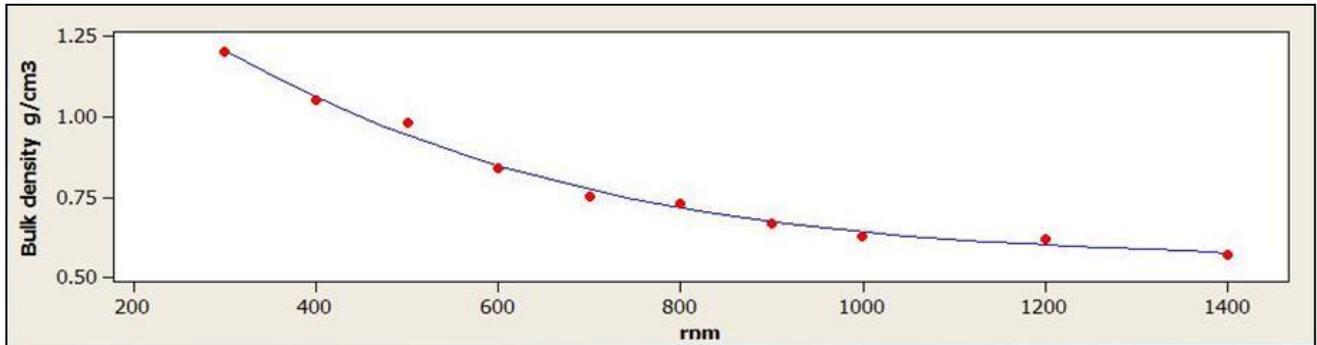


Figure 6.1 Variation of bulk densities of micro powder with respect to the rotation speed of grinder

The Bulk density was decreased gradually with increasing of rotation speed of grinder. The results obtained from the particle analyses test are shows four different graphs with respect to the each samples collected from rotating speed

configurations such as 300rpm, 500rpm, 700rpm and 1000rpm.

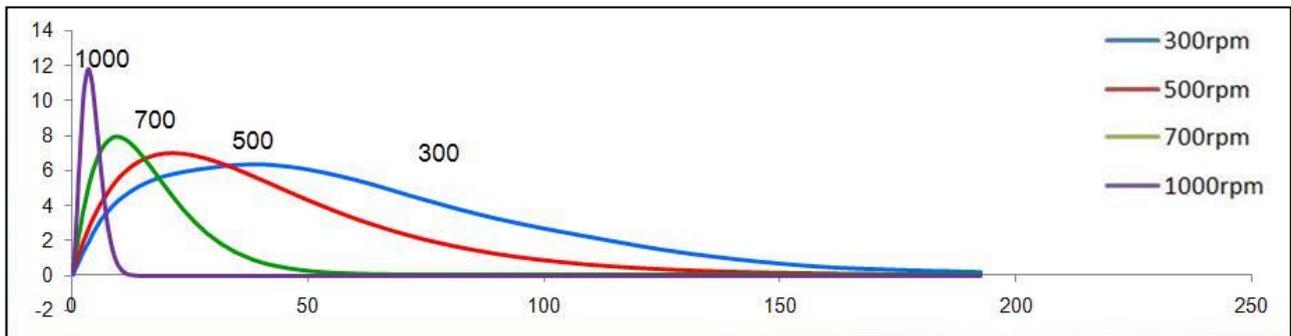
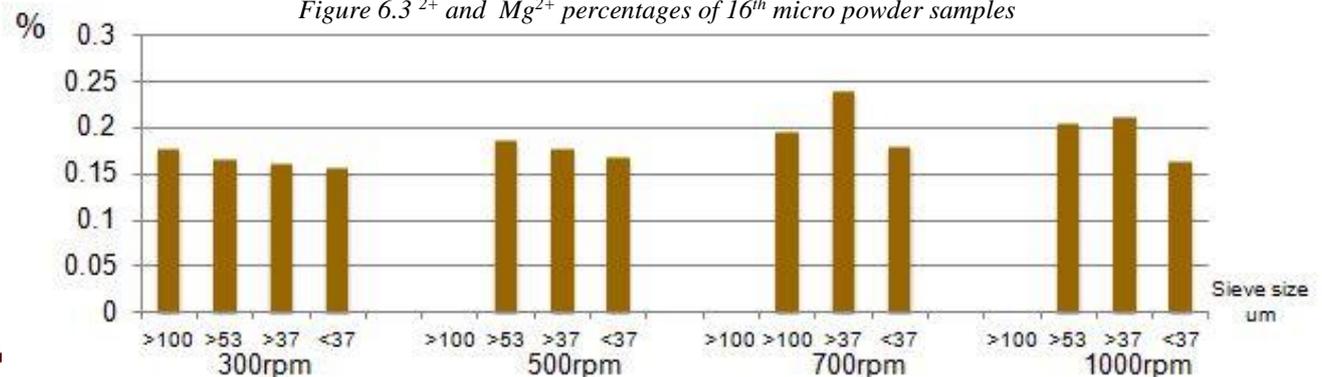


Figure 6.2 Particle size distribution of micro powder with respect to the rotation speed of grinder

With increasing of rotation speed of grinder, majority of mineral particles become in to finest level (very poorly graded), and with decreasing of

rotation speed the particle shows a well graded condition and the particles were distributed from Zero to 200 micron range.

Figure 6.3 Ca²⁺ and Mg²⁺ percentages of 16th micro powder samples



Measured Total Fe²⁺ & Fe³⁺ ion percentages were recorded less than 0.2% in all samples so that it's within the allowable limits.

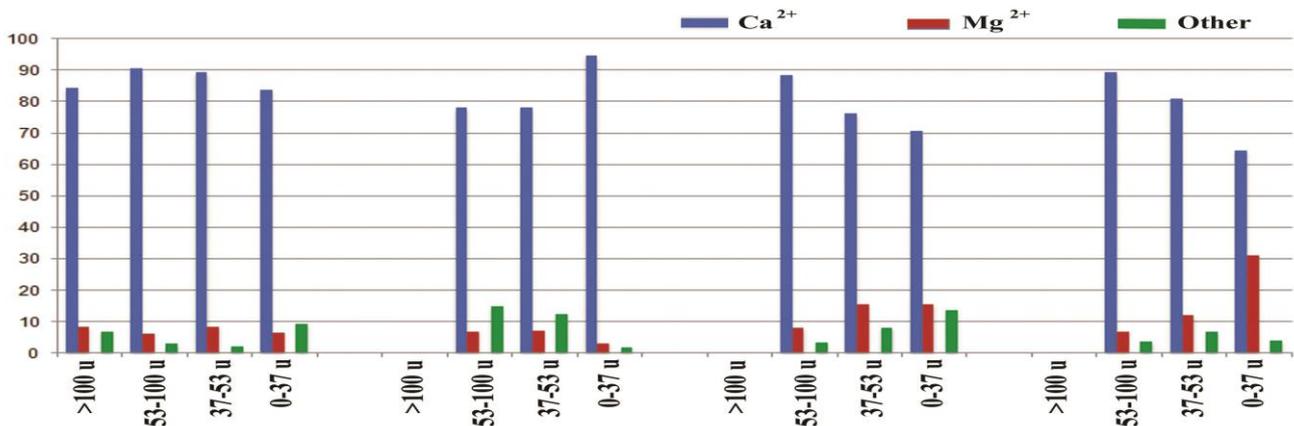


Figure 6.4 Fe²⁺ and Fe³⁺ percentages of 16th micro powder samples

The samples with highest percentage of Ca²⁺ and lowest percentage of Mg²⁺ were recorded in Samples of Sample 8, Sample 10 and Sample 14.

The Sample 10 and Sample 14 were prepared using the retained portions of 53 micron sieves with respect to the 700rpm and 1000rpm rotation speeds. Considering the particle size distribution graphs and percentages of retained, these samples represent less than 10% out of sieved samples. Hence, the separation process of sample 10 and sample 14 are not suitable to apply in commercial way.

Considering the percentage of retained and the particle analyses graphs the Sample 8 (53-100 micron) and associated separation steps can be used to enhance and grade the calcite micro powder in a commercial way.

micron fraction in 1000rpm, 53-100 micron fraction in 700rpm and Zero to 37 micron fraction in 500rpm samples. Out of them grinder speed and less than 37 sieved sample was shows the most suitable one for the next separation process. In addition to that particle variation data shows that about 80% particles are less than 37 µm diameter size. So that it is very feasible for the process.

References

April, L.U., Richard, L.D., (2008) Methods of soil analysis & Mineralogical Methods, 2nd edition, 511P.

Roy, E.H., (2005) Geotechnical investigation Methods & field guide for geochemical Engineers, Taylor & Francis group Publishers, 331P.

Andrew dutnis. (2008) .Introduction to mineral science, pp 296-388.

April, L.U., Richard, L.D. (2008) Methods of soil analysis & Mineralogical Methods. 2nd edition, 511P.

Bakshi, S.P. (2003). Engineering chemistry, pp 79-129.

Bhatia. S. C, Engineering chemistry, PP-540-565.

Dexter Perkins, Mineralogy (Second edition), pp 123-128.

Ford, W.E., Dana's. (1999).Text book of mineralogy. (Fourth edition), pp 498-499.

Gray D. Christian, Analytical chemistry. 6th edition, PP-294-304.

Jain, P.C. (2002).Engineering chemistry, pp 200-272,327-348.

John sinkankas, Mineralogy, pp 52, 369.

Mendham. J and Barnes J. D.Vogel's Text book of quantitative chemical analysis. 6th edition, pp-371-406.

Srivastava, M.M., Shalini srivastava.Recent trend in Chemistry, pp 369-372.