Surface Area of Calcium Carbonate

AN246

Calcium carbonate is a white crystalline chemical compound with the chemical formula $CaCO_3$. It is commonly found in nature in various forms, such as limestone, chalk, marble, and calcite.

Introduction

Calcium carbonate is one of the most abundant minerals in the Earth's crust and can be found in large deposits all over the world. It's often formed through the accumulation of shells, coral, and other marine organisms over millions of years. Many limestone and chalk deposits are the result of sedimentary processes. The accumulation of calcium-rich materials in aquatic environments can lead to the formation of calcium carbonate-rich rocks.

Calcium carbonate is used in construction materials. Limestone and marble, which are forms of calcium carbonate, are used as building stones and in the production of cement, concrete, and mortar. It is used as a filler and extender in a wide range of products, including plastics, paints, coatings, and rubber. It helps improve the physical properties and reduces costs by replacing more expensive materials. In the paper industry, calcium carbonate is used as a filler and coating pigment. It enhances the brightness, opacity, and smoothness of paper products. It is used in dietary supplements as a source of supplemental calcium, which is important for maintaining healthy bones and teeth. It's also used as an antacid to relieve symptoms of acid indigestion, heartburn, and upset stomach.

Other uses include the food and beverage industry, environmental uses, agricultural, personal care and animal feed applications.

The surface area of calcium carbonate plays a crucial role in determining its reactivity, effectiveness, and performance in various applications. In the case of calcium carbonate, a larger surface area means more opportunities for reactions with other substances, such as acids or other chemicals. For example, calcium carbonate's role as an antacid is based on its ability to react with stomach acids.

In dietary supplements and pharmaceuticals, a larger surface area provides more contact points to interact with digestive enzymes and be absorbed by the body.

Figure 1. Example of calcium carbonate powder.

Understanding and controlling the surface area can lead to optimized formulations and improved outcomes in industries ranging from pharmaceuticals to construction.

Gas Adsorption Technique

Surface area in this case is measured by a technique known as gas adsorption or physical adsorption (physisorption). Surface area is typically reported as meters squared per gram.

Adsorption takes place on the surface of a material. Gas molecules of known cross sectional area are carefully inventoried as they adhere to the surface. By knowing the number of moles of gas adsorbed and their cross section, the total surface area under test can easily be calculated.

The adsorption process occurs due to surface energy typically described as Van der Waals forces. Left alone, most materials will adsorb water or other vapors to satisfy this surface energy. Thus, to take advantage of these surface forces for measurement, the adsorbed impurities must be removed. This is accomplished through a process known as degassing, whereby a sample is placed in a holder and an inert flow of gas passes through the sample powdered bed as it is gently heated. The applied heat causes the adsorbed impurities to break free of the surface of the material and the flow of gas sweeps them away.

Cleaned in such a way, the sample holder may now be placed on an analysis station where it is cooled (typically to liquid nitrogen temperatures) in a cryogen Dewar as a gas mixture (typically nitrogen in a carrier gas of helium) flows across the surface. As the sample and gas cool, nitrogen molecules in the flowing gas mixture lose energy and are adsorbed onto the sample surface.

Again, monitoring the number of moles of gas adsorbed as a function of gas concentration allows us to calculate several adsorption data points. For more rapid analysis, a single point may be collected. The most common calculation method applied to derive specific surface area is the Brunauer, Emmett and Teller (BET) method.

Experimental

Two calcium carbonate powders were sent to the HORIBA Instruments Incorporated applications lab in Irvine, California for analysis. The samples were analyzed using the new SA-9650 surface area analyzer. Three separate aliquots of each sample each weighing approximately 0.5 g were placed in sample tubes. For each material, degassing was performed on the 3 integrated degas stations of the SA-9650 at 300°C for 2 hours. Samples were then transferred to the 3 analysis stations and separate analyses were performed for single point and multi point surface area. Each type of test was repeated 3 times. The results are shown in the tables below.

Calcium Carbonate 1 (Single Point*)

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	4.13	4.33	4.16	4.21	2.09
Test 2	3.96	4.23	3.99	4.06	2.98
Test 3	3.86	4.06	3.92	3.95	2.12
Average of 3 Tests	4.0	4.2	4.0		
CoV (%)	2.80	2.65	2.50		

Calcium Carbonate 1 (Multi-Point*)

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	3.94	4.08	3.94	3.99	1.66
Test 2	3.93	4.07	3.89	3.96	1.95
Test 3	3.95	4.06	3.85	3.95	2.17
Average of 3 Tests	3.9	4.1	3.9		
CoV (%)	0.21	0.20	0.95		

Calcium Carbonate 2 (Single Point*)

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	2.28	2.47	2.13	2.29	6.07
Test 2	2.31	2.36	2.2	2.29	2.92
Test 3	2.21	2.3	2.12	2.21	3.33
Average of 3 Tests	2.3	2.4	2.2		
CoV (%)	1.85	2.96	1.66		

Calcium Carbonate 2 (Multi-Point*)

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	2.32	2.44	2.26	2.34	3.20
Test 2	2.32	2.4	2.26	2.33	2.46
Test 3	2.27	2.4	2.25	2.31	2.88
Average of 3 Tests	2.3	2.4	2.3		
CoV (%)	1.02	0.78	0.21		

*The difference between single point and multi point analyses are linked to assumptions made in the single point calculation. Multi-point results are typically more accurate. However, in terms of repeatability and reproducibility, either measurement is quite robust and single point measurements are extremely fast – supporting high throughput, production, or quality control environments.

Conclusion

The HORIBA SA-9650 surface area analyzer proved to be an ideal instrument for measuring the surface area of calcium carbonate, both by single and multi-point analysis. The analyses were fast, repeatable and the instrument is robust. The methodology described in this document should be useful as a guide to customers using the SA-9650 for calcium carbonate or other powdered samples.