Application Note

Surface Area of Hydroxyapatite

AN245

Hydroxyapatite is a naturally occurring mineral form of calcium apatite with the chemical formula $Ca_5(PO_4)_3(OH)$. It is the main mineral component of vertebrate bones and teeth and is also found in some other tissues like dentin and enamel.

Introduction

The surface area of hydroxyapatite can vary depending on factors such as particle size, morphology, and preparation methods. Generally, hydroxyapatite particles have a relatively high surface area, ranging from around 20 to 150 m^2/g or even higher, depending on the synthesis method, due to their porous and crystalline structure. This surface area can significantly impact the material's reactivity, adsorption capacity, and overall performance.

Hydroxyapatite's unique properties make it useful in various fields and applications:

1. Biomedical Applications:

- Dental Implants and Fillings: Hydroxyapatite is widely used in dentistry for dental implants, fillings, and coatings on dental devices.
- Bone Grafts: In orthopedic surgery, hydroxyapatite is used as a bone substitute for grafting procedures as it provides a scaffold for new bone growth, promoting bone regeneration and integration.
- Coatings for Implants: Hydroxyapatite coatings on medical implants, such as joint replacements and screws, enhance their biocompatibility and facilitate bone attachment, reducing the risk of implant rejection.

2. Agricultural and Environmental Applications:

- Soil Remediation: Hydroxyapatite can be used to immobilize heavy metals and radionuclides in contaminated soil, reducing their mobility and environmental impact.
- Fertilizer Additive: It can be used as a slow-release source of phosphorus in fertilizers, promoting healthy plant growth and minimizing nutrient runoff.

Figure 1. Example of hydroxyapatite powder.

3. Other uses

- Catalysis
- Drug delivery
- Biomineralization processes
- Coatings and Films
- Cosmetics
- Research and material science

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

A commercially available hydroxyapatite powder sample was sent to the HORIBA Instruments Incorporated applications lab in Irvine, California for analysis. The sample was analyzed using the new SA-9650 surface area analyzer. Three separate aliquots each weighing approximately 0.11g were placed in sample tubes. Degassing was performed on the 3 integrated degas stations of the SA-9650 at 250°C for 3 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 following tables.

Single Point*

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	55.89	56.92	55.58	56.13	1.02
Test 2	55.62	56.57	56.12	56.10	0.69
Test 3	56.16	57.33	56.34	56.61	0.91
Average of 3 Tests	55.9	56.9	56.0	**All results in m^2/g**	
CoV (%)	0.39	0.55	0.57		

Multi-Point*

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	56.53	57.85	56.91	57.10	0.97
Test 2	56.52	57.92	56.71	57.05	1.09
Test 3	55.91	57.49	56.09	56.50	1.25
Average of 3 Tests	56.3	57.8	56.6	**All results in m^2/g**	
CoV (%)	0.51	0.33	0.62		

*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 hydroxyapatite 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 using the SA-9650 for hydroxyapatite or other powdered samples.

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