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Powder rheology of natural and synthetic graphite for optimizing anode slurry production in lithium-ion batteries

Authors
Fabian Meyer,
Thermo Fisher Scientific,
Karlsruhe, Germany

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Introduction

Lithium-ion batteries are vital for a wide range of applications, from portable electronics to electric vehicles and beyond, due to their high energy density, long cycle life, and efficiency.[1] The global production of lithium-ion batteries has seen substantial growth, with the market size estimated at USD 54.4 billion in 2023 and a projected compound annual growth rate (CAGR) of 20.3% from 2024 to 2030, driven by rising demand for electric vehicles and renewable energy storage[2]. Graphite powder is essential in the production of anode slurries for lithium-ion batteries due to graphite's excellent electrical conductivity, stability, and ability to intercalate lithium ions.[3] These properties make graphite an ideal choice for the anode material, which plays a pivotal role in the battery's charging and discharging processes. There are two primary types of graphite used in anode manufacturing: natural and synthetic graphite. Natural graphite is mined and typically has a flake-like structure, whereas synthetic graphite is produced through high-temperature processing of carbon materials, resulting in a more uniform structure. Synthetic graphite is used more frequently due to its higher purity, consistency, and structural integrity, all of which enhance battery performance. However, natural graphite is less expensive, making it an attractive option for cost-sensitive applications.[3]

Mixtures of both natural and synthetic graphite are sometimes used to balance cost and optimize anode characteristics. Electrode slurries can be produced through batch mixing, where precise amounts of graphite powder, binder, solvent, and additives are combined in a mixing vessel, or through continuous mixing in an extrusion process, which offers efficiency and consistent output for large-scale production. The rheological properties of graphite powder, including flowability, compressibility, and shear strength, are critical in both methods. Good flowability ensures easy handling and consistent mixing of powder. The compressibility of graphite powders allows conclusions to be drawn about their behavior during the calendaring process,[4] while shear strength measurements provide insights into storage behavior. Optimizing these rheological properties is crucial for producing high-quality anode slurries that enhance the performance and reliability of lithium-ion batteries. Understanding the differences in rheological behavior between natural and synthetic graphite, or of their mixtures, can provide insights into their suitability for various manufacturing processes and ultimately impact the efficiency and quality of the final battery product.

This report presents a comprehensive study of the powder rheology of natural and synthetic graphite powders, utilizing a rotational rheometer equipped with an accessory specifically designed for powder rheological investigations.

Materials and methods

Two types of graphite powder were tested for this study, one of natural origin and one of synthetic origin. Both samples are commercially available products.

All powder rheological tests were performed with a Thermo Scientific™ HAAKE™ MARS™ iQ Rheometer equipped with the powder rheology measuring geometry as shown in Figure 1. A more detailed description of this setup can be found in Reference [5].

Figure 1. HAAKE MARS iQ Air Rheometer with accessory for powder rheology.

The following tests were conducted on both samples:

- Powder compression testing: This test evaluates the compressibility and density of the graphite powders. In this test the sample is loaded into a cup and an initial conditioning cycle with a tip speed of 40 mm/s is performed using the powder vane rotor to remove all loading-related stresses and to ensure that the sample is homogenously filling the cup. After that, a sample split is performed by sliding the loading funnel sideways onto a reservoir to remove excess and to obtain a defined sample volume of 13.3 ml in the powder cup. After this, the rotor is changed to a porous piston. Stepwise increasing axial stresses of 0.5, 1.0, 2.0, 4.0, 6.0, 8.0, 10.0, 12.0 and 15.0 kPa are then applied to the powder, while its compression is measured by observing the change in sample height. These changes in applied stress lead to density changes at the different compression levels.
- Powder flow testing: This test assesses the flowability of the graphite powders, which is crucial for the mixing and handling processes in the production of battery electrode slurries. With this test, parameters such as basic flowability energy and flow stability are determined.
- Powder shear test: This test measures the shear strength and cohesiveness of the graphite powders, which influence its flow behavior under axial load. The test involves applying a shear force to the powder and measuring its resistance to flow under different stress levels.

A more detailed description of the powder flow and the powder shear test can be found in References [6] and [7] respectively.

All test procedures are available as predefined methods in the Thermo Scientific™ HAAKE™ RheoWin™ Software.

Results and discussion

Figure 2 shows the results of the compression test with the two different graphite powders.

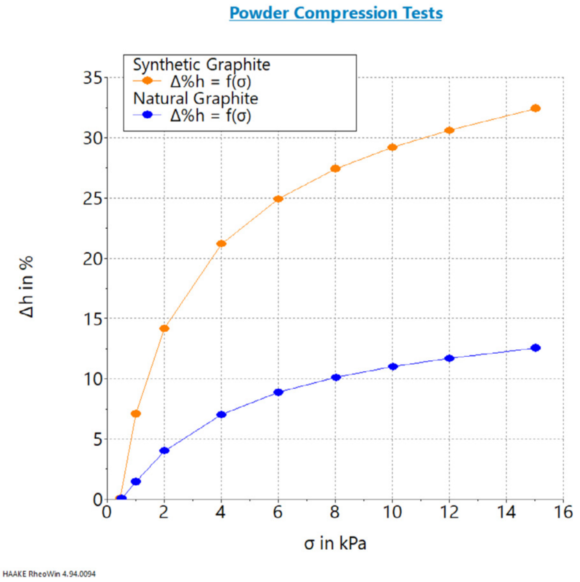

Figure 2. Results of the compression test for synthetic and natural graphite powders.

compressibility compared to the natural graphite powder. Under a final compression stress of 15 kPa, the sample volume of the synthetic powder decreased by more than 30%, whereas the natural graphite powder only showed a reduction of around 12%. Higher compressibility typically suggests larger amounts of entrapped air and/or smaller particle sizes, but may also be due to an increased cohesiveness of a powder sample. In battery slurry applications, increased cohesiveness of graphite powder can lead to challenges in achieving uniform mixing and dispersion, resulting in uneven electrode coatings and inconsistent battery performance.

Figure 3 shows the results of the powder flow test for the two graphite powders. The flow energies calculated from the torque, as well as the normal force recorded during the helical movement of the twisted blade rotor, are presented for a total of 11 test cycles.

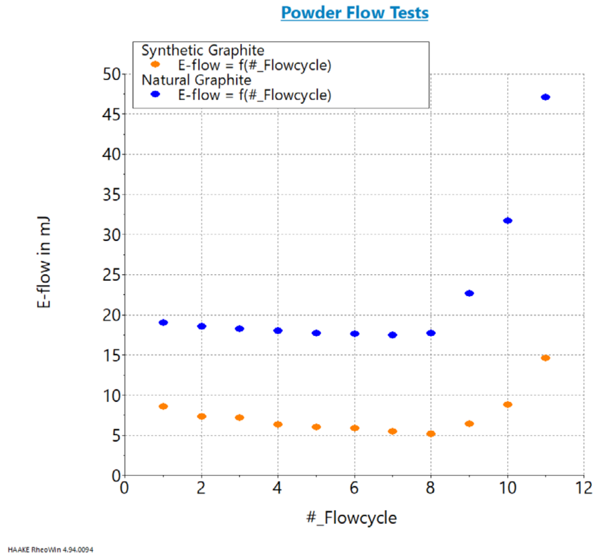

Figure 3. Results of powder flow test for synthetic and natural graphite powders.

The synthetic graphite powder shows lower overall values for the flow energy and can therefore be expected to be more flowable than the natural graphite powder under low stress conditions. A lower flow energy and therefore a higher flowability of a powder usually makes it easier to handle and process. A higher flow energy on the other hand can complicate tasks such as feeding the powder into a mixer or alternatively into an extruder for continuous slurry production.

However, upon examining the measurement data of the synthetic graphite sample in the flow test, it is evident that the data points initially drop at a constant rotor tip speed (data points 1 to 8) before rising again as the speed decreases (data points 9 to 11). A drop in flow energy at the beginning of the test may indicate the appearance of flow instabilities. This behavior is also reflected in the relatively low stability index (SI) value of 0.64 (see Table 1). The SI, defined as the ratio of the flow energy from the first to the eighth data point, should ideally be around 1. However, the significant deviation from this ideal value suggests inconsistent flow behavior.

Further inspection of the measuring geometry, specifically the measuring cup after the flow test, confirms that the powder flow process was suboptimal. A stable powder collar formed on the cup wall during the measurement (Figure 4a), indicating that the synthetic graphite powder did not flow evenly around the rotating blade rotor but was instead deposited on the wall. This behavior may be attributed to increased cohesiveness, rendering the flow energy measurement data unrepresentative of the actual powder behavior.

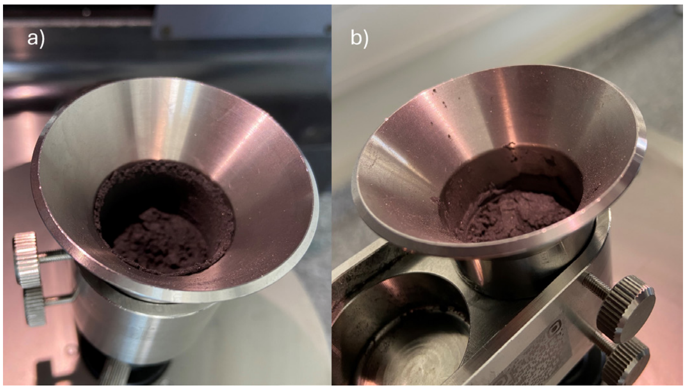

Figure 4a,b. Comparison of measuring cup after powder flow test. (a) With synthetic graphite powder, sample collar formation is visible, while (b) for the natural graphite powder no collar formation is observed.

Therefore, the powder flow test does not appear suitable for the synthetic graphite powder sample. In contrast, the natural graphite powder exhibited a significantly higher stability index of around 0.9, with no collar formation observed on the cup wall after the test (see Figure 4b).

A summary of the complete evaluation results for the powder flow test for the two samples is shown in Table 1.

Table 1. Evaluation results of the powder flow test for synthetic and natural graphite powders.

	Synthetic graphite powder	Natural graphite powder
Basic flowability energy (BFE) in mJ	5.50	17.5
Stability index (SI)	0.640	0.93
Specific energy (SE) in mJ/g	1.39	1.54
Flow rate index (FRI)	2.79	2.65
Conditioned bulk density (CBD) in g/ml	0.319	0.792

Figures 5a and 5b show the evaluation results with the yield locus analysis of the powder flow test performed at consolidation stresses of 6 kPa and 9 kPa for the two graphite powders.

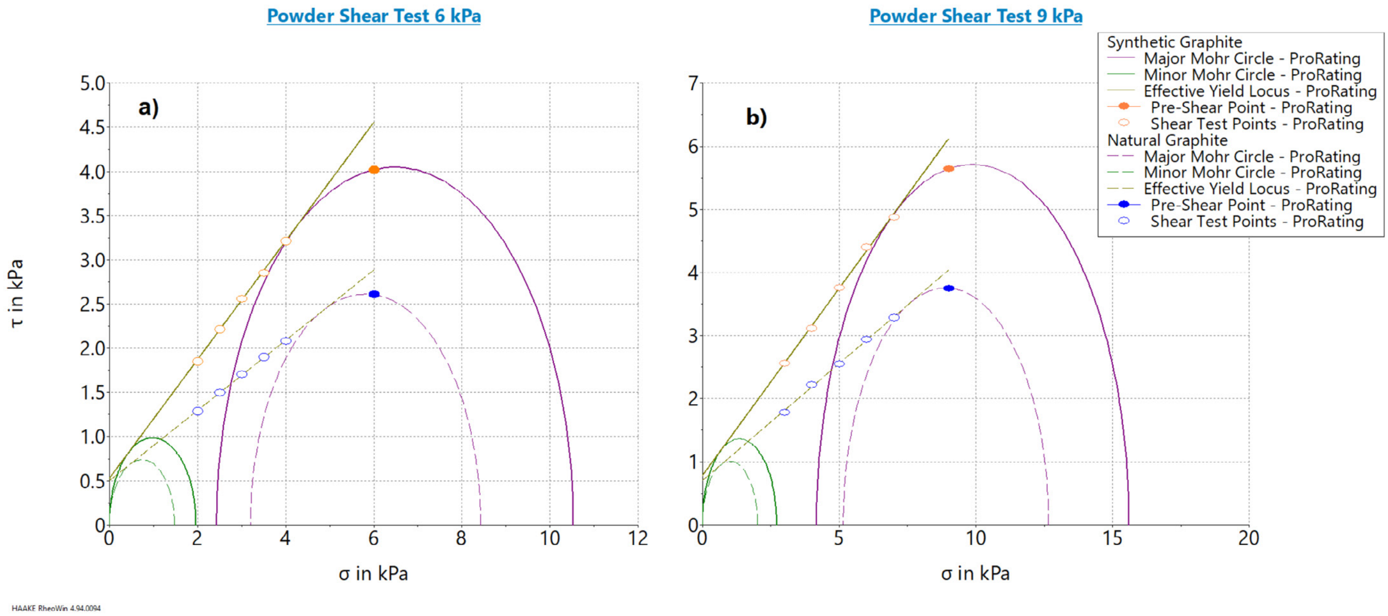

Figure 5. Results of the powder shear tests performed at consolidation stresses of (a) 6 kPa and (b) 9 kPa for synthetic and natural graphite powders

The minor and the major Mohr stress circles, shown in Figure 5 are used to determine the Unconfined Yield Strength (UYS) and the Major Principal Stress (MPS). The distance of the yield locus regression line to the X-axis at a normal stress of 0 kPa describes the cohesiveness of the sample.6

The synthetic graphite powder exhibits overall higher shear stress values compared to the natural graphite powder, indicating that more energy is required to induce flow under an axial load. Additionally, the cohesion values determined at each consolidation stress are somewhat higher for synthetic graphite than for natural graphite (see Table 2).

The flow behavior under varying axial loads can be further analyzed using a flow function plot, as shown in Figure 6. In this plot, the Unconfined Yield Strength (UYS) is plotted against the Major Principal Stress (MPS) for all consolidation stresses tested.

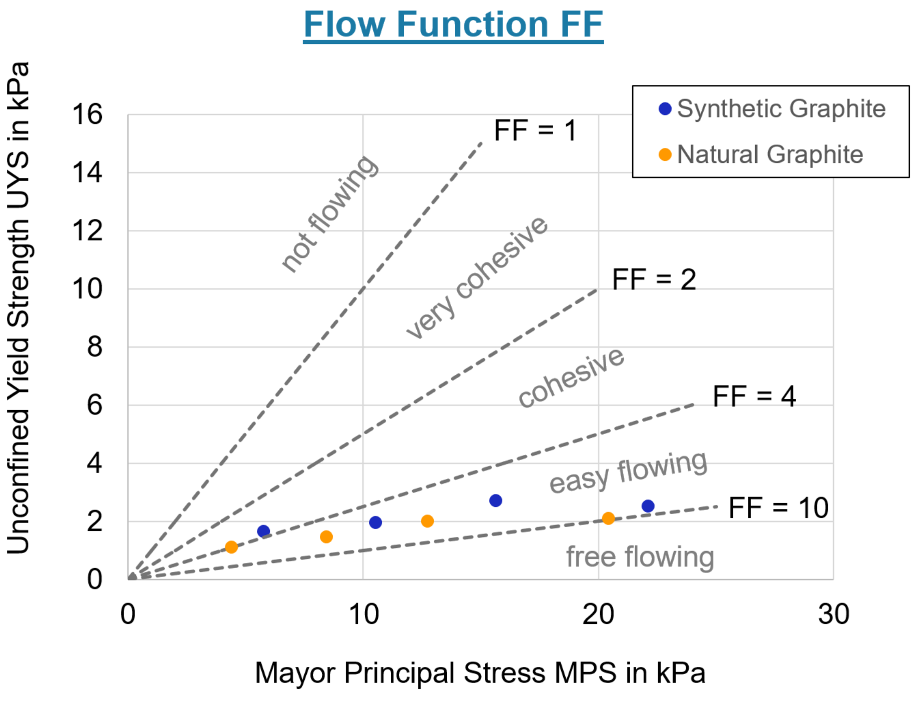

Figure 6. Flow function analysis for synthetic and natural graphite powders

Even though the synthetic graphite powder generates in general higher shear stress values, the overall flow behavior under axial load can be considered as ‘easy flowing’ for both powders. A summary of all flow parameters extracted from the powder shear test is shown in Table 2.

Table 2. Evaluation results of the powder shear test for synthetic and natural graphite powders.

	Synthetic graphite powder				Natural graphite powder			
Consolidation stress in kPa	3	6	9	15	3	6	9	15
Major principal stress (MPS) in kPa	5.74	10.5	15.6	22.1	4.37	8.42	12.7	20.4
Unconfined yield strength (UYS) in kPa	1.66	1.97	2.72	2.54	1.12	1.48	2.02	2.12
Cohesion in kPa	0.520	0.525	0.775	0.752	0.372	0.502	0.702	0.736
Effective angle of internal friction in °	39.5	38.7	35.3	31.2	30.5	26.7	24.9	23.5
Angle of internal friction in °	38.8	38.5	34.0	30.8	24.2	22.8	21.2	21.5
Flow function (FF)	3.50	5.35	5.73	8.70	3.89	5.70	6.27	9.61

When performing the complete cycle of powder rheological experiments, differences between the two powder samples become apparent. The natural graphite powder is less compactable, shows no flow anomalies in the powder flow test, and exhibits lower shear strength under axial load. The lower cohesiveness and generally better flow properties indicate that the natural graphite powder is easier to process than the synthetic graphite powder.

Conclusion

In this study, two different graphite powders were tested for their compression and flow behavior under varying conditions. All tests were performed using a rotational rheometer equipped with a specific accessory for powder rheology. Even though the tests revealed differences between natural and synthetic graphite powders, both are likely to be suitable for battery electrode slurry production. A thorough understanding of powder flow properties, as described in this report, can help manufacturers identify changes in slurry quality and adjust processing parameters accordingly.

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