

Wet grinding of activated carbon in a Retsch planetary ball mill

Instrument: SYNC

Activated Carbon

Activated carbon is a highly porous material with a large internal surface area, which has a wide range of applications due to its good adsorption properties, for example in water purifiers or in air filters. Activated carbon is usually produced from charcoal, and many substances with a high carbon content can be the precursor material: wood, nut shells, peat, petrochemical products, or bones. The internal structure of activated carbon consists of large pores created during the production process. Activated carbon is available in different specifications/forms - e. g. as powder, granulate or extrudate.

In the grinding experiment described here, a sample of activated carbon granulate from coconut shells with an initial particle size of 1-2 millimeters (Fig. 2) was to be ground to less than 1 μm . Such small particle sizes are hardly achievable by dry milling. Therefore, wet grinding was carried out in a Retsch PM100 planetary ball mill (Fig. 1). The liquid used was isopropanol, which has better wetting properties than water.

The grinding progress was continuously documented by laser diffraction. For the measurements, a Microtrac SYNC (Fig. 1) was used, which is suitable for particle analyses in the size range 10 nm to 4 mm.



Fig. 1: Retsch Planetary Ball Mill PM100 (left) and Microtrac Laser Diffraction Analyzer SYNC (right).

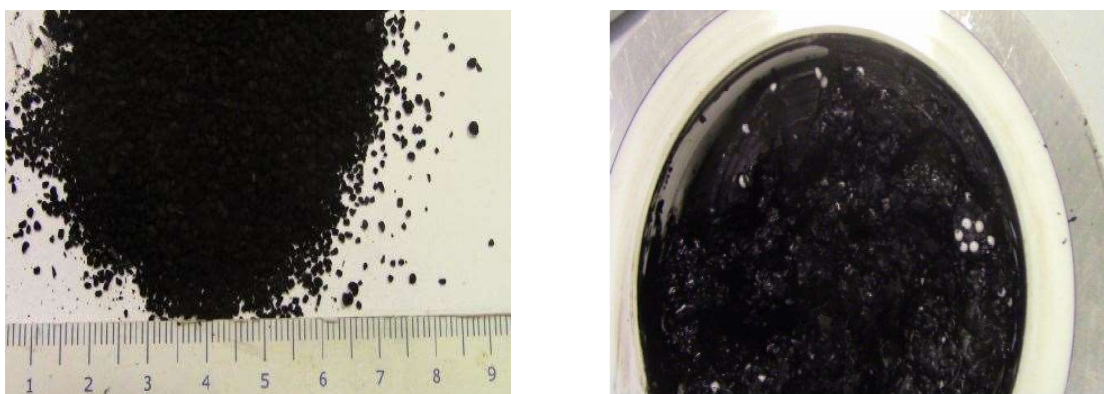


Fig. 2: Feed material was granular activated carbon (left), ground sample paste in a zirconia (ZrO_2) jar (right). White zirconia grinding balls are clearly visible.

Grinding Experiment and Measurement Conditions

Grinding Experiment:

- Material: Activated Carbon from coconut shells
- Feed size: approx. 1 mm
- Feed quantity: 32 g (approx. 125 ml)
- Material properties: hard, brittle
- Mill: Retsch PM100 Planetary Ball Mill
- Configuration: 500 ml zirconia grinding jar, 1000 g zirconia grinding balls, 3mm, 200 ml isopropanol
- Revolution Speed: 480 rpm
- Overall grinding time: 2 hours 30 minutes
- Particle Size Analysis with SYNC Laser Diffraction: after 5 min, 30 min, 60 min, 1 h 45 min und 2 h 30 min

After grinding, the sample can be separated from the grinding balls by sieving.

Measurement conditions Laser Diffraction:

- Instrument: SYNC, configuration 3R (three red lasers, wavelength 780 nm, Fig. 3)
- Dispersion: wet measurement with FlowSync in isopropanol
- Pump speed: 55 %
- Ultrasound: 60 seconds, Power 100 %
- Analysis time: 20 seconds
- Evaluation acc. to Mie theory, refractive index particle: 1.85, refractive index liquid: 1,38
- Particle model: irregular particles
- Standard: ISO 13320

The setup of laser diffraction analysis is illustrated in Fig. 3. The scattering or diffraction angle depends on the particle size, with large particles scattering light to small angles and small particles scattering light to large angles.

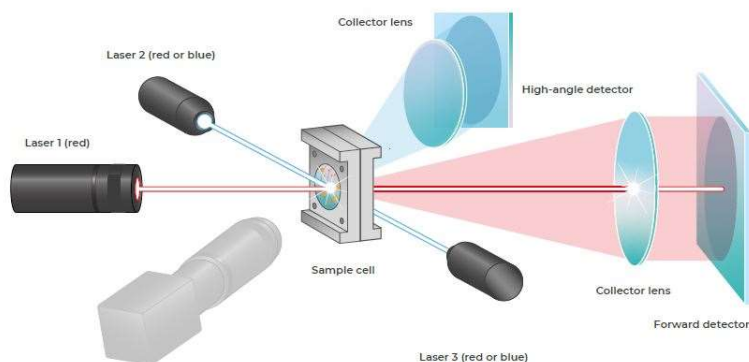


Fig. 3: Operating principle of laser diffraction analysis with SYNC. The sample in the measuring cell is illuminated by three lasers. Two detector arrays record the scattered light over an angular range from 0° to 163°. The particle size distribution is calculated from the scattered light pattern.

Results

The grinding results are shown in Table 1 and the corresponding diagram (Fig. 4). Usually, percentile values at 10%, 50% and 90% are given in particle analysis to describe the size distribution (d10, d50, d90). These values respectively indicate the size below which 10%, 50% or 90% of the sample is located (example: d90 = 1 µm means 90% of the sample is smaller than 1 µm). The percentages are always by volume.

After only 5 minutes, a d50 (median) of less than 5 µm and a d90 of approx. 10 µm is reached. After 60 minutes, the d50 is reduced to 1 µm, i. e. half of the sample volume consists of particles smaller than 1 µm.

After 150 minutes, a median of 680 nm is reached, and the d90 is just below 2 µm. No progress can be detected here compared to the measurement after 105 minutes, so there is hardly any further progress. Higher final fineness could possibly be achieved if smaller grinding balls were used.

The reports of the individual measurements are listed in the appendix.

Grinding time [min]	d10 [µm]	d50 [µm]	d90 [µm]
5	2,00	4,53	10,25
30	0,57	1,75	3,78
60	0,48	1,00	2,64
105	0,43	0,76	1,76
150	0,40	0,68	1,88

Table 1: Grinding progress expressed as percentiles.

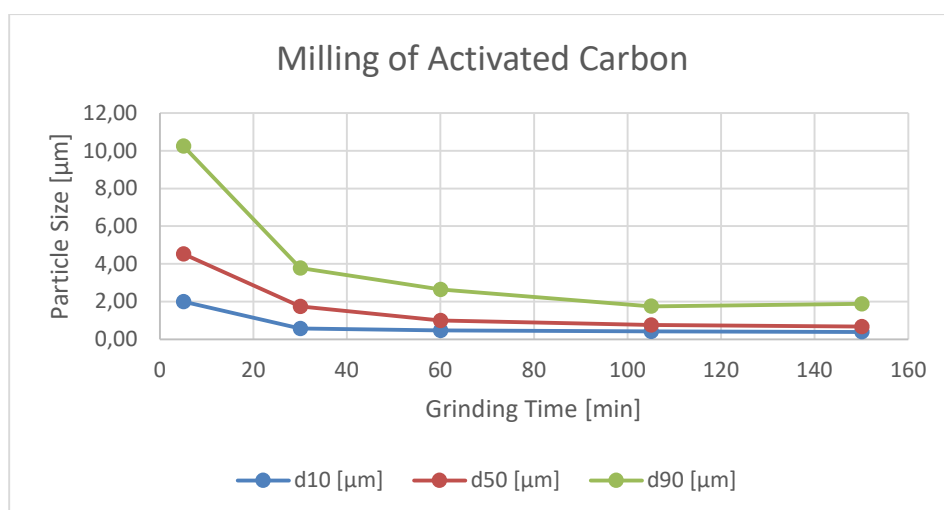


Fig. 4: Change of d10, d50 and d90 with progressive grinding time.

Summary

Thanks to the wide size range and short analysis times, the Microtrac SYNC is ideally suited for documenting grinding processes. By repeated sampling after a defined grinding time, it can be determined that the fastest progress is made at the beginning of the milling. This makes it easy to find out when the desired result has been achieved. When the particle size distribution no longer changes with extended grinding time, the final fineness has been reached.

For further information please contact us at:

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