APPLICATION NOTE

MICROTRAC MRB

PARTICLE CHARACTERIZATION

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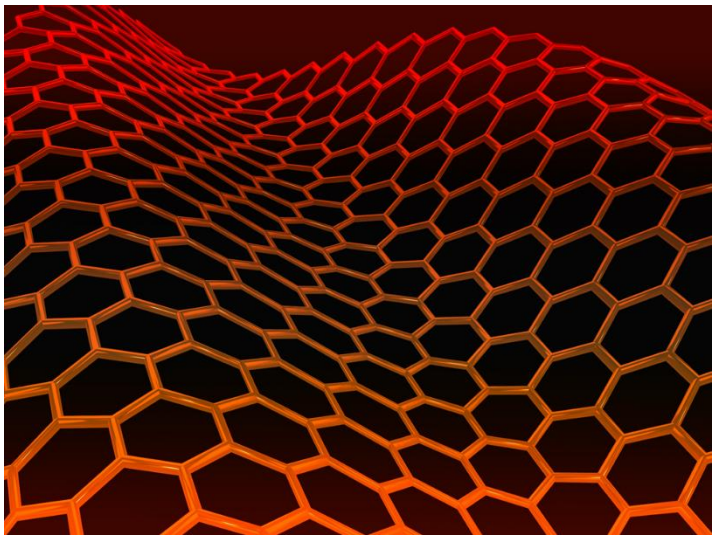
Carbon compounds – a question of the surface and how to determine it

Introduction

Carbon occurs in many different allotropic modifications, with very different physical properties and therefore can be used in a wide range of applications. Carbon black takes many different forms; as a pigment and in the rubber industry, an abrasive in the form of diamond and as a lubricant due to its layer structure in the form of graphite. conductive modifications are also possible, graphite and CNT can be used as an admixture to polymers to increase their conductivity, but also as an electrode material, e.g. in fuel cells (SOFC) or energy storage (Li-ion batteries).



Although the modifications differ strongly macroscopically, the surface chemistry is comparable, but also very complex. Due to production, purification and specific surface modification, different functional groups (such as carboxyls, lactones, phenols, carbonyls, anhydrides) are formed, which have a decisive influence on processing and product properties.

Spectroscopic methods are often used to characterize the surface groups, but these only allow qualitative statements to be made. The quantitative determination is mostly done by Boehm titration (https://de.wikipedia.org/wiki/Boehm-Titration). Volumetric titration (back titration) is carried out at different pH values to differentiate between different groups. However, this procedure is very time-consuming, and the evaluation is inaccurate. In addition, not only the functional groups on the surface are recorded, but also those in bulk (for agglomerates and porous materials), which have only a minor influence on processing and product properties.

Determination of the functional groups with the StabinoZeta:

Both the measurement of the pH dependence of the zeta potential and the determination of the surface charge provide important information about the type and number of functional groups of carbon compounds.

- pH titration

In pH titration, the dependence of the zeta potential on the pH value is determined. In this way, on the one hand, stability ranges can be identified and on the other hand the isoelectric point can be determined. Figure 1 illustrates the influence of the manufacturing conditions on the stability ranges and the IEP.

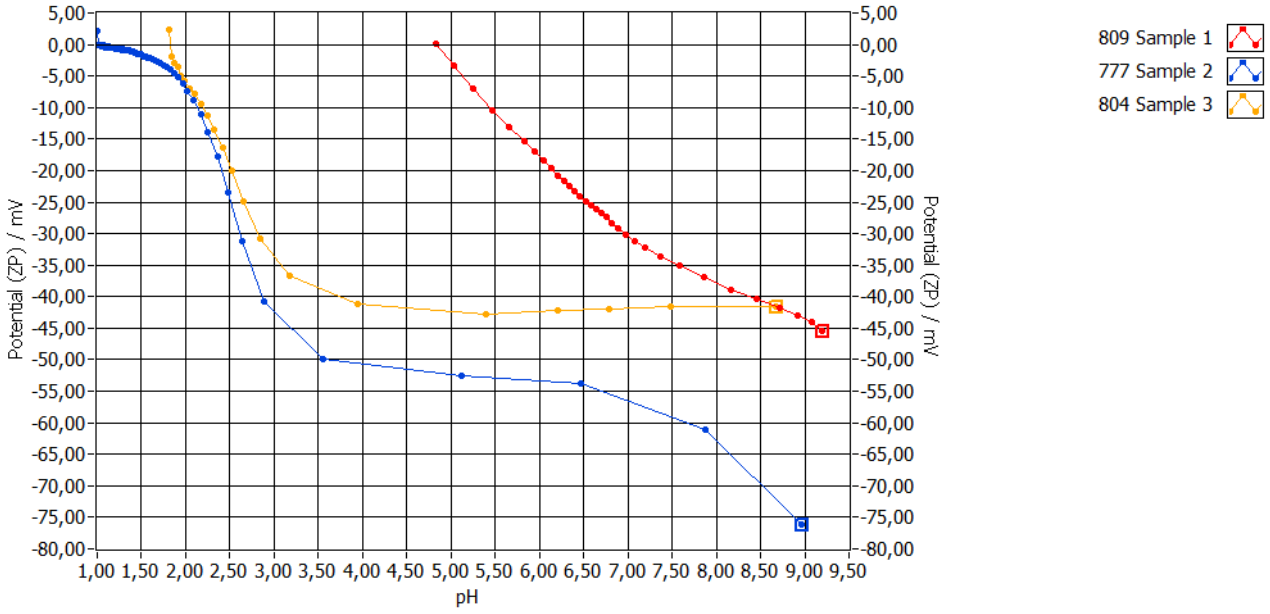
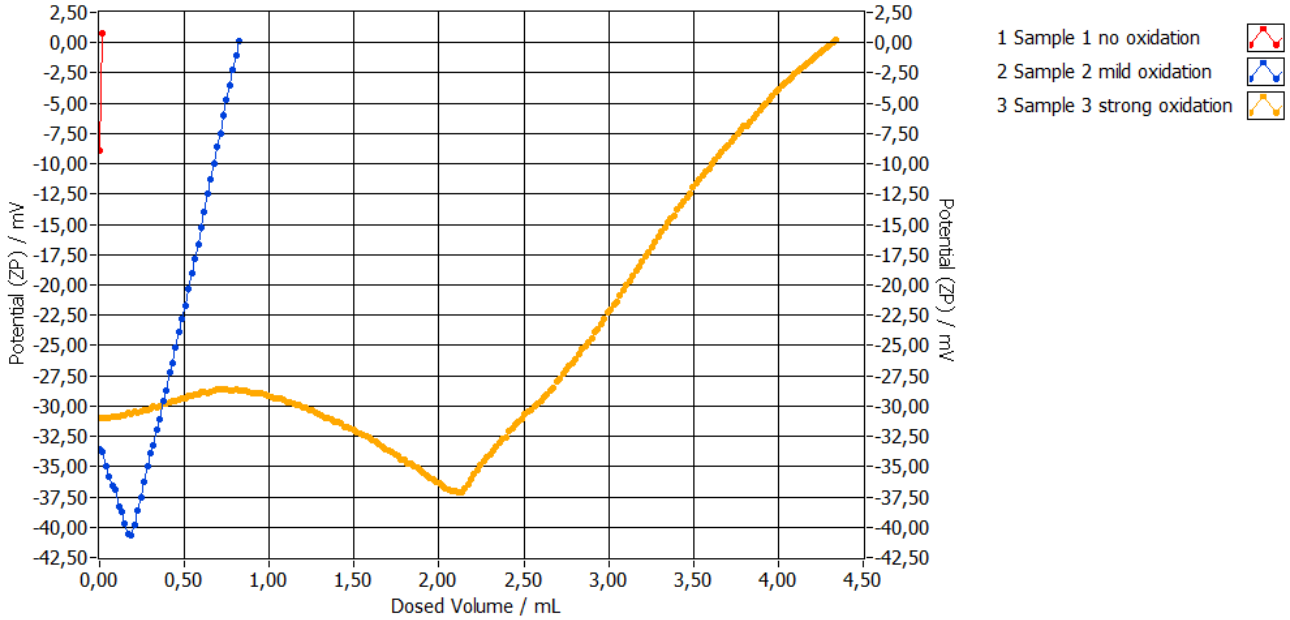


Figure 1: pH titration of differently prepared carbon black samples

Knowledge of the IEP makes it possible to quickly and precisely identify suitable starting materials for different applications and to recognize stable pH ranges.

- Polyelectrolyte titration

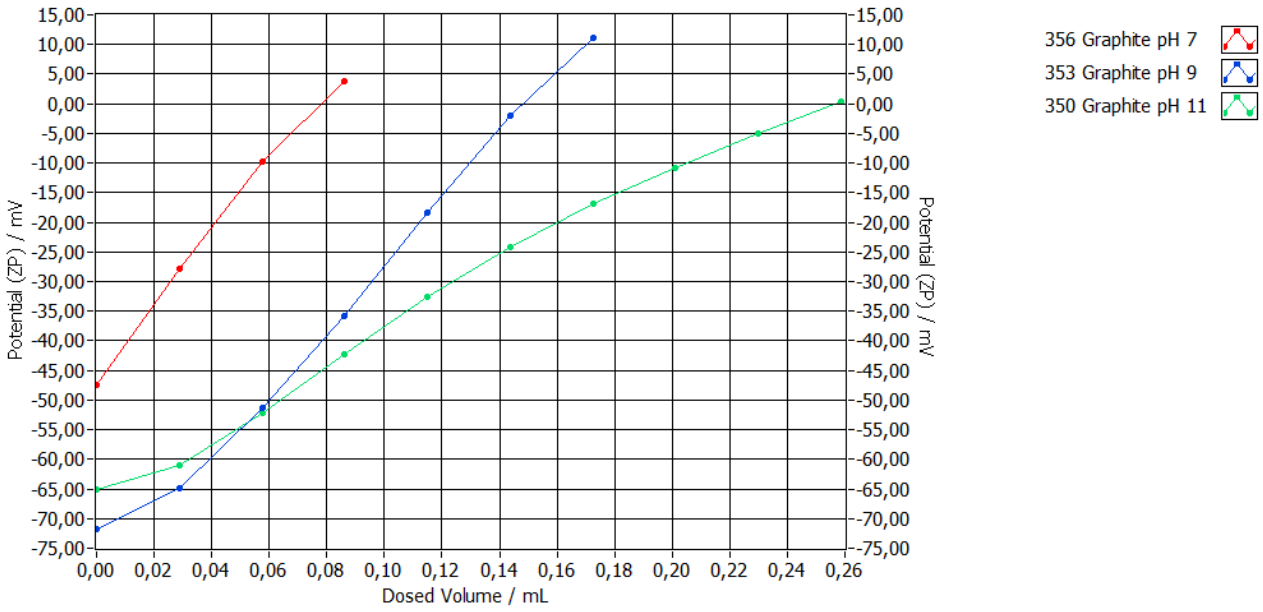
Much more detailed information about the surface chemistry is obtained by determining the surface charge density. Figure 2 shows the influence of different reaction conditions during the oxidation of carbon black produced by flame pyrolysis on the surface charge density.



Sample	Titrant	Volumen mL	Charge / C g^{-1}	Charge/ µeq g^{-1}
1 Sample 1 no oxidation	P-DADMAC	0.017	0.042	431
2 Sample 2 mild oxidation	P-DADMAC	0.820	1.979	20507
3 Sample 3 strong oxidation	P-DADMAC	4.321	10.423	108026

Figure 2: Polyelectrolyte titration of differently oxidized carbon black samples

During the titration the Poly-DADMAC adsorbs on the particle surface and neutralizes the charge In addition to the quantification of the adsorption sites or coordination sites, polyelectrolyte titration is also suitable for quality control. The combination of both methods, i.e. the measurement of the surface charge density at different pH values, enables the functional surface groups to be differentiated on the basis of their acid strength (Fig. 3).



Sample	Titrant	Volumen mL	Charge / C g^{-1}	Charge/ µeq g^{-1}
Graphite pH 11	P-DADMAC	0.257	0.155	1607
Graphite pH 9	P-DADMAC	0.148	0.089	966
Graphite pH 7	P-DADMAC	0.078	0.047	488

Figure 3: Charge determination of graphite at different pH values

Polyelectrolyte titration provides information about the charge conditions at different pH values. By difference formation the amount of charge can be assigned to the different functional groups.

Conclusion

With the Stabino®, stability ranges of various carbon compounds can be determined and compared very quickly and reproducibly. The determination of the charge density allows a quick quantification of the functional groups on the surface. Besides the time saving in charge determination, a major advantage is that, in contrast to Böhm titration, only the functional groups localized on the surface and thus reactive are detected.