

Measuring comparative electrical conductivity after mixing polyethylene pellets with carbon black

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Introduction

Conductive carbon black is used with a wide range of polymers to obtain permanently antistatic, dissipative or electro-conductive properties in plastics, rubber and paints. The carbon black impacts both electrical and thermal conductivity. It also influences the electromagnetic properties and coloration of paints and varnishes as well as the coloration of plastics and rubber. Typical application areas include extruded profiles, pipes, sheets, injection molded parts, and blown and cast films.

Carbon black types are available with different conductive properties to serve in various applications. Carbon black is also produced in batches and can vary from batch to batch. That is why it's important to have a reliable characterization method to quantify conductivity for different types and batches of carbon black.

Test equipment

High density polyethylene (HDPE) was melted in a laboratory mixer and blended with three different carbon black (CB) types in three consecutive experiments. The system used to prepare the three HDPE + CB samples was a Thermo Scientific™ HAAKE™ PolyLab™ OS Modular Torque Rheometer and an electrically heated Thermo Scientific™ HAAKE™ Rheomix Lab Mixer (600 OS version) with pneumatic ram, roller rotors and the option to measure comparative electrical conductivity. The sensor for measuring both temperature and conductivity (see Fig. 1) can penetrate the sample and is isolated from the mixing chamber. The system measures the resistance between the conductivity sensor and the mixing chamber for an output value of Siemens $S = \frac{1}{\Omega}$. The higher the comparative conductivity value in Siemens, the better the conductance in the final mixture.

Test conditions

33 g of HDPE pellets were put into the mixer chamber and mixed for 5 minutes at a rotor speed of 100 rpm and a mixer-temperature of 150 °C. During this period, the mixer torque and conductivity of the polymer was measured and displayed in a rheogram (Graphs 1, 2 and 3). After

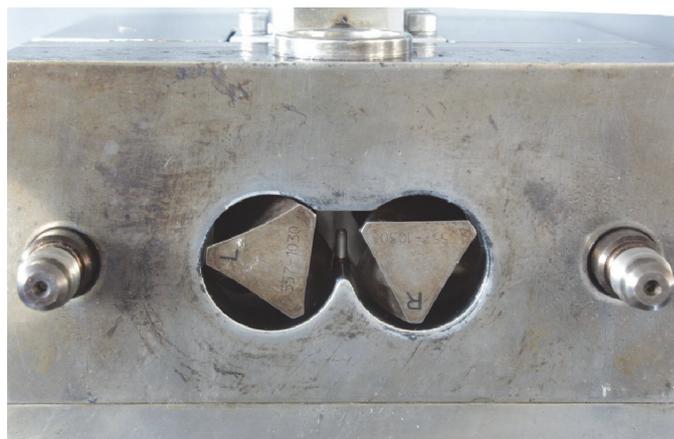


Figure 1: The chamber of the Rheomix Lab Mixer (600 OS version) with combined melt temperature and conductivity sensor.

the polymer was molten, the pneumatic ram was lifted up and 4 g of carbon black was added. The ram was lowered to close the mixer chamber. Mixing and measuring continued for another 10 minutes.

Materials and results

Three different samples were examined using the same grade of HDPE and 3 different types of carbon black (CB1, CB2 and CB3). For CB1, two manufacturing batches were tested to check for variation between batches.

The mixer results for the different samples and batches are shown in Table 1. The torque (left y-axis) and the electrical conductivity (right y-axis) of all tests are displayed with the same scaling for the y-axis in all rheograms.

To show the reproducibility of the test method, tests with carbon black type 1 batch 2 (sample numbers 2 and 3) were done twice (see Graph 1). Both test results showed the same comparative conductivity base line (red curves) of 230 mS, and the torque signal showed the typical loading peak and decreases to 13.4 Nm when the HDPE was completely molten.

Sample no.	Sample CB type	Specific Resistance [$\Omega \cdot m$] [1]	Electrical Conductivity [$mS \cdot m^{-1}$] [1]s	Comparative Conductivity [mS]
1	CB1-Batch1	max. 8000	max. 0.125	1,890 @ 15 min
2	CB1-Batch2	max. 8000	max. 0.125	620 @ 15 min
3	CB1-Batch2-Rep	max. 8000	max. 0.125	550 @ 15 min
4	CB2	max. 2000	max. 0.5	12,200 @ 15 min
5	CB3	∞	0	240 @ 15 min

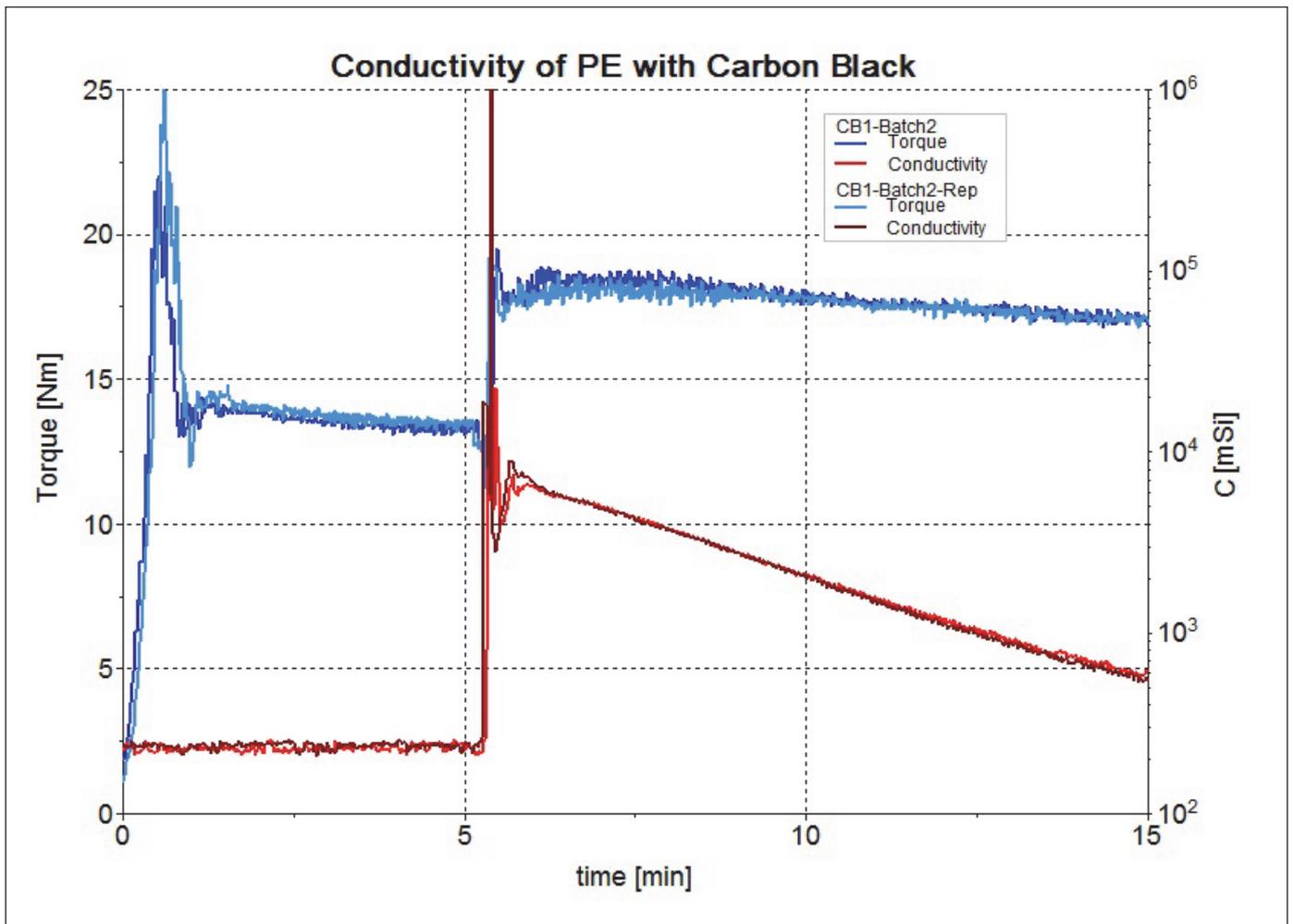
Table 1: HDPE + CB samples, their electrical resistance and productivity. Electrical conductivity obtained by van der Pauw method [1].

After 5 minutes, the carbon black was added. The measure of conductivity showed an immediate increase from the carbon black addition due to the conductivity sensor's location in the mixer chamber. After more thorough mixing, the comparative conductivity decreased again as the carbon black particles were better distributed within the polymer matrix. After 15 minutes, the output value was 620 mS and 550 mS respectively. Adding the carbon black resulted in a higher torque signal (blue curves) because it reinforced the polymer melt. It leveled out at the end of the mixing time at 17 Nm. The results show good repeatability for the test method.

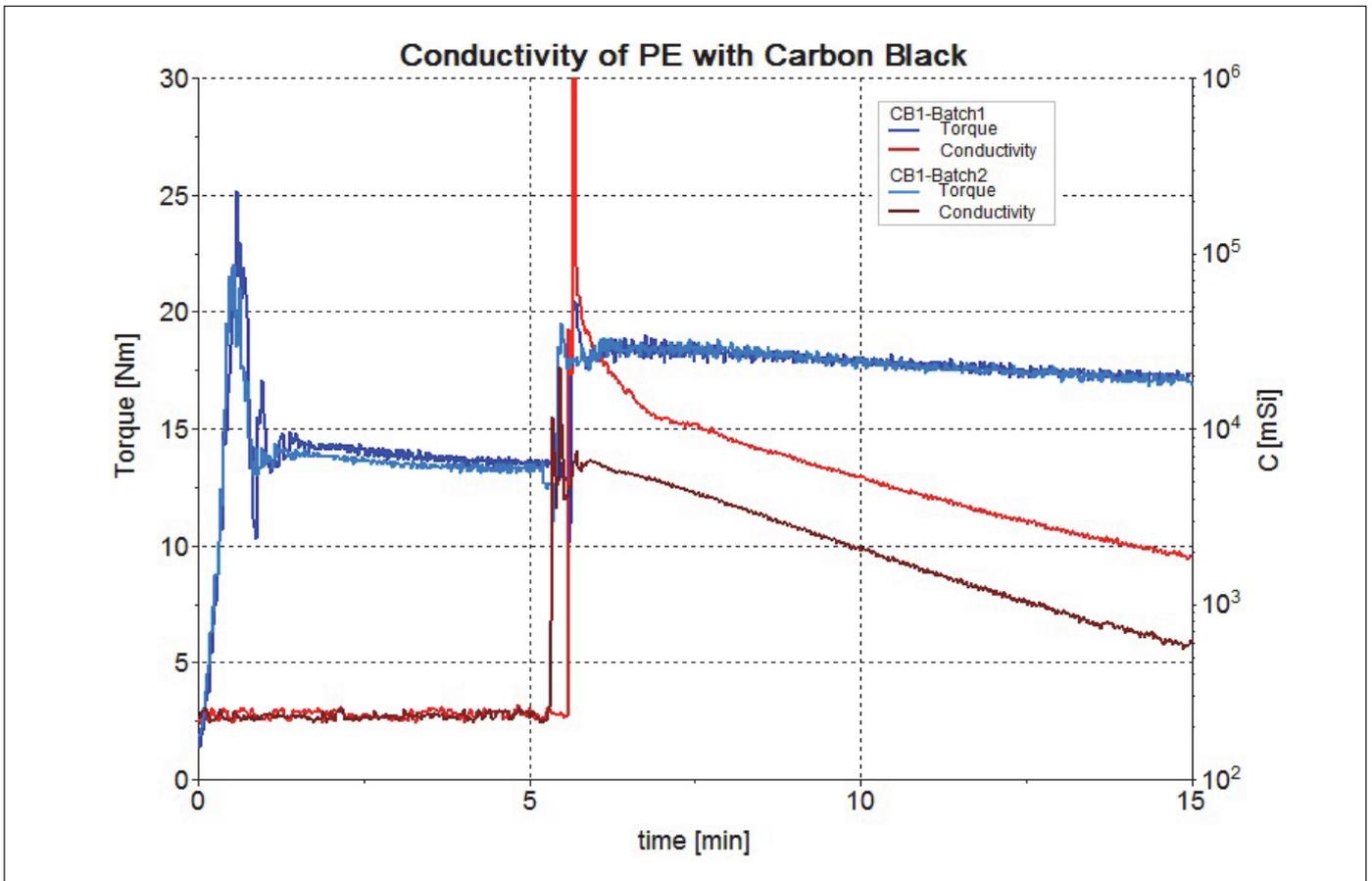
The differentiation between the two batches of carbon black type 1 (sample 1 and sample 2) is shown in Graph 2.

The readings of the comparative conductivity show a difference while the torque for both samples (blue curves) is equal with 17 Nm at the end of the test. After 15 minutes of mixing, batch 1 of carbon black type 1 (sample 1) has a value of 1890 mS (light red curve) compared to batch 2 of carbon black type 1 (sample 2) with only 620 mS (dark red line).

The comparison of all conducted tests (see Graph 3) shows good correlation with the electrical conductivity of the raw carbon blacks listed in Table 1.



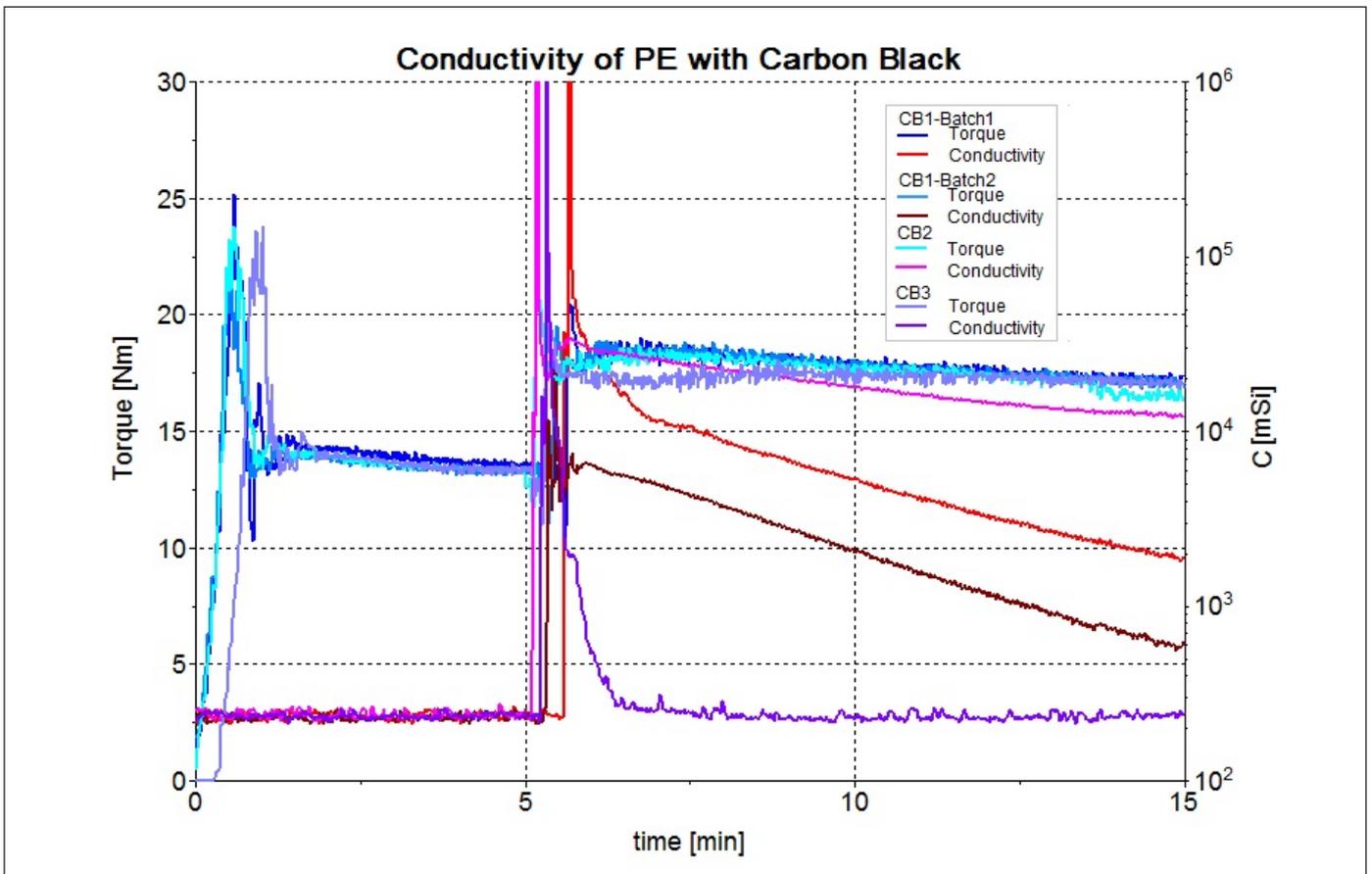
Graph 1: Repeatability of the mixer test with samples 2 and 3.



Graph 2: Variance of two batches of carbon black type 1 (sample 1 and sample 2).

Carbon black type 2 (pink line) shows the highest comparative conductivity at the end of the mixer test with 12,200 mS. Carbon black type 3 (violet line) with a base line value of 245 mS after 15 minutes of mixing time isn't conductive.

All conductive carbon blacks show similar torque curves. The torque curve of the nonconductive CB3 is slightly different after the carbon black is added to the polymer melt.



Graph 3: Rheograms of all four types of carbon black tested.

Conclusion

Using the HAAKE PolyLab OS system with a batch mixer enables the researcher to obtain reproducible results of comparative conductivity data with minimal effort in a few minutes. This report shows how that can be done in an example using samples of HDPE with different carbon black types. Differences in batches and among various carbon black types can be reliably monitored. The measured results have good correlation with those values obtained by the van der Pauw method [1] for electrical conductivity (see Table 1). Beyond that, the comparative conductivity measurement allows for a more precise characterization of the value to help material researchers further optimize their formulations. This test method can also be used with rubber applications [2].

Literature

- [1] A Method of Measuring Specific Resistivity and Hall Effect of Discs of Arbitrary Shape. - J. van der Pauw: Philips Res. Reports 13 (1), 1958, p. 1-9.
- [2] Macro- and microdispersion of carbon black in liquid silicone rubbers Le, H. H.; Ilisch, Sybill; Radusch, Hans-Joachim; Steinberger, H.; Plastics, rubber and composites. - London: IOM, Bd. 37.2008, 8, S. 367-375.

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