

Thank you for considering Ketjenblack for your Research project. To get the maximum results with our product please study the below document before conducting experiments. The success of your experiments will greatly depend on your ability to disperse our Ketjenblack well in your polymer matrix. Experience learned that obtaining a good dispersion can be difficult to achieve, therefore below some information, tips and tricks in dispersing and measuring. Please note that just mixing Ketjenblack into your compound generally will not lead to the desired results.

Please read instructions

Dispersion is key

Using correct equipment

Tips & tricks

## Conductive blacks

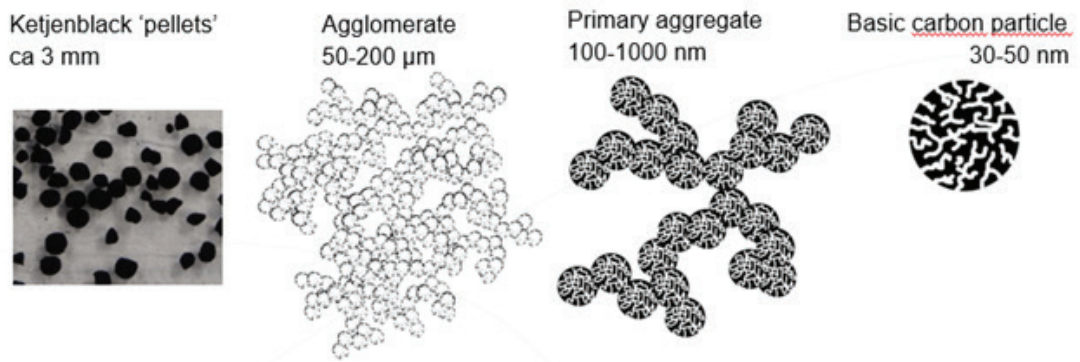
Conductive blacks are classified as specialty carbon blacks. Ketjenblacks, as well as many other conductive blacks in the market, are furnace blacks. They are produced with the furnace process, where oil related streams undergo an incomplete combustion in an oven at drastic conditions.

### Material structure

Ketjenblack itself is a material with an intrinsic resistivity of about 0.01 - 0.1 [ $\Omega$ -cm].

Ketjenblack is mostly available as pellets of around 3 mm (Figure 1). These pellets can be easily broken into the smaller particles that constitute them: the agglomerates, with a size of roughly 100 micrometers ( $\mu$ m). Agglomerates are formed by several aggregates, which are even smaller particles of roughly 0.1 - 1  $\mu$ m. Aggregates are the key particles for conductivity. They tend to stick together, and high shear forces are necessary to break the agglomerates into aggregates.

Figure 1. Ketjenblack structure



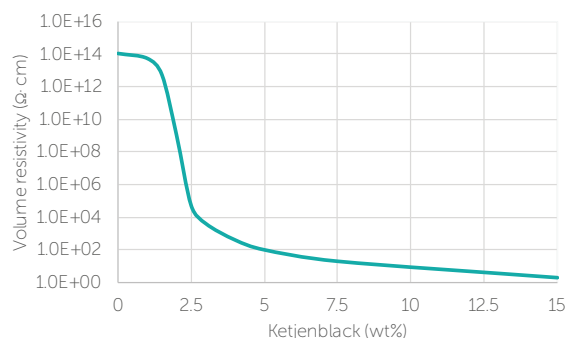
## Mode of action

To get conductivity, the carbon black particles need to form a network through which the electrons can be transferred. The percolation curve shows the formation of a conductive network (Figure 2). Until a certain dosage, no much change in conductivity (the inverse of resistivity) occurs. At a certain critical concentration, the polymer/rubber starts becoming conductive (percolation point).

When the percolation curve becomes flat, addition of more conductive black will not change the conductivity. Before the percolation curve, there is just not enough conductive filler: the distance between the carbon black particles is too long and electrons cannot transfer from one particle to the other.

When percolation is reached, the distance is just enough to allow electron transfer.

Figure 2. Percolation curve



## Mixing

Do not  
mix by  
hand

During mixing or compounding, Ketjenblack must first be wetted by the polymer in the compound matrix, which takes time, due to its high surface area and pore volume (low bulk density). The wetting process can be controlled regulating the mixing temperature. Furthermore, sufficient mixing-energy must be provided to disperse the wetted carbon black uniformly into the compound.

Essential for such a uniform distribution of carbon black is enough shear during compounding. The regulation of temperature (wetting) and shear forces (dispersion of Ketjenblack agglomerates) are two very important parameters that need to be carefully calibrated to find the best compromise, especially considering the type of polymer in use.

### Do not mix by hand! This will not lead to the desired dispersion!

Suitable mixing equipment to incorporate Ketjenblack into a matrix are:

- Polymers
  - Extruder (i.e. twin screw)
  - Kneader (i.e. Buss)
  - Internal mixer (i.e. Banbury or Brabender)
- Rubber
  - 2 or 3 roll mill
  - Kneader
  - Internal mixer (i.e. Banbury or Brabender)
- Coatings
  - High speed disperser + Bead mill

## How to work with Ketjenblack

We have experience on lab-scale. This may be suitable for industrial use with bigger machines.

### Raw materials

- Standard UP resin: Palatal P4 (any resin would work)
- Ketjenblack EC300J intake: 1.5 wt% normally enough to reach percolation point

### Lab procedure

- Weigh out resin + optional dispersing additive (viscosity reducer)
- Stir for 1 minute, add KB, stir gently until all KB is wetted – 1 minute
- Mix for 15 minutes: 4 cm diameter dissolver with 4660 rpm (tip speed 10 m/s). The blade being 1/3 of the vessel diameter and being mounted in the center of the mix vessel
- Temperature increase up to 40-50°C is to be expected, if a larger set up (vessel + dissolver) is used, the rpm can be recalculated to keep tip speed on 10 m/s

Some notes on curing:

Ketjenblack might affect the curing, depending on the curing system used. However, this is not only related to Ketjenblack; all blacks have the same effect. Nouryon has some knowledge and can assist/ advise when needed.

## Polyolefins (like polyethylene/polypropylene)

We have experience on lab-scale using the internal mixer.

Raw materials:

- Polymer
- Ketjenblack EC300J intake around 5-8%, Ketjenblack EC600JD around 3-5% (with good dispersion enough to reach the percolation point)

Compounding Lab Procedure using the internal mixer:

- Weigh out the polymer and add half of the amount to the mixer when the desired temperature is reached (at ca 50 rpm)
- Weigh out the correct amount of Ketjenblack
- Lower the speed of the driver unit and carefully add Ketjenblack to the mixer
- Add the (left over) polymer directly on top of the Ketjenblack and close the internal mixer by lowering the stamp, and increase the speed to its original rpm
- Mix for 15 minutes
- Empty the internal mixer

After compounding you can use the material for further experimentation like compression molding (CM). When a sheet is CM the conductivity can be determined using the proper equipment.

## Rubber

Do not mix on 2-roll mill

Do not mix Ketjenblack directly on a two-roll mill! We have experience on lab-scale using first the internal mixer to incorporate Ketjenblack into the polymer matrix. The regulation of temperature (wetting) and shear forces (dispersion of Ketjenblack agglomerates) are two very important parameters that need to be carefully calibrated to find the best compromise, especially considering the type of rubber used.

Raw materials:

- Rubber, Ketjenblack, Oil, Antidegradant system

Proposed mixing procedure using the internal mixer (temperature and mixing speed depending on the polymer matrix used):

Time (min.)	Action
0	Addition Rubber
1	½ Ketjenblack + oil
2	½ Ketjenblack + antidegradant system
4	Sweep ram
5	Discharge mixer

Another example is a mixing method for high extended EPDM compounds

Raw materials:

- High extended EPDM rubber, Ketjenblack, Other ingredients (e.g. oil, fillers)

Settings for internal mixer (upside down procedure) for high extended EPDM compounds:

- rotor speed 114 rpm
- start temperature 50°C; direct cooling

Mixing procedure:

Time (min.)	Action
0	Add all ingredients (except the rubber)
0.5	Add the rubber
3	Sweep ram
4	Discharge mixer

Following step might be using a two-roll mill to create a nice workable sample, and to add all kind of ingredients needed in your experiment like curing agents or other additives.

## Dispersion

Inert fillers, processing aids and crosslinking agents can influence the conductivity of the compound since they affect the dispersion of the conductive black in the matrix.

Inert fillers (talc, glass fibers, ...) can reduce the resistivity of the compound.

Processing aids (like Armowax W-440) can improve the dispersion of Ketjenblack in the matrix and therefore lowers the volume resistivity.

A better dispersion of the conductive black will also improve the mechanical properties of the compound.

Crosslinking agents (like peroxides) are in most cases beneficial for the electroconductivity of a product, as it is thought to promote the flocculation of carbon black into conductive paths. But this is depending on the type of polymer used. When crosslinking an EPDM compound the crosslinking improves the conductivity, while in an EVA rubber the crosslinking deteriorates the conductivity.

Equipment to determine dispersion:

- Disper tester (MonTech), Dispergrader (Alpha Technologies), Optical microscopy

Figure 3. Good vs poor dispersion

