# Processing Guidelines

**for TUBALL™ MATRIX 605**
for High Consistency Silicones
in the hardness range from 30 to 70 Shore A

# 1. DILUTION PRINCIPLES

Uniform distribution of TUBALL™ MATRIX in the silicone plays a key role in enhancing the electrical conductivity of the final compound. In order to obtain a high-quality TUBALL™ MATRIX dispersion, OCSiAl recommends that close attention be paid to the dilution procedure.

- The resistivity level achieved will depend on the loading of TUBALL™ MATRIX. The target dosage of TUBALL™ MATRIX refers to the loading in the whole HCR formulation by weight.
- The mixing time, number of mixing cycles and mixing speed may need to be adapted for different machinery size/type to obtain a final mixture that is homogeneous.
- The dilution ratio depends on the required level of resistivity and the loading of TUBALL™ MATRIX.

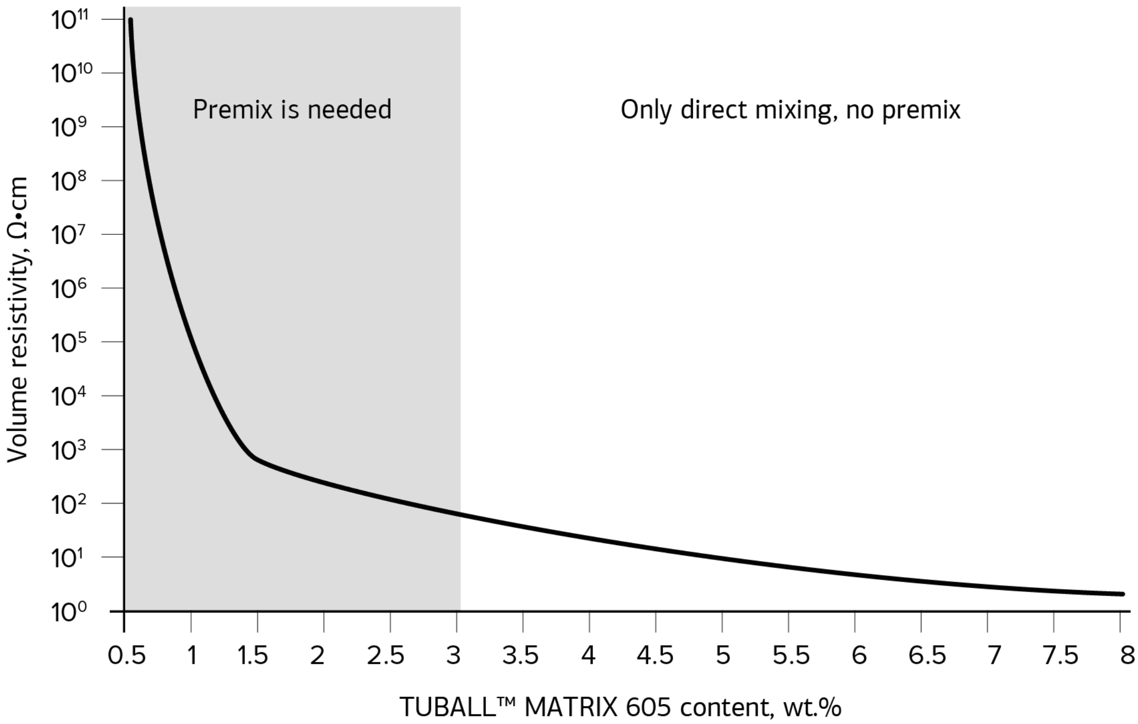



**Figure 1.** Volume resistivity of 60 Hardness HCR silicone (ELASTOSIL® R 401/60) with TUBALL™ MATRIX 605 in the range $10–10^{11}$ Ω·cm (sample shape: compression-moulded rubber sheet of 2 mm thickness, compounded on two-roll mill)

**! Please do mention, that due to extremely low dosage of conductive filler active content in HCR, percolation threshold could be affected by type of the filler in HCR (fumed silica based and precipitated silica based).**

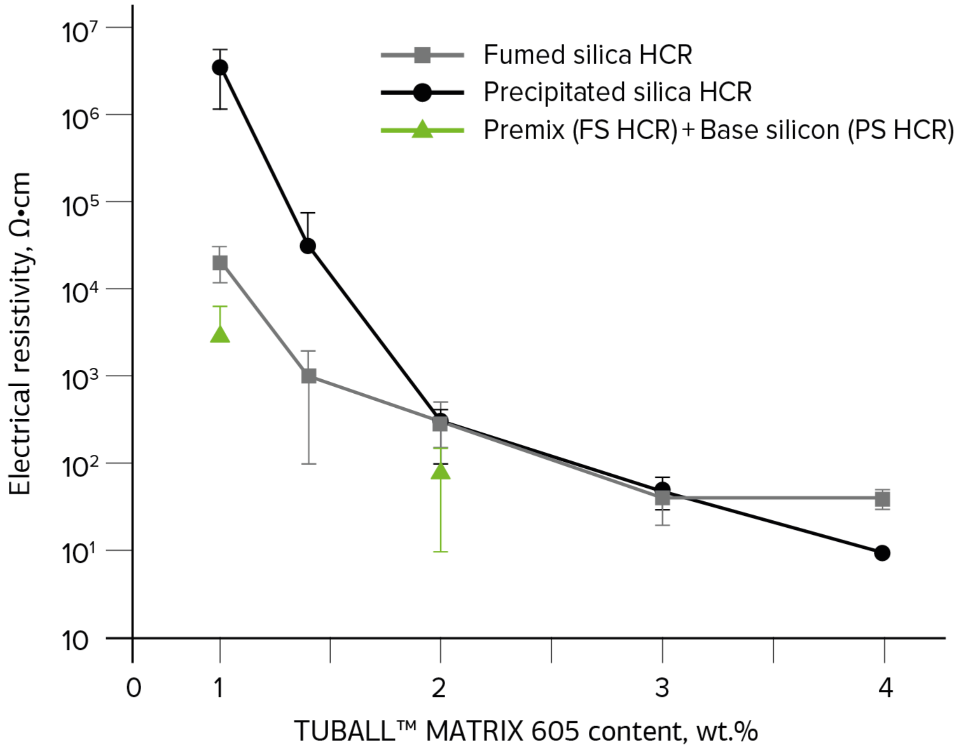



**Figure 2.** Volume resistivity of two types of 60 Hardness HCR silicone (Fumed silica based and Precipitated silica based) with TUBALL™ MATRIX 605 in the range $10^1$–$10^7$ Ω·cm (sample shape: compression-moulded rubber sheet of 2 mm thickness, compounded on two-roll mill)

**Key principles of dispersion of TUBALL™ MATRIX:**

The dispersion quality depends strongly on two factors:

a) the mechanical dispersion characteristics (shear forces during the compounding):
- type of machinery
- mixing modes

b) the carrier compatibility:
- viscosity of silicone base.

TUBALL™ MATRIX 605 is a high viscous carrier and thus the best compatibility is achieved with HCR in the hardness range from 50 to 70 Shore A. For HCR with lower hardness, such as 30 Shore A, a slightly higher concentration of TUBALL™ MATRIX 605 is required than that discussed in the guidelines below, because of the different shear force.

**! For exact percolation curves for low Hardness HCR (30 Shore A) and high Hardness (50-70 Shore A) mixed on two-roll mill and kneader please refer to Technical datasheet for TUBALL™ MATRIX 605.**

Figure 3 shows how the volume resistivity, the number of cycles on a two-roll mill and the TUBALL™ MATRIX distribution correlate. Plotting such a graph allows the optimal number of cycles to be determined.

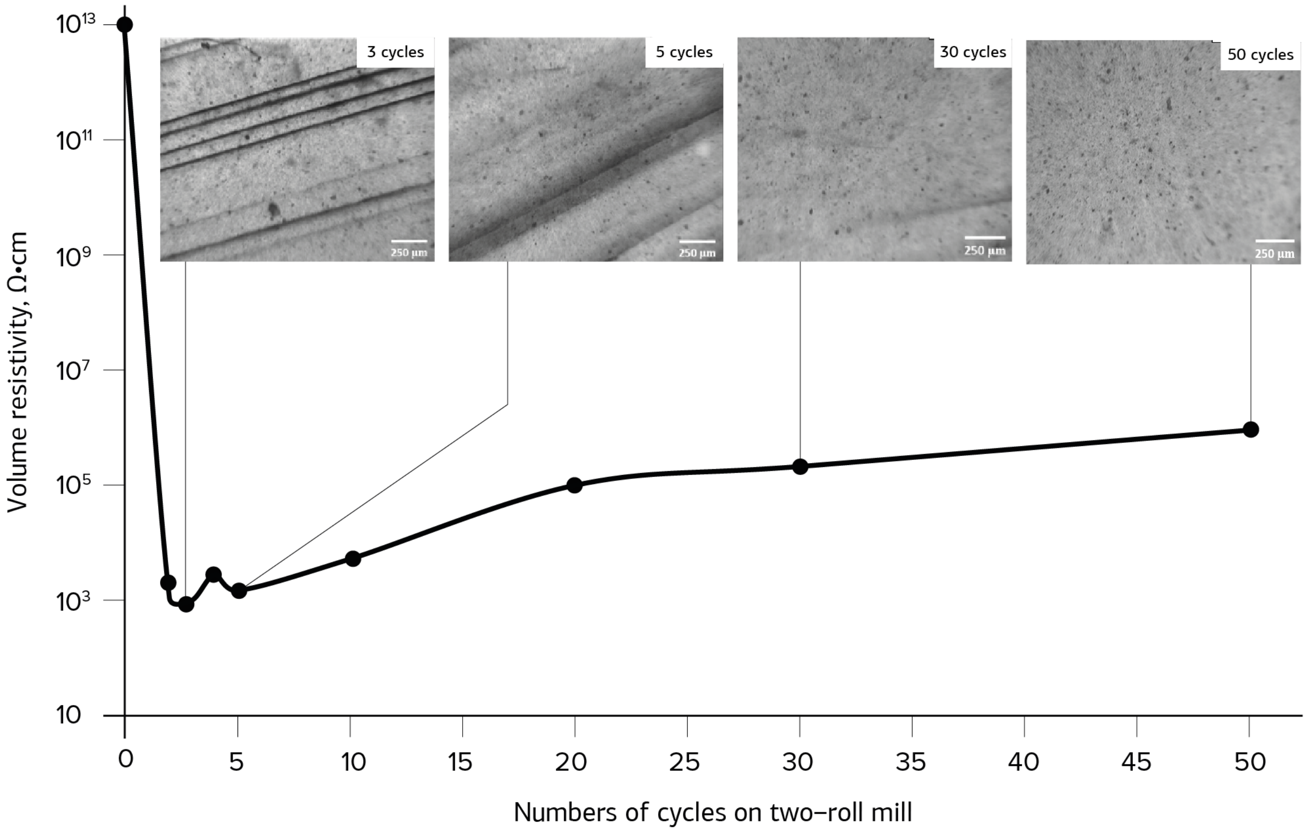
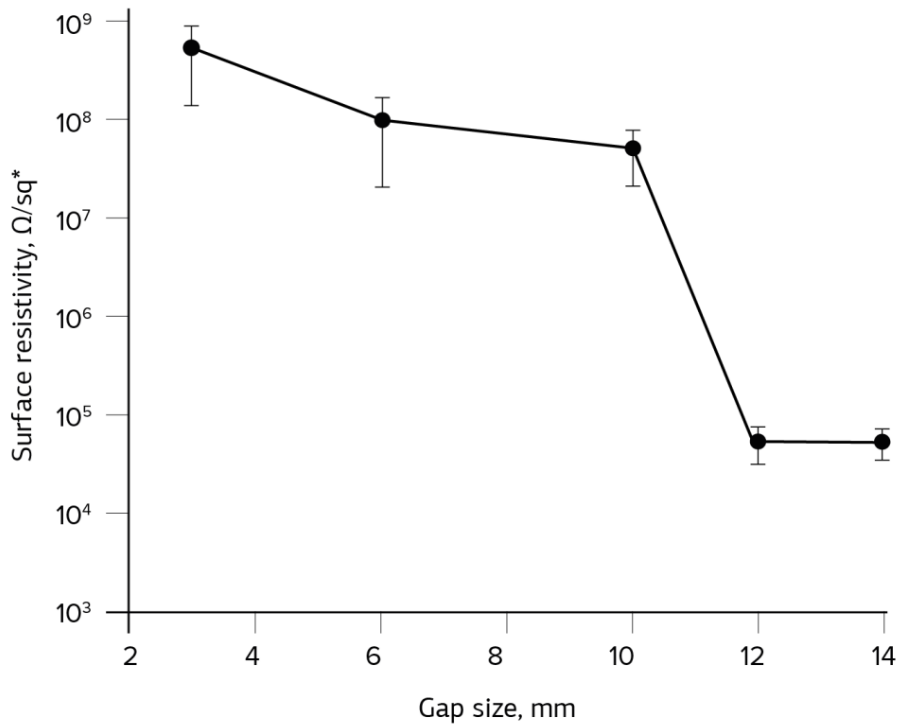



**Figure 3.** Correlation of volume resistivity, number of cycles on a two-roll mill and TUBALL™ MATRIX 605 distribution, TUBALL™ MATRIX 605 content — 0.8 wt. %

Figure 4 shows the correlation between surface resistivity and gap size for a two-roll mill with diameter of rollers 14 inches. Plotting such a graph allows the optimal gap size to be determined.




*Sample thickness 2 mm

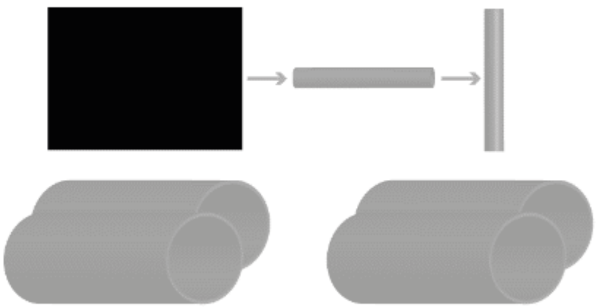
**Figure 4.** Surface resistivity as a function of gap size for a two-roll mill with diameter of rollers
14 inches, TUBALL™ MATRIX 605 content — 1 wt. %

# 2. DILUTION WITH A TWO–ROLL MILL WITH AND WITHOUT PREMIXING

**Figure 5.** Two-roll mill

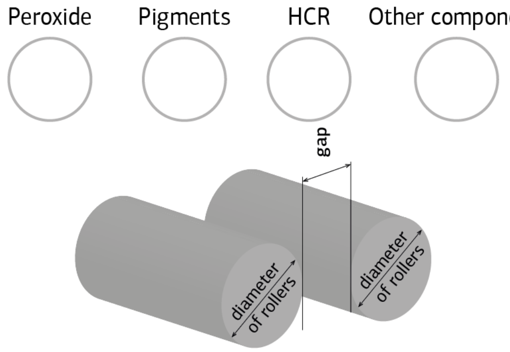
**Explanation, what is one cycle on a two-roll mill. Below cycle description ("Doll" mixing) is described:**

1. Turn on the machine and feed the materials between the rollers.
2. Remove material and form it into a tube – this is called a "cycle".
3. Turn the material by 90° and feed it back between the rollers as shown below.
4. Repeat steps 2 and 3 until **required number of cycles** have been fed through.



### *INITIAL STAGE*

Basic compound preparation

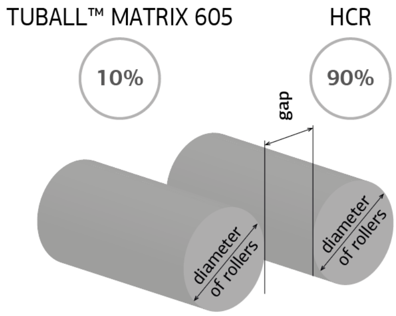



Normal compounding procedure

### *VOLUME RESISTIVITY $10^3$—$10^9$ Ω•cm — ANTI-STATIC COMPOUNDS*

Loading 0.5–3 wt.% of TUBALL™ MATRIX

a) Premixing stage of TUBALL™ MATRIX 605



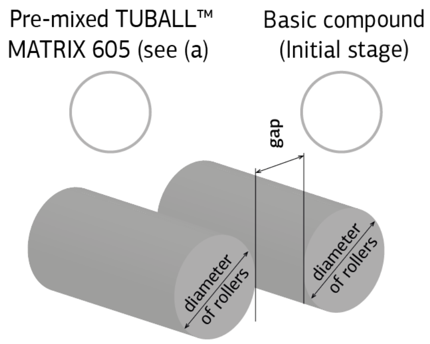

→ Set the gap between rollers

| Diameter of rollers | Gap |
|---|---|
| 6, 10, 14 inch | 3 mm |

→ Number of cycles

**30**

b) Final mixing




→ Set the gap between rollers

| Diameter of rollers | Gap |
|---|---|
| 6 inch | all batch sizes – 6 mm |
| 10 inch | <0.5 kg batch – 3 mm, 0.5–3 kg batch – 8 mm |
| 14 inch | 5–10 kg batch – 12 mm |

→ Choose number of cycles

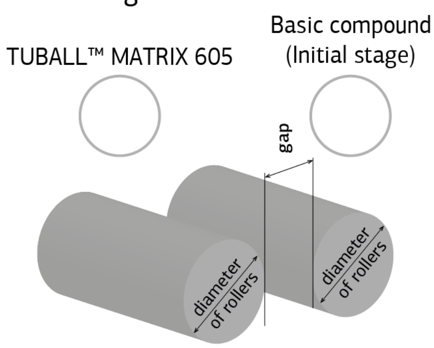
| Compound | Number of cycles |
|---|---|
| Black compound | 15 |
| Colour compound | 30 |

If the batch volume is different, please make an adjustment according to section 2 in TROUBLESHOOTING chapter

### *VOLUME RESISTIVITY <$10^3$ Ω•cm — CONDUCTIVE COMPOUNDS*

Loading 3–10 wt.% of TUBALL™ MATRIX

Direct mixing




→ Set the gap between rollers

| Diameter of rollers | Gap |
|---|---|
| 6, 10, 14 inch | <0.5 kg batch – 3 mm, >0.5 kg batch – 6 mm |

→ Number of cycles

**30**

# 3. DILUTION WITH A KNEADER WITH AND WITHOUT PREMIXING

**Figure 6.** Kneader

### First stage

Premixing of 10% TUBALL$^{TM}$ MATRIX 605 in 90% HCR on kneader according to the following procedure:

- Set the rotors speed 20:25 rpm.
- Put HCR and MATRIX 605 into mixing chamber
- Turn on rotors rotation
- Mix during 5 minutes
- Stop rotors rotation
- Finish mixing.

### Second stage

Dispersion of Peroxide and TUBALL$^{TM}$ MATRIX 605 (or Premix from the stage 1) in HCR on kneader according to the following procedure:

- Set the rotors speed 20:25 rpm. Put HCR and Peroxide into mixing chamber
- Turn on rotors rotation. Mix during 20 minutes
- Stop rotors rotation
- Add Pre-mixed TUBALL$^{TM}$ MATRIX 605 or pure TUBALL$^{TM}$ MATRIX 605 (depending on the recipe, refer to Fig.1) mix during 5 minutes
- Finish mixing.

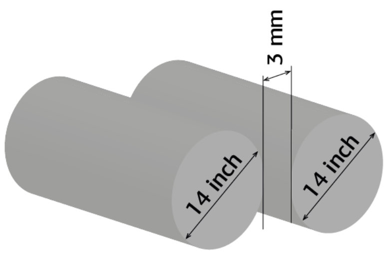
# 4. EXAMPLE OF COMPOUNDING WITH A TWO–ROLL MILL

Example of preparation of a coloured anti-static compound, based on 60 Shore A Hardness HCR:

- Targeted level of volume resistivity is $10^5$–$10^7$ Ω·cm.
- Required dosage of TUBALL™ MATRIX 605 is 0.8 wt.%.
- Equipment: a two–roll mill with 14 inch roll diameter.

Stage 1. Basic compound preparation

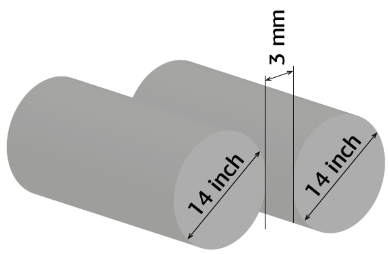
Luperox F 40P: 1.8%
Coloring paste: 5%
$TiO_2$: 1.5%
HCR: 83.7%




**Normal compounding procedure**

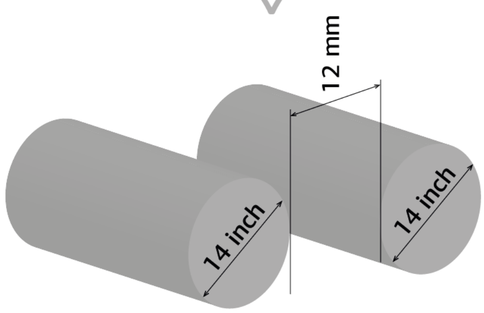
Stage 2. TUBALL™ MATRIX 605 premixing

HCR: 7.2%
TUBALL™ MATRIX 605: 0.8%




**30 cycles**

Stage 3. Final mixing




**30 cycles**

**READY!**

# 5. MOULDING AND CURING

**1. Shelf life of compound.** The shelf life of the final compound in the uncured state must be determined experimentally for each particular compound.

**2. Curing agents.** Compatibility of curing agents with TUBALL™ MATRIX.

Standard curing agents that are compatible with TUBALL™ MATRIX:

- 2,5-bis(tert-butylperoxy)-2,5-dimethylhexane (example trademark DHBP-45-PSI);
- 1,3(4)-bis(tert-butylperoxyisopropyl)benzene (example trademark Luperox F40P);
- dicumyl peroxide, also known as bis(1-methyl-1-phenylethyl)peroxide or bis(α,α-dimethylbenzyl) peroxide;
- platinum catalyst and crosslinker.

**NOTE!**

Standard peroxide, i.e. 2,4-dichlorobenzoylperoxide (DHBP), is unsuitable as it is strongly inhibited by any filler.

As all curing agent systems are impacted by a high amount of fillers, adjustment of the curing system is required for loadings of TUBALL™ MATRIX of more than 6% (which is required to achieve a volume resistivity of less than 10 Ω·cm).

**3. Post curing.** Electrical conductivity is typically increased by the post curing process. For example, a volume resistivity of $10^8$ Ω·cm after curing will improve further down to $10^6$ Ω·cm after post curing for 4 hours at 200 °C. An exact impact depends on curing agents and moulding process.

**4. Moulding process.** Electrical conductivity could be affected by the moulding process due to differences in the shear forces. All the data provided in technical documents is based on a compression moulding process.

- Compression moulding – no impact on electrical conductivity.
- Extrusion moulding – no impact on electrical conductivity.
- Transfer moulding – resistivity is higher than with compression or extrusion-moulded parts.
- Injection moulding – resistivity is higher than with compression or extrusion-moulded parts.

An exact impact to electrical resistivity by moulding process should be determined an experimental way.

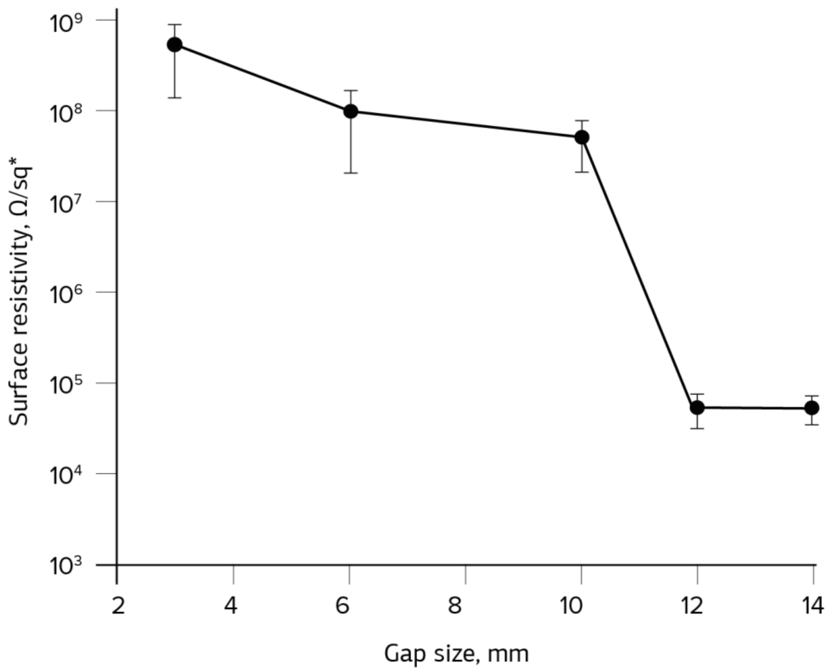
# 6. TROUBLESHOOTING

1. **The shelf life of the final compound in the uncured state must be determined experimentally for each particular compound.**

2. **Optimization of parameters for a two-roll mill in a roll diameter size different from 6, 10, 14 inch.**

   **a) Determination of the optimal gap size**

The optimal gap should be determined by experiment for each size of machinery to achieve the required parameters (electrical conductivity, colour, etc.).
Figure 6 shows an example of this optimisation process for two-roll mill with diameter of rollers 14 inches. The gap size was varied and the surface resistivity of the various cured samples was measured. The conclusion from Figure 7 is that the optimal gap size is 12–14 mm (to achieve the lowest surface resistivity). This is the gap size that should then be used during final mixing.

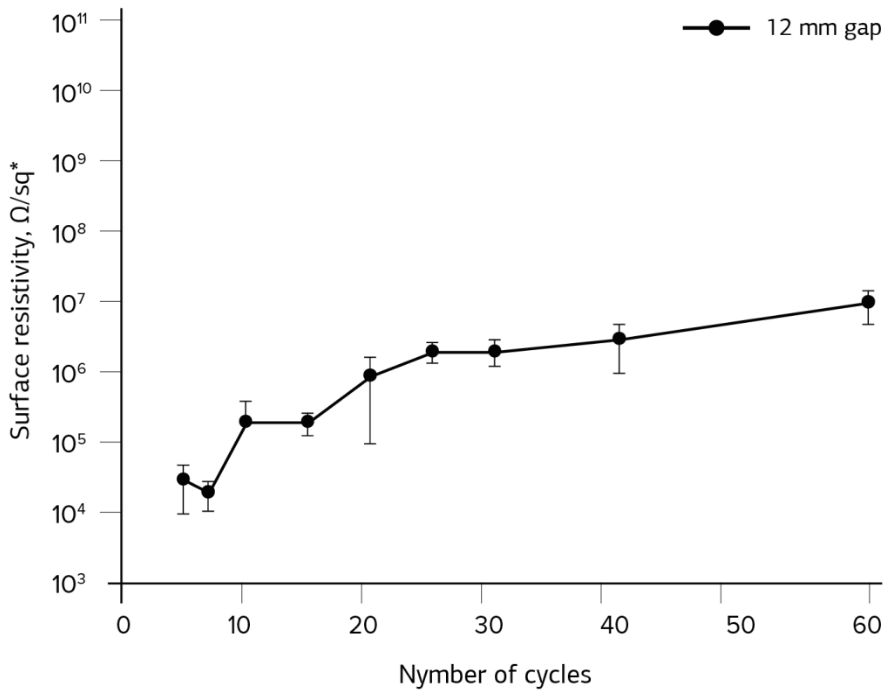



*Sample thickness 2 mm

**Figure 7.** Surface resistivity as a function of gap size for a two-roll mill with diameter of rollers 14 inches, TUBALL™ MATRIX 605 content — 1 wt. %

### b) Determination of the optimal number of cycles

The number of cycles (7–30 cycles) should be varied to achieve the required parameters (electrical conductivity, colour, etc.) and an optimum should be chosen. The optimal number of cycles is different for each machinery size.
Figure 8 shows an example where, at a gap size of 12 mm, the number of cycles was varied and the surface resistivity of the various cured samples was measured. The conclusion is that, if the desired surface resistivity is in the range $10^5$–$10^7$ Ω/sq, then the optimal number of cycles is 10–20.




*Sample thickness 2 mm

**Figure 8.** Surface resistivity as a function of number of cycles on two-roll mill at a gap size of 12 mm, TUBALL™ MATRIX 605 content — 1 wt. %

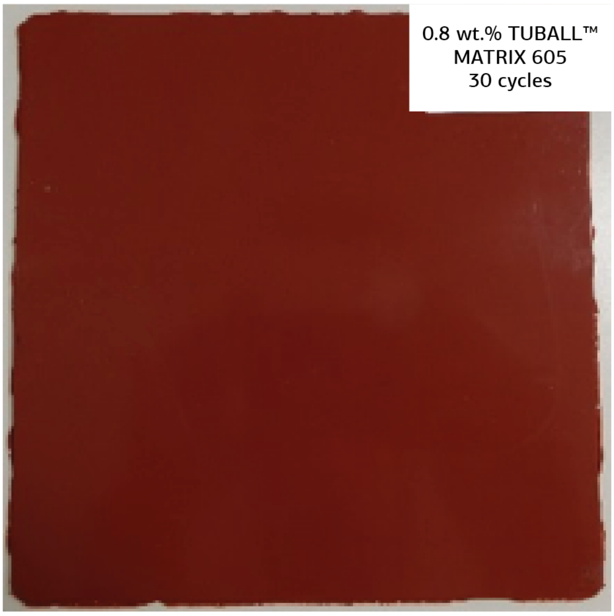
## 3. Coloured compounds

**If the product is coloured**, then the number of cycles during final mixing should be optimised by considering the colour uniformity. For this, visual analysis is needed. For example, for the colour recipe shown below, it is evident that at least 30 cycles are needed to achieve the best colour uniformity.

0.8 wt.% TUBALL™ MATRIX 605
3 cycles

0.8 wt.% TUBALL™ MATRIX 605
5 cycles

0.8 wt.% TUBALL™ MATRIX 605
15 cycles



0.8 wt.% TUBALL™ MATRIX 605
30 cycles