



PROCESSING GUIDELINES

TUBALL™ MATRIX 814 FOR PVC PLASTISOL

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INTRODUCTION

TUBALL™ MATRIX 814 is available in black color flakes with a pasty texture form.

Uniform distribution of TUBALL™ MATRIX 814 in the PVC plastisol plays a key role in enhancing the electrical conductivity of the final compound. In order to obtain a high-quality TUBALL™ MATRIX 814 dispersion, OCSiAl recommends that close attention be paid to the dilution procedure.

Figure 1. TUBALL™ MATRIX 814 appearance



RECOMMENDED EQUIPMENT

For laboratory tests: a stirrer with a mixing speed of up to 2000 rpm (such as the Heidolph RZR series or the IKA EUROSTAR series).

For industrial production: dissolvers similar to the DISPERMAT CA series.

Dilution should be conducted in a cylindrical mixing container with a flat bottom.

Figure 2. Recommended impeller blade shape.



PROCEDURE

STEP 1. Target dosage determination

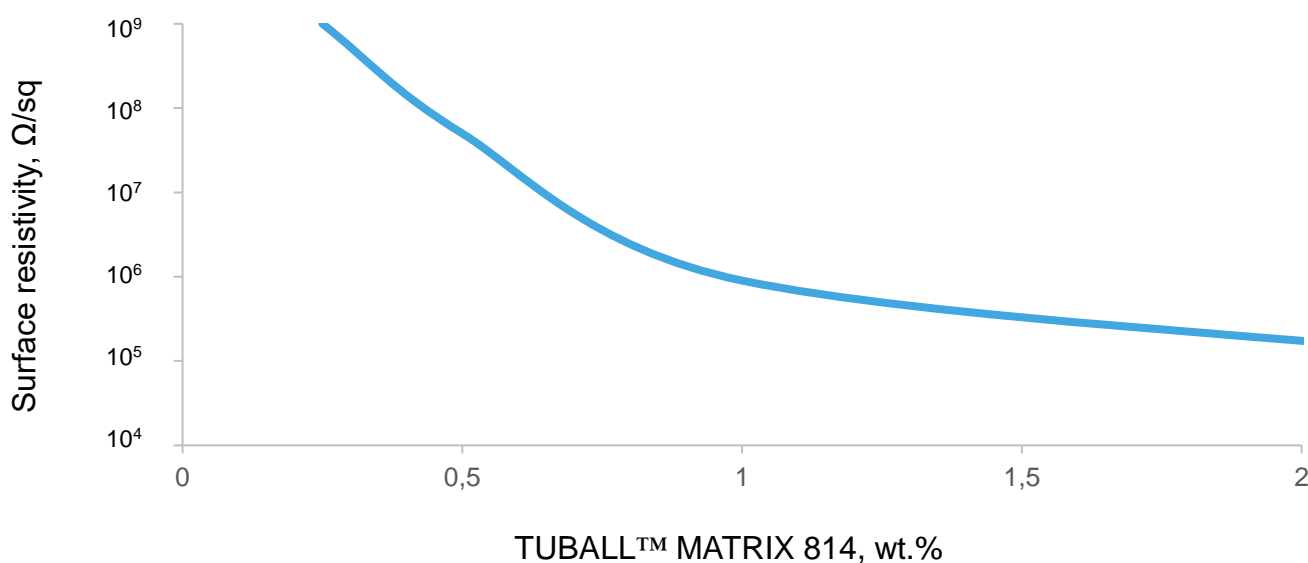
Use the percolation curve (Figure 3) to determine the initial target dosage of TUBALL™ MATRIX 814 for your formulation. The TUBALL™ MATRIX 814 dosage should be calculated according to the whole PVC plastisol formulation.

Table 1. Example of TUBALL™ MATRIX dosage for compounding

Recommended TUBALL™ MATRIX 814 starting dosage	Target surface resistivity*
0.5 wt.%	10^7 – 10^9 Ω /sq
1.0 wt.%	10^6 – 10^7 Ω /sq

* OCSiAl internal PVC plastisol formulation, ASTM D257

Figure 3. The dependence between TUBALL™ MATRIX 814 dosage and surface resistivity*.



* OCSiAl internal PVC plastisol formulation, ASTM D257.

STEP 2. Adding of TUBALL™ MATRIX

Add the calculated TUBALL™ MATRIX 814 dosage into the PVC plastisol and apply the mixing procedure described in Step 3. If inorganic fillers are required in the formulation it is recommended to add them after Step 4 and incorporate them at moderate mixing speed.

Note: the temperature of PVC plastisol and TUBALL™ MATRIX 814 should be higher than 15°C as TUBALL™ MATRIX 814 can be solid at lower temperature.

STEP 3. Mixing

Mix the system with a peripheral speed of 3.5-10 m/s during 10 to 20 minutes.

The dependence between the peripheral and shaft speed is shown below.

$$V = \frac{\pi \cdot d \cdot N}{6 \cdot 10^4}$$

V – Peripheral speed [m/s] N – Shaft speed [rpm]
 d – Blades diameter [mm] π – 3.14

The dependence between shaft speed and diameter of impeller blade to achieve the recommended peripheral speed of 7 m/s is shown below in Table 2.

Table 2. The dependence between shaft speed and diameter of impeller blade

	Peripheral speed, 7 m/s									
Diameter, mm	50	100	150	200	250	300	350	400	450	500
Shaft speed, rpm	2675	1340	890	700	535	445	380	335	300	270

STEP 4. Quality control

Check the quality of the dilution (see “Quality Control” section).

STEP 5. Adding of other components

If necessary, add the other relevant components of your formulation. After adding each component, it is necessary to mix the system until it is homogeneous.

STEP 6 (optional). Vacuum degassing

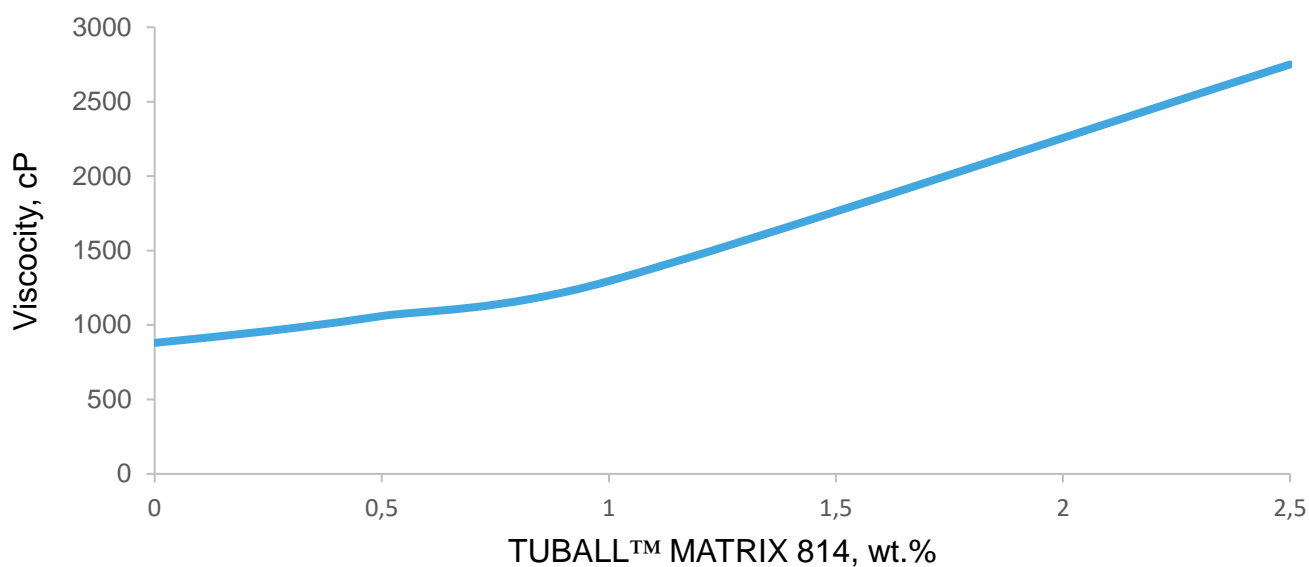
Apply the vacuum degassing procedure.

Note: the shelf life of the final compound must be determined experimentally for each particular PVC plastisol compound.

VISCOSITY MANAGEMENT

Adding TUBALL™ MATRIX 814 leads to an increase in the viscosity of the formulation. Figure 4 shows the typical increase in viscosity resulting from the introduction of TUBALL™ MATRIX 814 in a PVC plastisol.

Figure 4. Influence of TUBALL™ MATRIX 814 dosage on viscosity



* Brookfield viscosity measured at 25°C using viscometer DV2T with spindle RV-06 and spindle speed 50 rpm.
OCSiAl internal PVC plastisol formulation.

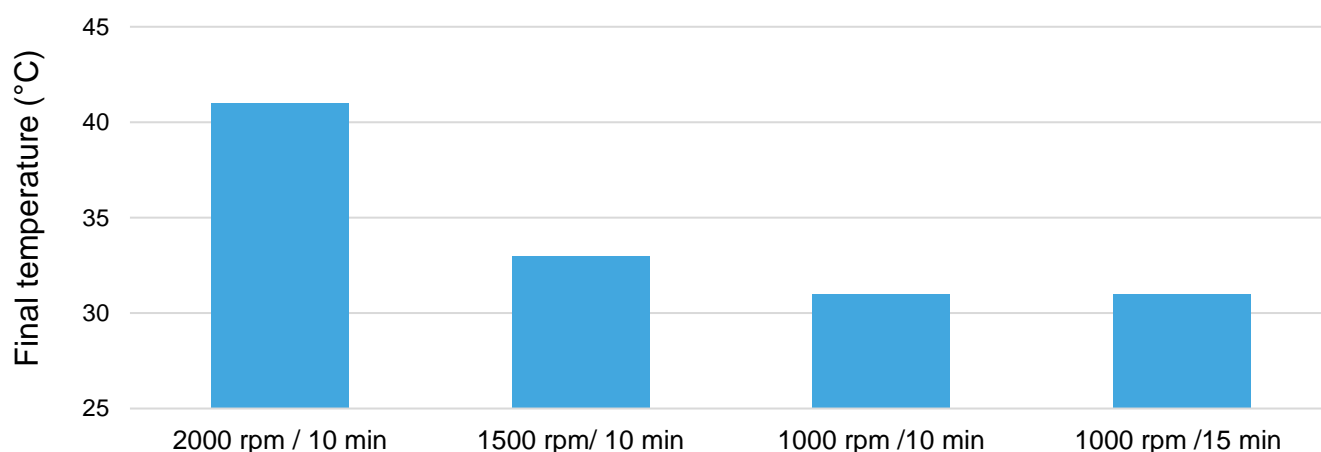
The optimization of TUBALL™ MATRIX 814 dosage is one of key tool to regulate the viscosity.

MIXING TEMPERATURE CONTROL

Depending on customer formulation and equipment utilized, the dispersion of TUBALL™ MATRIX 814 into a plastisol formulation can result in an increase of the temperature that in some cases can induce premature gelation of the formulation. In this case it is important to adjust the mixing conditions in order to avoid heat build-up during the dispersion phase.

The main parameter affecting the heat generation is the mechanical energy introduced by the dispersing equipment. A set of experiments has been conducted starting from a formulation selected to show significant temperature increase at 2000 rpm (7 m/s) reaching 41°C after 10 minutes mixing. Figure 5 indicates that reduction of the mixing speed can alleviate this heat build-up. All samples from this experiment show after filler/pigment introduction and curing similar surface resistivities in the range $6 \cdot 10^5$ – $1 \cdot 10^6$ Ohm indicating that good dispersions of TUBALL™ MATRIX 814 were achieved in all cases.

Figure 5. Plastisol final temperature upon 1.7 wt.% TUBALL™ MATRIX 814 incorporation according to different mixing conditions.



A separate experiment has shown that a one-step mixing of the end formulation ingredients at reduced speed can be envisaged as an alternative to the multiple step approach hereabove. It results in a decrease of final temperature of 10°C and in similar resistivities after curing. This one step approach was achieved by mixing at low speed (1000 rpm) TUBALL™ MATRIX 814 with the liquid ingredients of formulation, then adding the PVC and finally incorporating the filler/pigment then mixing the resulting liquid for 15 minutes (total mixing time is 18 minutes).

QUALITY CONTROL

Quality control should be conducted after dilution stage. The quickest and easiest method of examining the dilution quality is to take tip samples with a glass or plastic stick and then to flatten the sample into a thin layer on a white sheet of paper (Figure 6). If non-uniformities are present (Figure 7), continue stirring until another sample shows that complete dispersion has been achieved (Figure 8).

Figure 6. Quality control procedure

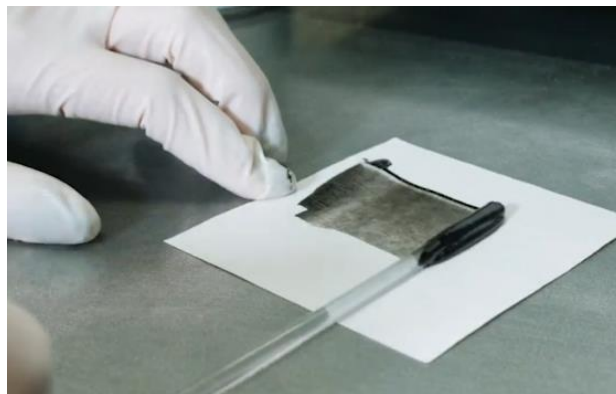


Figure 7. “Bad” quality dispersion (many large particles of TUBALL™ MATRIX 814)

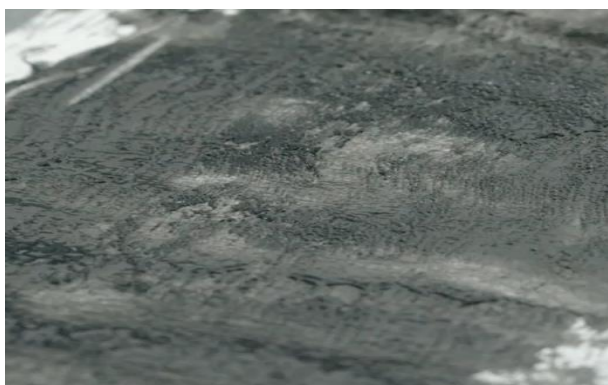


Figure 8. “Good” quality dispersion (homogeneous mixture)



Dispersion quality can be evaluated according to ISO 1524. After TUBALL™ MATRIX 814 complete dilution the fineness of grind level should be less than 15 μm (Figure 9) for a PVC plastisol-based formulations with a filler having size less than 15 μm .

Consult the instructions for your specific model of grindometer to conduct a measurement.

Figure 9. “Good” quality dispersion (particle size $\leq 15 \mu\text{m}$)



EXAMPLES OF FORMULATIONS AND PROCESSING

Target level of resistivity is 10^7 – 10^9 Ω/sq

COMPOSITION OF FINAL COMPOUND (1 kg)	INDUSTRIAL EXAMPLES
50.0% of PVC powder (500 g)	LG PB1302
35.5% of plasticizer (355 g)	Hexamol Dinch (BASF)
10.0% of filler (100 g)	CaCO ₃ (20 μm)
2.5% of stabiliser (25 g)	Lastab Ca/Zn
0.5% of de-aerator(5 g)	BYK–3550
0.5% of color paste (5 g)	Pigment so-strong green
1.0% of conductive additive (10 g)	TUBALL™ MATRIX 814

Note: all components and TUBALL™ MATRIX 814 should be at more than 15°C before being mixed.

DILUTION PROCEDURE

- Mix plasticizer and all liquid component (stabilizer, de-aerator) to homogeneous stage;
- Add PVC powder mix to homogeneous stage;
- Add filler and colour paste, mix to homogeneous stage;
- **Add TUBALL™ MATRIX 814 and mix 3.5-10 m/sec during 10–15 min;**
- Check the dilution quality;
- Apply degassing procedure (optional);
- Cast into the mold;
- Put in drying oven 200°C for 5 min.

WARRANTIES AND DISCLAIMER

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