TUBALL™ MATRIX

PROCESSING GUIDE

FOR CAST POLYURETHANES AND COATINGS
TUBALL™ MATRIX 202/209

Recommended Equipment
Principles
Procedure
Viscosity Management
Quality Control

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.

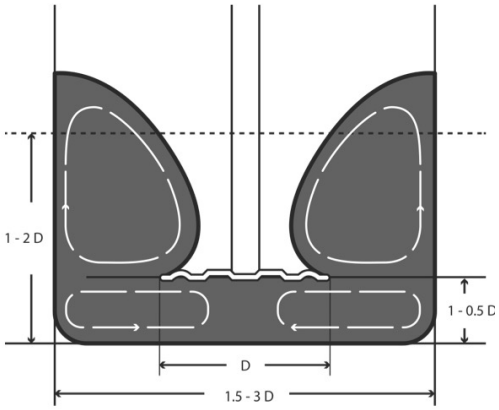
Dilution should be conducted in a cylindrical mixing container with a flat bottom.

Figure 1. Recommended impeller blade shape.

PRINCIPLES

- The temperature, time and mixing speed may need to be adapted to obtain a final mixture that is homogeneous.
- Increasing the rotation speed is a more effective way to obtain better dispersion quality than increasing the mixing time. The recommended peripheral speed is 10 m/s; the speed should not exceed 15 m/s, otherwise resistivity may increase.
- During the dilution process, check the impeller blade and the walls and bottom of the container for stuck masses of TUBALL™ MATRIX and reintroduce them if needed.

Figure 2. The optimal relative position of the stirrer, container and mixed volume.



- For best results, add the curing agents and polymerise the compound containing TUBALL™ MATRIX as soon as possible after diluting the TUBALL™ MATRIX. When dispersed compound, single wall carbon nanotubes tend to agglomerate over time. This process is reversable by dispersing again.
- The shelf life of the final compound in the liquid state must be determined experimentally for each particular compound.

PROCEDURE

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

Uniform distribution of TUBALL™ MATRIX in the resin 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.

Figure 3. TUBALL™ MATRIX appearance

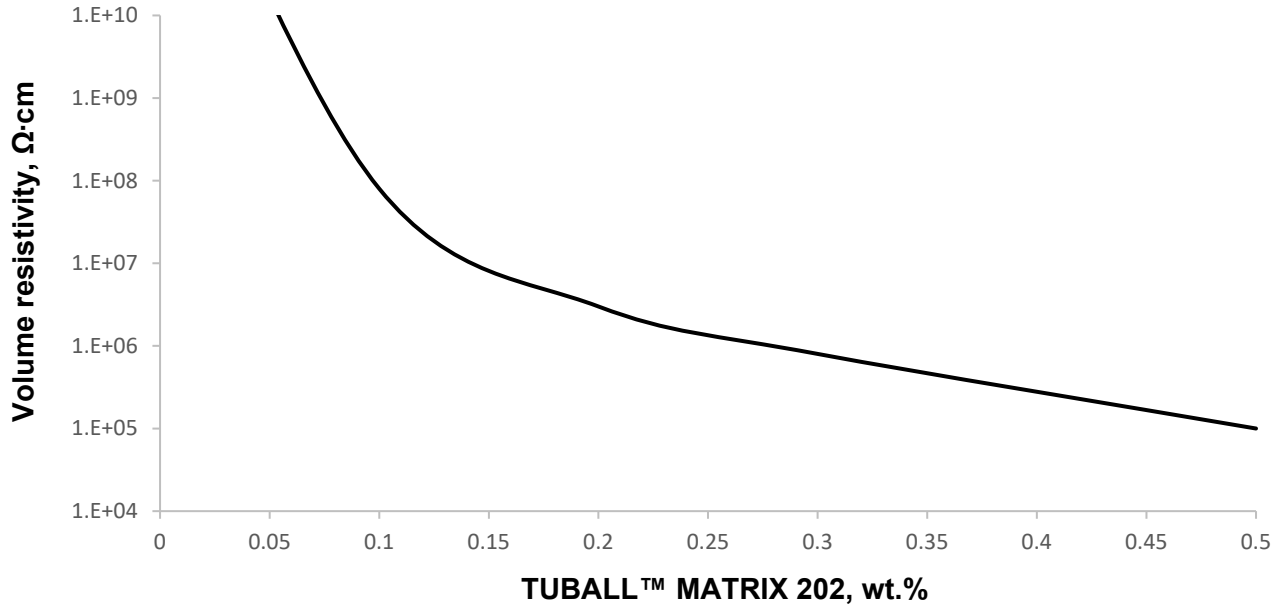
A video about the dilution procedure is available at **www.ocsial.com/en/products/tuball-matrix/**.

STEP 1

Use the percolation curve to determine the target dosage of TUBALL™ MATRIX for your formulation. The TUBALL™ MATRIX dosage should be calculated according to non-volatile matter.

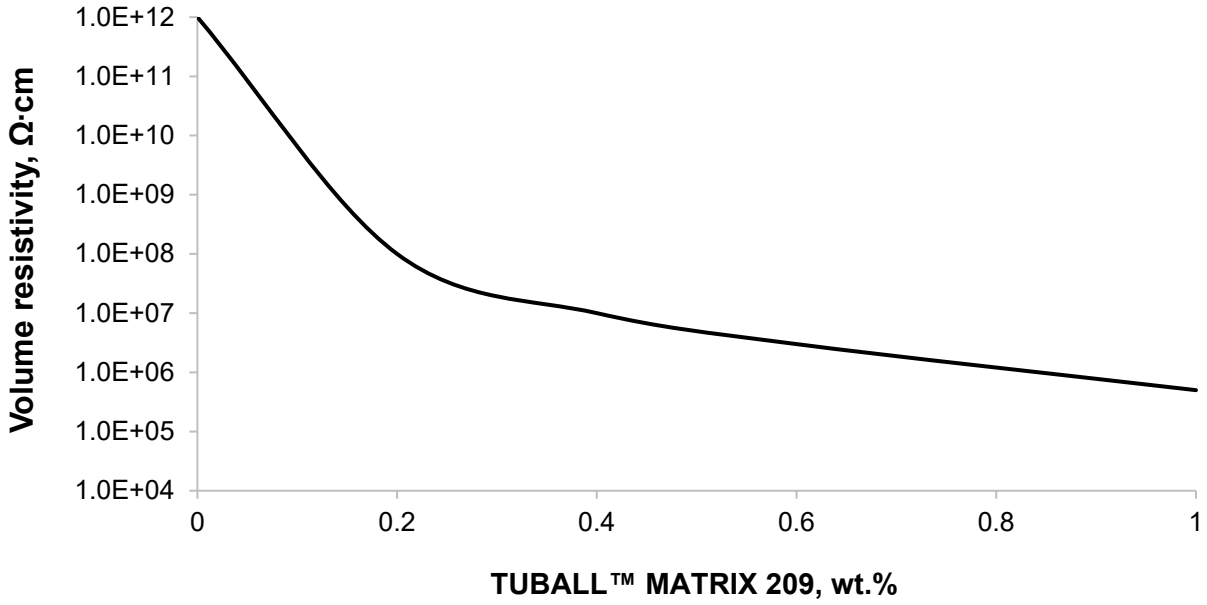
Recommended starting dosage	Target resistivity
0.2 wt.% TUBALL™ MATRIX 202 or 0.4 wt.% TUBALL™ MATRIX 209	10^9–10^7 Ω·cm
0.4 wt.% TUBALL™ MATRIX 202 or 0.8 wt.% TUBALL™ MATRIX 209	10^6–10^5 Ω·cm

Figure 4. The dependence between TUBALL™ MATRIX 202 dosage and volume resistivity*.



* OCSiAl internal PU formulation, ASTM D257.

Figure 5. The dependence between TUBALL™ MATRIX 209 dosage and volume resistivity*.



* OCSiAl internal PU formulation, ASTM D257.

STEP 2

Add the calculated TUBALL™ MATRIX dosage in the necessary component.

TYPE OF PU SYSTEM	Component for preliminary TUBALL™ MATRIX mixing
Prepolymer **Hardener** **Chain extender**	Prepolymer (heated to recommended processing T°C) Or non-isocyanate reactive component
Oligomer (Quasi-prepolymer) **Hardener** **Chain extender**	Oligomer Or non-isocyanate reactive component
Polyol **Isocyanate** **Chain extender**	Polyol

STEP 3

TUBALL™ MATRIX 202: mix the system with 7-15 m/s during 10 minutes.

NOTE: Do not mix the system more than 15-20 minutes. It can lead to the sufficient heating and TUBALL™ MATRIX flocculation.

TUBALL™ MATRIX 209: mix the system with 4-5 m/s during 15 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 10 m/s is shown below.

	Peripheral speed, 10 m/s									
DIAMETER, mm	50	100	150	200	250	300	350	400	450	500
SHAFT SPEED, rpm	3820	1910	1270	950	760	640	540	480	420	380

STEP 4

Check the quality of the dilution using the quality control procedure in the “Quality Control” section.

STEP 5

Apply the vacuum degassing procedure: mix the system for 5 minutes at 3 m/sec using vacuum.

STEP 6

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

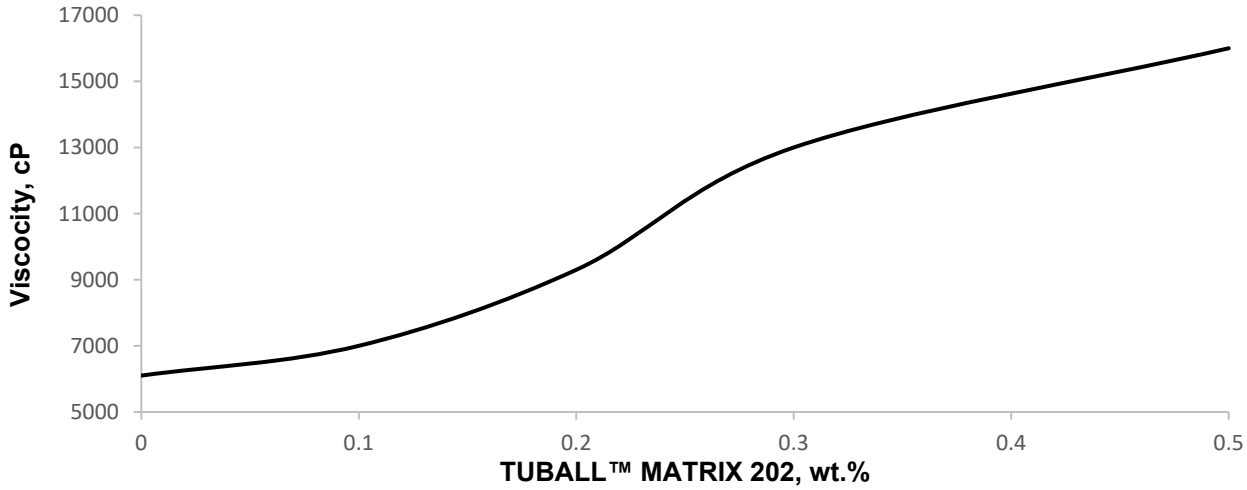
STEP 7

Add the curing agent (accelerator or catalyst) to polymerize the system.

VISCOSITY MANAGEMENT

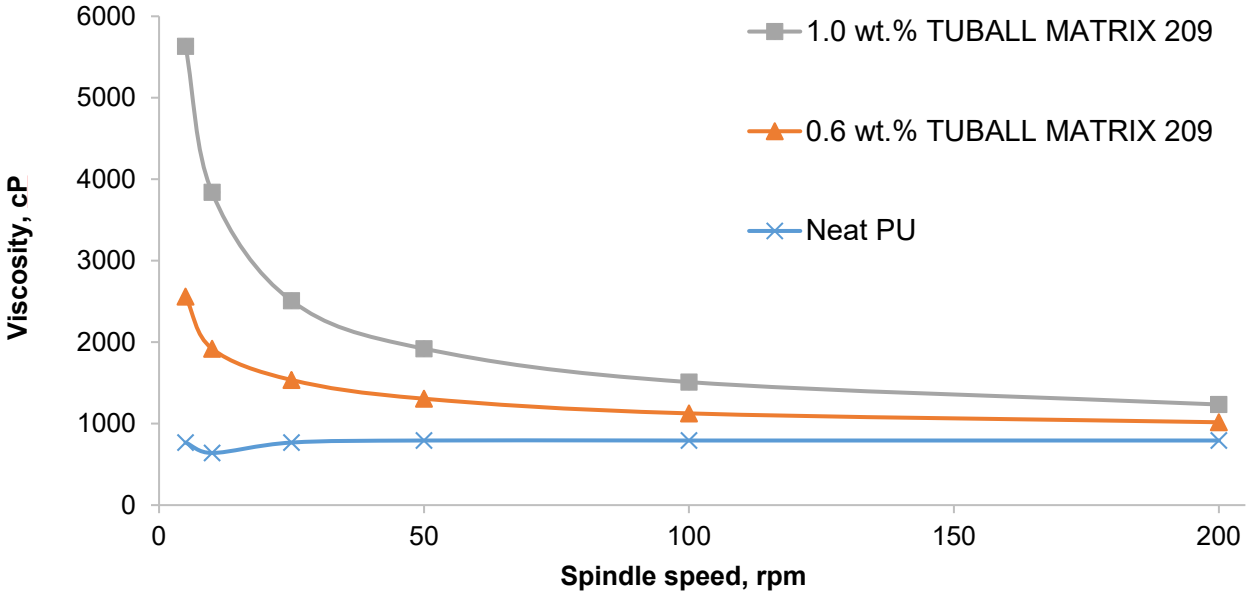
Adding TUBALL™ MATRIX leads to an increase in the viscosity of the formulation. Figure 6 and 7 show the typical increases in viscosity resulting from the application of TUBALL™ MATRIX concentrate.

Figure 6. Viscosity rate with TUBALL™ MATRIX 202



* Brookfield viscosity measured at 75°C using viscometer DV2T with spindle RV-07 and spindle speed 50 rpm. Tested in MDI-based PU system (85 Shore A).

Figure 7. Viscosity rate with TUBALL™ MATRIX 209



* Brookfield viscometer DV2T, spindle RV-06. Tested in OCSiAl internal PU formulation

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

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 8). If non-uniformities are present (Figure 9), continue stirring until another sample shows that complete dispersion has been achieved (Figure 10).

Figure 8. Quality control procedure

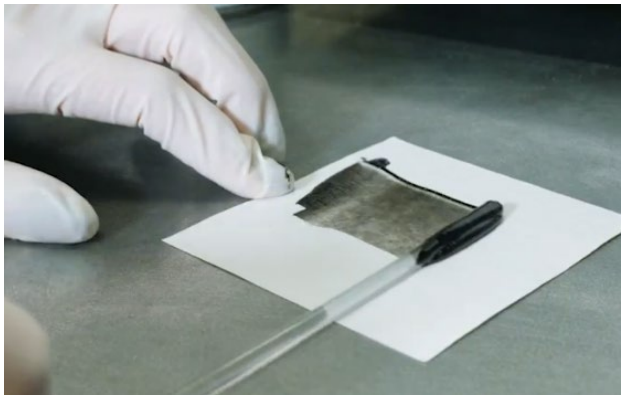


Figure 9. "Bad" quality dispersion (many large particles of TUBALL™ MATRIX)

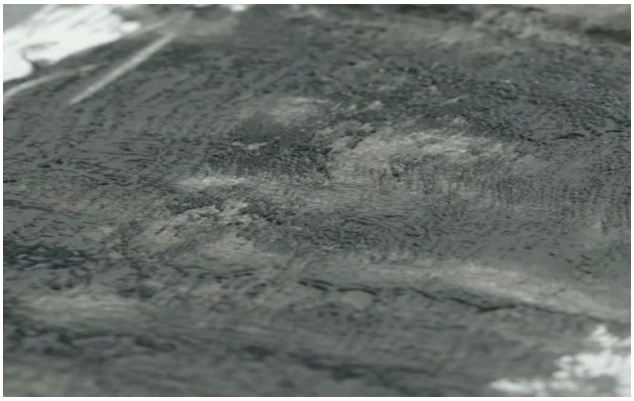
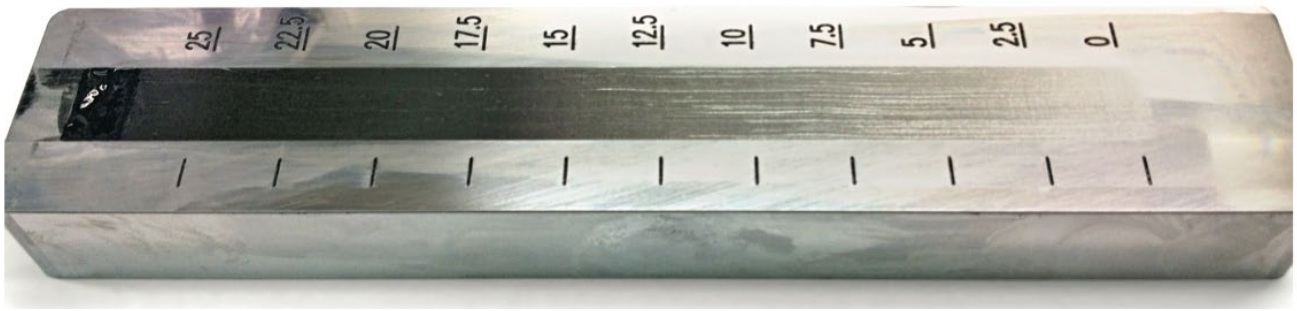


Figure 10. "Good" quality dispersion (homogeneous mixture)

Dispersion quality can be evaluated according to ISO 1524. After the second stage of TUBALL™ MATRIX dilution the fineness of grind level should be less than 15 μm (Figure 11).

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

Figure 11. "Good" quality dispersion (particle size ≤ 15 μm)



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