



PROCESSING GUIDELINES

TUBALL™ MATRIX 200-x and 300-x series

Recommended Equipment
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Quality Control
Troubleshooting

RECOMMENDED EQUIPMENT

For laboratory tests: a mechanical overhead stirrer with an optimised impeller blade configuration should provide the peripheral speed in the range of 7-15 m/sec (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 target dosage of TUBALL™ MATRIX refers to the loading in the whole final formulation. For example, if the system consists of 50 wt.% resin and 50 wt.% fiber, and the required level of volume resistivity is $10^6 \Omega \cdot \text{cm}$, the target TUBALL™ MATRIX dosage should be 0.5 wt.% for the whole system and 1.0 wt.% for the resin.
 - 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.
- Figure 2.** The optimal relative position of the stirrer, container and mixed volume.
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- 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 in a thermoset compound, single wall carbon nanotubes tend to agglomerate.
 - 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 from the Technical Data Sheet 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.% | $10^8 - 10^6 \Omega \cdot \text{cm}$ |
| 0.5 wt.% | $10^6 - 10^4 \Omega \cdot \text{cm}$ |

STEP 2

Use a two-stage dilution technique to dilute TUBALL™ MATRIX in the base resin.

For solvent-based systems: it is also possible to add TUBALL™ MATRIX in the system with solvent (e.g. paint, lacquer, primer, etc.). Do not dilute TUBALL™ MATRIX in the neat solvent due to weak shear force. If the final formulation consists of resin with solvent, to dilute TUBALL™ MATRIX in neat resin and after that add relevant quantity of solvent.

| | Stage description | Mixing conditions |
|----------------|---|-------------------|
| STAGE 1 | Mix the total required quantity of TUBALL™ MATRIX with the base resin; the ratio of TUBALL™ MATRIX : resin should be 1 : 49 | 7-15 m/s, 20 min |
| STAGE 2 | Add the remaining quantity of the base resin according to your formulation | 7-15 m/s, 5 min |

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]
d – Blades diameter [mm]

N – Shaft speed [rpm]
n – 3.14

The example of 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 3

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

STEP 4

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 5 (optional)

Mix the system at 100–200 rpm for 5 minutes (defoaming step). OCSiAl also recommends that the mixture be left to stand for a period of time before adding the curing agent. This will help the rest of the air bubbles to leave the mixture.

STEP 6

Add the curing agent (accelerator or catalyst) to polymerise the system, if necessary.

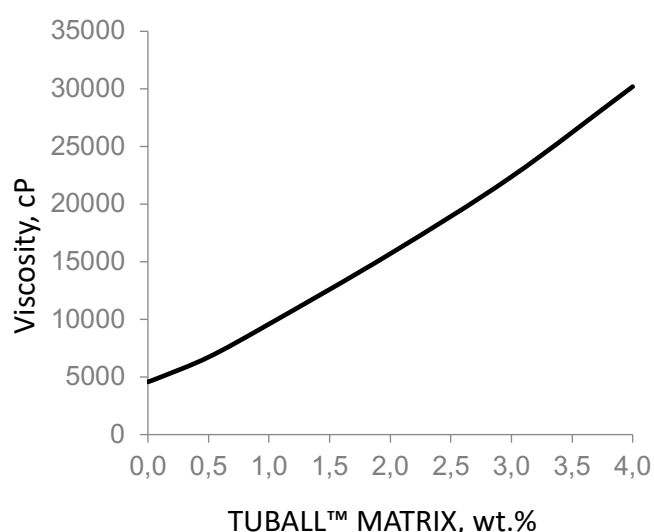
VISCOSITY MANAGEMENT

Adding TUBALL™ MATRIX leads to an increase in the viscosity of the formulation. Figure 4 shows the typical increase in viscosity resulting from the application of TUBALL™ MATRIX concentrate.

Methods for reducing viscosity:

- reduce thixotropic and thickening agents
- add levelling additives (e.g. 0.3–0.5 wt.% of BYK-306 based on total formulation)
- add viscosity reducing additives (e.g. 0.5–1.5 wt.% of BYK-W 980 based on fillers, or solvent or reactive diluent),
- heat the mixture if allowed (up to 40°C).

Figure 4. Viscosity rate with TUBALL™ MATRIX*



* Brookfield viscosity measured at 25°C, spindle SC4-6, 20 rpm.

Tested in D.E.R. 351 epoxy resin. Measurements conducted according to ASTM D2983.

QUALITY CONTROL

Quality control should be conducted after each 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 5). If non-uniformities are present (Figure 6), continue stirring until another sample shows that complete dispersion has been achieved (Figure 7).

Figure 6. “Bad” quality dispersion (many large particles of TUBALL™ MATRIX)

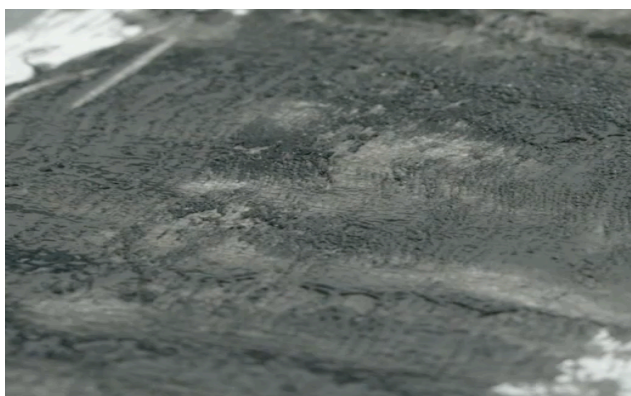


Figure 5. Quality control procedure

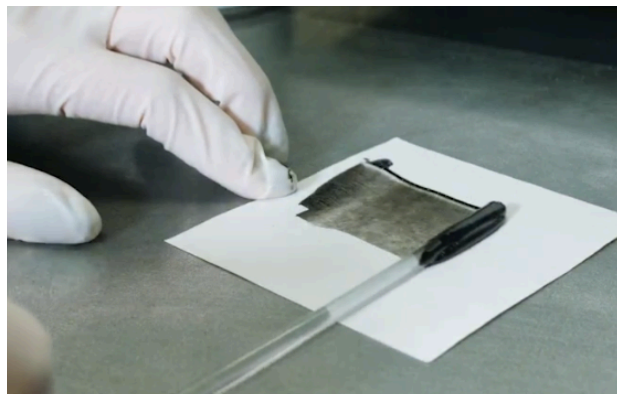


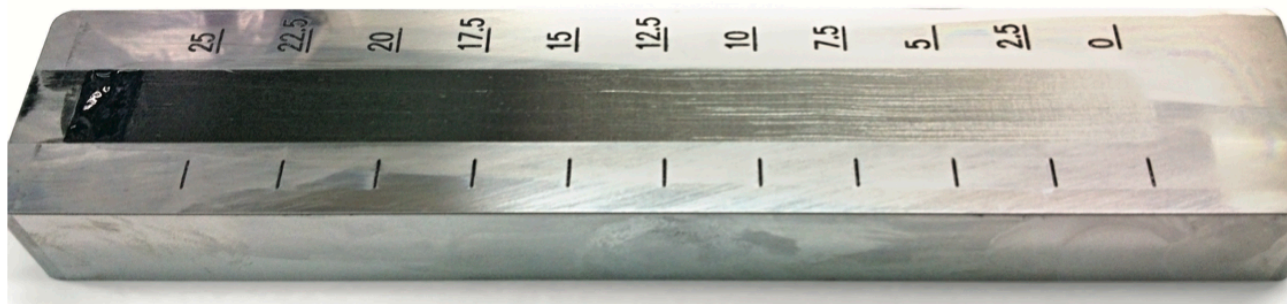
Figure 7. “Good” quality dispersion (homogeneous mixture)



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 8).

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

Figure 8. “Good” quality dispersion (particle size $\leq 15 \mu\text{m}$)



TROUBLESHOOTING

| PROBLEM | POSSIBLE SOLUTION |
|--|--|
| Lack of resistivity | increase TUBALL™ MATRIX dosage by 0.1 wt.% |
| High level of viscosity | add reactive diluent |
| | add solvent |
| | heat the mixture if allowed (up to 40°C) |
| | reduce amount of thixotropic and thickening agents |
| Unsatisfactory dilution (large particles of TUBALL™ MATRIX remaining) | use impeller blade suitable for high-viscosity mixing |
| | increase mixing time |
| | increase temperature of mixing |
| | increase rotation speed |
| Many bubbles Foaming | add deaerating/antifoaming agent |
| | increase time of defoaming step |
| | apply vacuum degassing procedure |
| | use deaerating impeller such as Visco Jet |
| Flocculation | use dispersing agents such as: <ul style="list-style-type: none"> DISPERBYK-2152 (for solvent-free systems) DISPERBYK-2155 (for solvent-based systems) the dosage should be around 0.1–0.5 wt.% based on total formulation |
| Poor self-levelling in flooring applications | add reactive diluent or solvent |
| | add levelling additives (e.g. 0.3–0.5 wt.% of BYK-306 based on total formulation) |
| | Add viscosity-reducing additives (e.g. 0.5–1.5 wt.% of BYK-W 980 based on fillers) |
| | remove or reduce thixotropic and thickening agents |
| Colour too dark | add white pigment (such as TiO ₂ , etc.) |
| Appearance of white pigment on the surface | add dispersing agent for white pigment (e.g. DISPERBYK-110 for TiO ₂) |
| Sticking material on impeller blade | clean out the mixing box |
| | mix for up to an additional 15 minutes |