TUBALL™ MATRIX

PROCESSING GUIDE

TUBALL™ MATRIX 200-x and 300-x series

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TUBALL™ MATRIX

CONTENT

1. INTRODUCTION 2

2. TUBALL™ MATRIX DILUTION WITH HIGH-SPEED MIXER 3

3. TUBALL™ MATRIX DILUTION WITH ROTOR-STATOR MIXER 5

4. QUALITY CONTROL 6

5. VISCOSITY MANAGEMENT 8

6. TROUBLESHOOTING 8

1. INTRODUCTION

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 1. TUBALL™ MATRIX appearance.



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.

To define the product performance in the certain formulation following dosages are proposed for the starting trials.

Recommended starting dosage	Target resistivity
0.2 wt.%	10^6–10^8 Ω·cm
0.5 wt.%	10^4–10^6 Ω·cm

The following equipment is recommended for TUBALL™ MATRIX dilution:

- a high-speed mixer with a cowles mixing blade,
- a rotor-stator mixer.

2. TUBALL™ MATRIX DILUTION WITH HIGH-SPEED MIXER

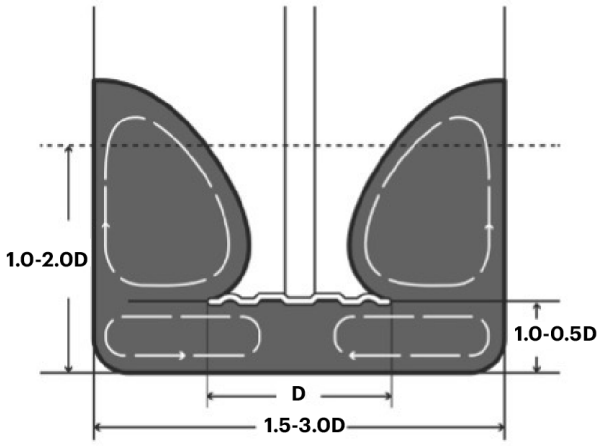
2.1. PRINCIPLES OF A HIGH-SPEED MIXER USAGE

- For laboratory tests and industrial production: a mechanical overhead stirrer or a dissolver with an optimized impeller blade configuration should provide the peripheral speed in the range of 7–15 m/sec.
- 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.
- Dilution should be conducted in a cylindrical mixing container with a flat bottom.
- During the dilution process, check the impeller blade and the walls and bottom of the container for stuck masses of TUBALL™ MATRIX.
- For best results, add the curing agents and polymerize 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.

Figure 2. Recommended impeller blade shape.



Figure 3. The optimal relative position of the stirrer, container, and mixed volume.



2.2. PROCEDURE OF TUBALL™ MATRIX DILUTION WITH HIGH-SPEED MIXER

A video about the dilution procedure is available:
TUBALL™ MATRIX - Processing guide

STEP 1

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]
π – 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	3,820	1,910	1,270	950	760	640	540	480	420	380

STEP 2

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

STEP 3

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 4 (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 5

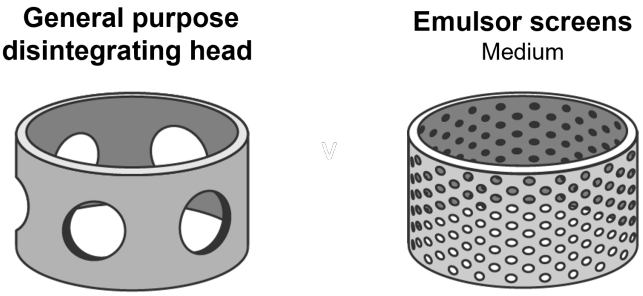
Add the curing agent (accelerator or catalyst) to polymerize the system, if necessary.

3. TUBALL™ MATRIX DILUTION WITH ROTOR-STATOR MIXER

3.1. PRINCIPLES OF A ROTOR-STATOR MIXER USAGE

- Rotor-stator mixers could be used for TUBALL™ MATRIX dilution: a good dispersion quality and the electrical performance could be achieved.
- There is a basic limitation of the liquid viscosity depending on the neat resin viscosity (e.g., <15 000 cP) and TUBALL™ MATRIX dosage.
- The design of mixing head has a direct impact on material flux. E.g., screen system with too small holes could negatively affect on mixing time because material flux will be limited or prevented.
- The mixing head should be reconfirmed individually in each case. Recommended starting heads are shown in Figure 4.
- Temperature increase can take place in a very short time. This should be taken into account for °T sensible systems.

Figure 4. Recommended starting heads of a rotor-stator mixer.



3.2. PROCEDURE OF TUBALL™ MATRIX DILUTION WITH ROTOR-STATOR MIXER

STEP 1

1 dilution step is recommended. Mixing conditions (speed, time) should be clarified individually.

Recommended starting conditions:

- Dilution time from 2–5 minutes
- Speed for laboratory trials 6,000 rpm
- Speed for production 3,000 rpm

STEP 2

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

STEP 3

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 4 (optional)

Mix the system 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 5

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

4. QUALITY CONTROL

Following methods could be used for the quality control of diluted TUBALL™ MATRIX products:

- Visual analysis (express test);
- Grindometer test (ISO 1524);
- Optical microscopy;
- Electrical resistivity measurement.

A video of the quality control methods demonstration is available:

[How to evaluate the dilution quality of dispersed graphene nanotube in thermosets]

4.1. QUICK VISUAL ANALYSIS

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 5. Quality control procedure

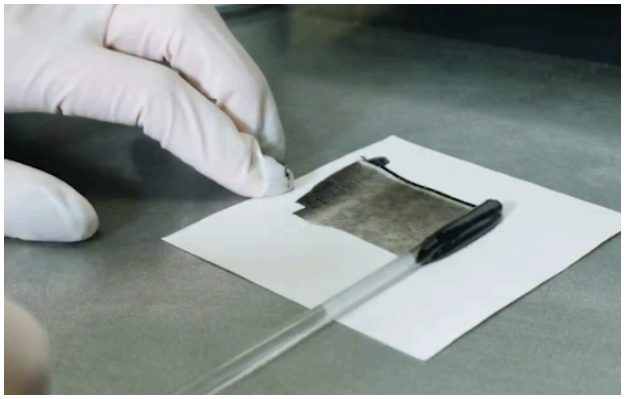


Figure 6. Poor quality dispersion (many large particles of TUBALL™ MATRIX)

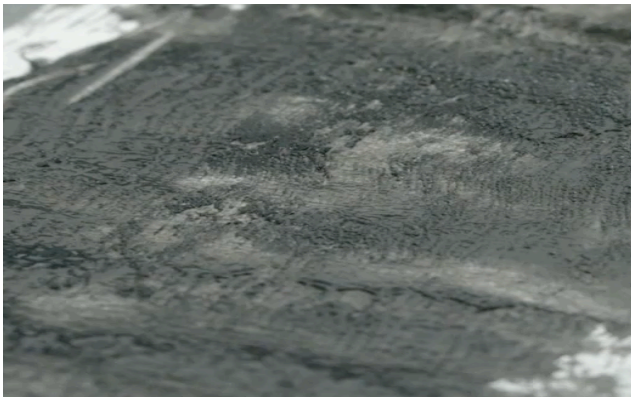


Figure 7. Good quality dispersion (homogeneous mixture)

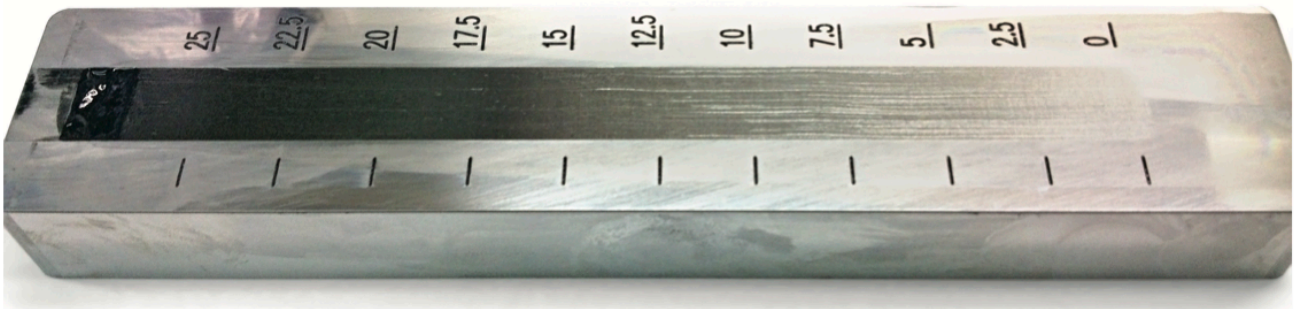


4.2. GRINDOMETER TEST (ISO 1524)

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 ≤ 15 μm)



4.3. OPTICAL MICROSCOPY

To estimate the dilution quality using optical microscope, prepare a sample first. Prepare a glass slide with double-sided tape. Use a glass rod to apply a droplet of the mixture to the glass slide. Then put a coverslip or second glass slide on top and gently press to spread the droplet.

Images can be taken at 50x, 100x and 400x magnification to analyse the number, size, and shape of agglomerates.

Examples of well and poorly diluted TUBALL™ MATRIX can be found in Figure 9 and Figure 10. While no residual significant black domains of TUBALL™ MATRIX are seen in the well diluted formulation, a high number of black agglomerates of undiluted concentrate can be seen when the processing conditions are not optimized.

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

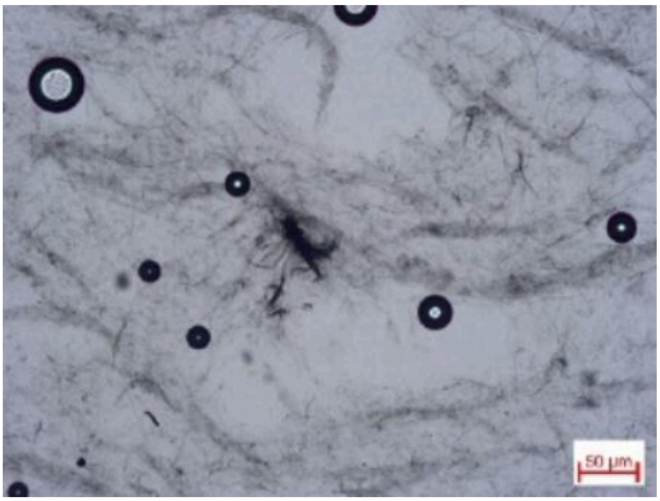
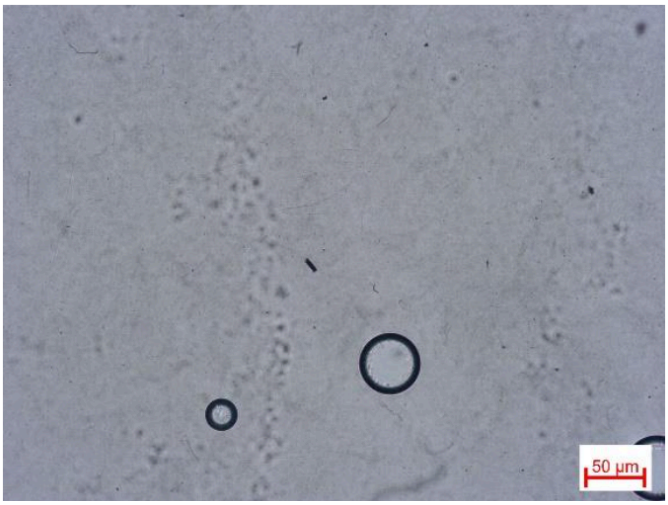


Figure 10. Good quality dispersion (homogeneous mixture)



4.4. ELECTRICAL RESISTIVITY MEASUREMENT

To identify appropriate method, OCSiAl video guide is available:

Electrical resistivity guide: measurement, standards & troubleshooting

To get the full document version of the electrical resistivity measurement guide, contact your local OCSiAl representatives: https://tuball.com/contacts.

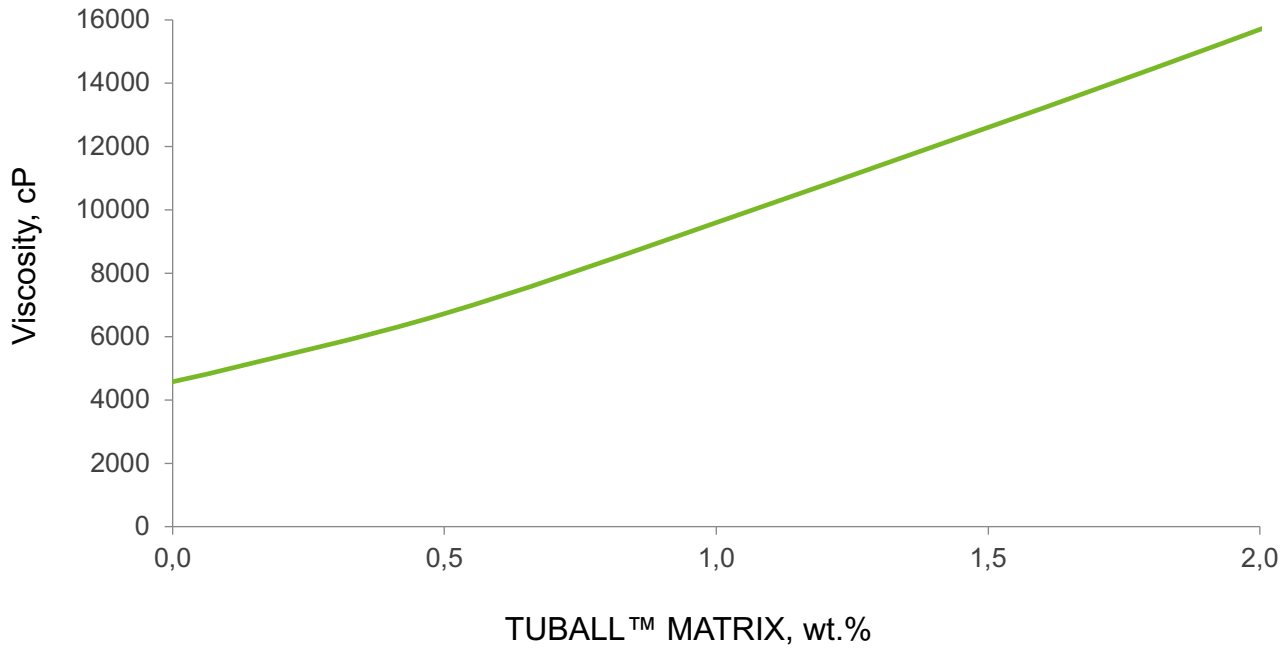
5. VISCOSITY MANAGEMENT

Adding TUBALL™ MATRIX leads to an increase in the viscosity of the formulation. Figure 11 shows the typical increase in viscosity resulting from the application of TUBALL™ MATRIX concentrate.

More practical information about viscosity management is presented in troubleshooting section.

Brookfield viscosity measured at 25°C, spindle SC4-6, 20 rpm. Tested in epoxy resin. ASTM D2983.

Figure 11. Viscosity rate with TUBALL™ MATRIX*



6. TROUBLESHOOTING

PROBLEM	POSSIBLE SOLUTION
Lack of resistivity, Unsatisfactory dilution (large particles of TUBALL™ MATRIX remaining)	Check mixing configuration; Increase mixing time, speed; Increase TUBALL™ MATRIX dosage at least by 0.1 wt.%.
High level of viscosity	Add reactive diluent, solvent; Heat the mixture if allowed; Reduce amount of thixotropic and thickening agents; Add viscosity reducing additives. Starting recommendations below: • epoxy systems: BYK–W 980, • polyurethane systems: BYK–P 9915, • vinyl ester systems: BYK–W 908, BYK–W 980, BYK–W 966, • various solvent-free systems: CGPS 277B, DISPERBYK–2014, • various water-based systems: DISPERBYK–2012, DISPERBYK–2013, DISPERBYK–2015 and DISPERBYK–190.

Many bubbles Foaming	Add deaerating/antifoaming agent. Starting recommendations below: • epoxy systems: BYK–088, BYK–A 530, BYK–054, BYK–066, TEGO Airex 900, • polyurethane systems: BYK–088, BYK–054, BYK–066, TEGO Airex 900, • vinyl ester systems: BYK–A 515, BYK–A 555, • various water-based systems: TEGO Airex 901 W; Increase time of defoaming step; Apply vacuum degassing procedure.
Flocculation	Use dispersing agents. Starting recommendations below: • epoxy systems: DISPERBYK–2152, DISPERBYK–2155, DISPERBYK–180, • polyurethane systems: DISPERBYK–2150, DISPERBYK–2155, • vinyl ester systems: TEGOMER DA 646, BYK–9076, DISPERBYK–2013, DISPERBYK–2014, • various solvent-based systems: DISPERBYK–118, DISPERBYK–2014, • various water-based systems: DISPERBYK–2014.
Poor self-levelling	Add reactive diluent or solvent; Add viscosity-reducing additives; Remove or reduce thixotropic and thickening agents; Add levelling additives. Starting recommendations below: • epoxy systems: BYK–306, BYK–3550, • vinyl ester systems: BYK–361 N, Dynasylan MEMO, BYK–333.
Color 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)

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