TUBALL™ COAT_E

# PROCESSING GUIDE

TUBALL™ COAT_E $H_2O$
Surfactant types: SDBS, DBD, CD and other grades

Equipment
Process
Quality Control
Key principles and troubleshooting
Viscosity Management

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Uniform distribution of TUBALL™ COAT_E in the target formulation plays a key role in enhancing the electrical conductivity of the final system.

**To obtain a high-quality TUBALL™ COAT_E dispersion, pay close attention to the dilution & mixing procedure.**

## COLLOIDAL COMPATIBILITY

For initial trials before processing, the compatibility of TUBALL™ COAT_E with other components of the target system formulation must be determined.

- Slowly add 10 ml of COAT_E $H_2O$ suspension to 90 ml of the final coating liquid formulation. Mix for 10 minutes at 500 rpm in a transparent container.
- After mixing, the mixture should have a gray color, and no visible particles or agglomerates (e.g. settling, inhomogeneity, creaming etc.) should be observed. TUBALL™ COAT_E can then be used in further processing according to procedure below.
- If the system with TUBALL™ COAT_E is not homogeneous, additional compatibility tests of all system components should be conducted step by step. Another grade of TUBALL™-based suspension can be selected as a compatible solution or an incompatible component of the coating should be changed to another alternative.

Please contact your local OCSiAl representatives[1] in case of TUBALL™ COAT_E incompatibility to clarify details.

## 1. EQUIPMENT

**LabScale Evaluation**
A mechanical overhead stirrer is necessary
The optimal impeller shape is a propeller
Or anchor types (Figure 1)
Other impeller types can be also used.

Dilution should be conducted in a cylindrical mixing container.

**Figure 1.** Examples of optimal impeller shape.

Propeller Anchor

[1] tuball.com/contacts

**Industrial Scale Production**

An industrial dissolver is recommended for the optimal mixing of COAT_E $H_2O$ with coating liquid formulation. Recommended impeller shapes are shown in Figure 2. Other impeller types can be also used, such as a blade-type impeller. But for such types of impeller, the speed should be adjusted to avoid shear stress in the suspension.

COAT_E $H_2O$ suspension also can be added to the formulation during other steps such as homogenization using a bead mill or other equipment.

To determine the most suitable and optimal method of introduction in a particular system, please contact your local OCSiAl representative.

**Figure 2.** Examples of optimal dissolver impellers


## 2. PROCESS

Determine the target dosage of TUBALL™ COAT_E for your formulation **according to the required dosage of TUBALL™ in dry coating**.

| Target resistivity | The recommended starting dosages | | | |
|---|---|---|---|---|
| | TUBALL™ content in dry coating | Example: TUBALL™ COAT_E 0.4% dosage depending on the solid content of the target system | | |
| | | 50% | 40% | 30% |
| **$10^8$–$10^6$ Ω/sq** | **0.02–0.04 wt.%** | 2.5–5 wt.% | 2–4 wt.% | 1.5–3 wt.% |
| **$10^6$–$10^4$ Ω/sq** | **0.03–0.05 wt.%** | 3.75–6.35 wt.% | 3–5 wt.% | 2.25–3.75 wt.% |

**STEP 1.** Substitute solvent with an equal part of TUBALL™ COAT_E before introducing the binder to achieve a homogeneous distribution of TUBALL™ in the liquid formulation.

**STEP 2.** Mix the total required quantity of TUBALL™ COAT_E with the binder solution.

| Recommended procedure conditions | |
|---|---|
| **Peripheral speed** | 2–6 m/s |
| **Time** | 15–20 minutes |

**STEP 3.** Check the quality of the dilution according to 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 until it is homogeneous.

! **NOTE:** to avoid colloidal shock, it is possible to mix the system in a different order and add TUBALL™ COAT_E at a different stage of formulation while mixing slowly.

## 3. QUALITY CONTROL

The quickest and easiest method to examine the dilution quality is the visual method. If it is not possible to check dilution quality in the mixed volume, an express glass test can be used.

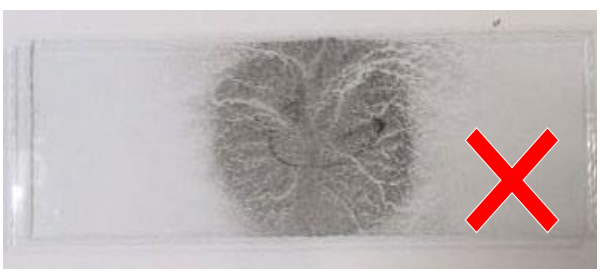
Pour 1 ml of the system with suspension onto a flat glass surface and place another glass plate on top, so that the drop of system is spread across the glass surface.

A good quality dispersion is a homogeneous mixture with uniform color (Figure 3).

If TUBALL™ COAT_E is incompatible with other formulation components or the mixing conditions are not correct, the quality of the dispersion is poor – non-uniform with black particles (Figure 4).

**Figure 3.** Good quality dispersion (homogeneous mixture)

**Figure 4.** Poor quality dispersion (inhomogeneity and many black particles)



# 4. KEY PRINCIPLES AND TROUBLESHOOTING

- The target working dosage of TUBALL™ **depends on the particular formulation and must be determined experimentally**.
- **The speed and duration of mixing are determined for each particular system** and **may need to be adapted** to obtain a final mixture that is homogeneous. Mixing speed limit is determined by the parameters of the target system and should be optimized so that mixing of the entire volume can be achieved.
- **Increasing the mixing speed** is a more effective way to obtain better dispersion quality than increasing the mixing time.
- **The order of mixing of components must be determined** for each particular system.
- Compatibility of TUBALL™ COAT_E with other components of the formulation **must be determined as a result of lab tests before processing**.
- TUBALL™ COAT_E can be applied in the final ready-to-use system. But in order to achieve better results, OCSiAl recommends **optimizing the formulation and procedure before using TUBALL™ COAT_E**.

| PROBLEM | POSSIBLE SOLUTION |
|---|---|
| Lack of conductivity | Increase TUBALL™ COAT_E dosage |
| High level of viscosity | Add TUBALL™ COAT_E before rheological agents |
| | Add diluent or solvent |
| | Reduce amount of rheological agents |
| Low level of viscosity | Optimize the amount of rheological agents |
| Unsatisfactory dilution (inhomogeneity of the system) | Check compatibility of COAT_E with other formulation components |
| | Increase mixing time |
| | Increase mixing speed |
| | Optimize the order of adding components to the formulation |
| Many bubbles<br>Foaming | Add deaerating/antifoaming agent |
| | Mix the final system for 5–10 min at low rotation speed using special deaerating impeller |
| | Apply vacuum degassing procedure (if possible) |

# 5. VISCOSITY MANAGEMENT

## SYSTEM VISCOSITY WITH TUBALL™ COAT_E

TUBALL™ COAT_E leads to an increase in the viscosity of the system.

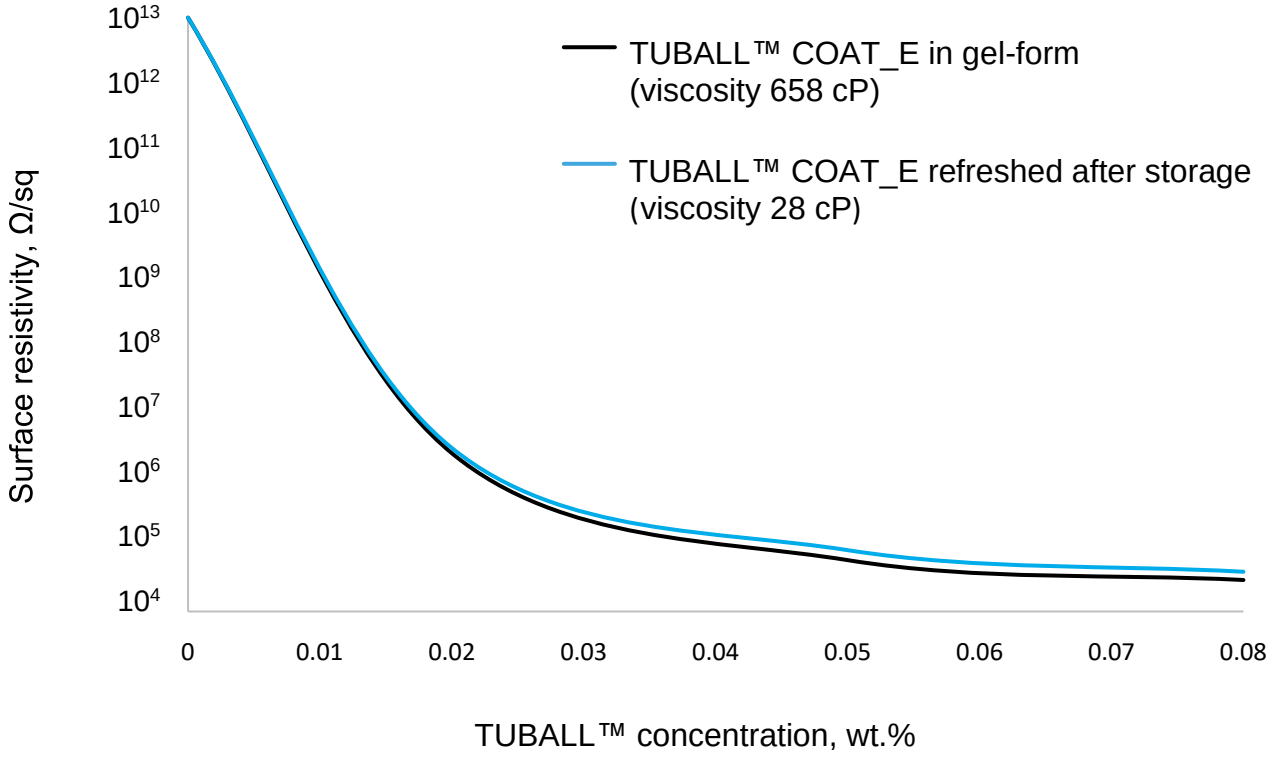
OCSiAl recommends adding rheological agents into the system after TUBALL™ COAT_E if the final viscosity and thixotropic index is not adequate.

## TUBALL™ COAT_E VISCOSITY

TUBALL™ COAT_E suspensions with a concentration of TUBALL™ higher than 0.1% is a highly thixotropic colloidal system that tends to sol-gel transition after manufacturing. As a result, the viscosity of TUBALL™ COAT_E can increase during storage.

The viscosity of TUBALL™ COAT_E after storage can be reduced for the gel-sol transition by applying (stress) shear force. If usage of TUBALL™ COAT_E with a high viscosity is acceptable, it can be applied in gel form with the same performance.

**Figure 5.** The dependence of surface resistivity for TUBALL™ COAT_E in gel form with increased viscosity and after refreshing with decreased viscosity.




* Tested in waterborne polyurethane lacquer system.

** Surface resistivity was measured according to ASTM D257.
Viscosity measured with Brookfield DV3TRV device at 25°C, ULA0 and SC21 spindle, 20 rpm. Measurements according to ASTM D2983.

TUBALL™ COAT_E

## REFRESHING TUBALL™ COAT_E $H_2O$ 0.4% (SDBS) FOR REDUCING VISCOSITY

- Due to its surfactant nature and high TUBALL™ content, TUBALL™ COAT_E $H_2O$ tends to increase in viscosity during storage. If required, TUBALL™ COAT_E $H_2O$ suspension can be refreshed to reduce its viscosity.
- OCSiAl recommends refreshing suspension TUBALL™ COAT_E $H_2O$ 0.4% (SDBS) **in the amount that is required for further use over the next 2 weeks**.
- The refreshed suspension should be used **within 1–3 weeks**.
- In order to prevent foaming, **contact of the suspension with air during refreshing should be minimized**. OCSiAl recommends loading tanks with suspension at maximum volume and removing any foam that may appear during the loading procedure.

### Equipment & Procedure

For reducing the viscosity of the suspension, a shear stress must be applied to TUBALL™ COAT_E $H_2O$. Below are presented recommended methods that can be successfully used:

| Equipment | Procedure |
|---|---|
| **Horizontal Bead Mill** | 1. Turn on the cooling system of the bead mill. Load the beads with size 0.8–1.2 mm in bead mill chamber (volume recommended by bead mill manufacturer ~60–80%).<br>2. Slowly fill the tank with the suspension using a funnel with a long spout that reaches the bottom of the inlet chamber to avoid foaming. It is recommended to load the tank to maximum volume to remove any foam that appears during the loading procedure. Switch on the tank stirrer to 100 rpm.<br>3. Apply 85% of maximal bead mill rotation speed and start circulation.<br>4. Continue bead mill treatment. Total circulation time should be calculated according the ratio $T = 9min*V_{batch}/V_{beadmill}$, where T is circulation time, $V_{batch}$ is the refreshing batch volume, and $V_{beadmill}$ is the beadmill chamber volume.<br>5. Check the viscosity of the batch. If viscosity is below 100 cP, finish the process. Otherwise, add 10% more treatment time and check again.<br>6. Slowly unload the refreshed COAT_E $H_2O$ batch.<br>! **NOTE**: Avoid foaming during circulation (it is preferable to have the circulation tank inlet deep in the liquid batch volume). Viscosity after refreshing must be below 100 cP. |

| | |
|---|---|
| **Rotor Stator High-Shear In-line homogeinzer** | 1. Make sure that the apparatus is clean and dry.<br>2. Turn on the cooling system.<br>3. Slowly fill the tank with the suspension using a funnel with a long spout that reaches the bottom of the inlet chamber to avoid foaming. It is recommended to load the tank to maximum volume and remove any foam that appears during the loading procedure.<br>4. Turn on the mechanical stirrer at 100 rpm in the tank.<br>5. Turn on the high-shear homogenizer and start circulation for 30 min at maximum homogenizer speed.<br>6. Check the viscosity of the batch. If the viscosity is below 100 cP, finish the process. Otherwise, add 10% more treatment time and check again.<br>7. Slowly unload the batch, avoiding foaming.<br>8. Regulate the pressure with a manometer and “high pressure” controller: the pressure should be no higher than 100 MPa.<br>! **NOTE**: Avoid foaming during circulation (it is preferable to have the circulation tank inlet deep in the liquid batch volume). Viscosity after processing must be below 100 cP. |

! **NOTE:** viscosity should be measured at 25°C. For viscosity measurement, a Brookfield viscometer is recommended with suitable measurement range.

TUBALL™ COAT_E

## WARRANTIES AND DISCLAIMER

The Products correspond to the chemical composition indicated in the Technical Data Sheet and the Safety Data Sheet. The information contained in this document (Information) is based on trials carried out by OCSiAl and may contain inaccuracies or errors that could cause injury, loss or damage.

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