Standard Operating Procedure
-
Ultrasonic Dispersion of Multi-walled Carbon Nanotubes using the Hielscher SonicatorUP400ST

1. Introduction

Multi-walled carbon nanotubes (MWCNTs) exhibit exceptional mechanical, electrical, and thermal properties. However, their inherent hydrophobicity and tendency to agglomerate necessitate efficient dispersion techniques to achieve homogeneous and stable suspensions. Ultrasonic dispersion is an effective method for breaking MWCNT agglomerates, ensuring uniform distribution in aqueous and organic solutions.

This SOP outlines an optimized protocol for dispersing MWCNTs in aqueous solutions using the Hielscher UP400ST probe-type sonicator. The protocol ensures minimal damage to the nanotube structure while achieving maximum dispersion quality.

2. Materials and Equipment

2.1 Materials

- Multi-walled Carbon Nanotubes (MWCNTs)
- Surfactant: Sodium Dodecylbenzenesulfonate (SDBS, Sigma-Aldrich, 289957)
- Purified water

2.2 Equipment

- Hielscher UP400ST Ultrasonic Processor (400 W, 24 kHz, digital probe-type sonicator with programmable settings)
- Sonotrode S24d22 (Ø22 mm cylindrical probe)
- Pluggable Temperature Sensor
- Programmable Temperature Limit Function (built into UP400ST)
- Magnetic Stirrer (for surfactant pre-mixing)
- Low-form Pyrex glass beakers (2000 mL capacity)
- Micropipettes (for sample collection)
- Laser Diffraction Particle Size Analyzer

- Permanent Cooling System: Sample vessel placed in an ice bath to maintain low

3. Experimental Procedure

3.1 Preparation of Surfactant Solution

1. Add the required mass of SDBS surfactant to 1000 mL water in a 2000 mL beaker.
2. Stir using a magnetic stirrer for 60 s until an opalescent solution forms, indicating complete surfactant dissolution.

3.2 Addition of MWCNTs

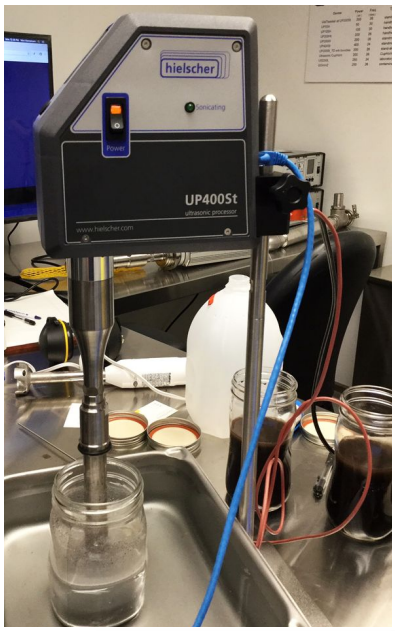
1. Gradually introduce 5 g of MWCNTs (0.5 wt%) into the surfactant solution.
2. Stir for 2 min to distribute the nanotubes.

3.3 Ultrasonication Parameters

Sonicator Settings:

- Sonicator Model: Hielscher UP400ST
- Sonotrode: S24d22 (Ø22 mm)
- Amplitude: 100%
- Ultrasonic energy input: 200–300 Ws/mL
- Pulse mode: 7 s on / 3 s off (70% cycle)
- Temperature Control:

- Pluggable Temperature Sensor to monitor sample temperature
- Programmable Temperature Limit to ensure the suspension does not exceed 40°C
- Permanent Cooling: The sample vessel must be placed in an ice bath during sonication process



3.4 Sonication Process

1. Immerse the sonotrode into the suspension. The sonotrode should not touch the walls of the vessel.
2. Set the sonicator to 70% cycle mode and an energy limit of 3000 Ws/mL. (Multiply the “Ws/ml” by the volume in ml that you wish to process, in our example 300Ws/mL x 1000mL.)
3. Start ultrasonication at 100% amplitude.
4. Monitor temperature continuously using the pluggable temperature sensor.
5. Ensure temperature remains below 40°C using the programmable temperature limit and permanent ice bath cooling.
6. Take samples after 100Ws/mL, 200Ws/mL and 300Ws/mL for analysis.

3.5 Post-Sonication Analysis

1. Visual Inspection: Check for homogeneity.
2. Laser Diffraction Particle Size Analysis (LMLD):
   - Evaluate CNT agglomerate size distribution.
   - Ensure dominant peaks appear at 0.3, 0.9, and 2.5 µm, indicating well-dispersed individual MWCNTs.
3. Stability Assessment:
   - Allow the suspension to rest for 3 days.
   - Check for sedimentation.

4. Optimization Insights

- Amplitude significantly influences the power transmitted to the suspension.
- Excess sonication does not improve dispersion, but may degrade nanotube integrity.
- Surfactant concentration (SDBS/CNT ratio = 1:1) is sufficient; higher ratios (2:1) lead to micelle formation without additional dispersion benefits.
- Temperature regulation is crucial: The pluggable temperature sensor, programmable temperature limit, and permanent ice bath cooling ensure optimal dispersion without thermal degradation.

5. Safety Considerations

- Always wear PPE (gloves, lab coat, safety glasses) while handling MWCNTs.
- Operate the sonicator in a fume hood to prevent aerosolization.

6. Conclusion

This SOP provides a validated and reproducible methodology for the ultrasonic dispersion of MWCNTs using the Hielscher UP400ST sonicator. Controlled sonication with ~200–300 Ws/mL ensures a homogeneous dispersion without compromising nanotube integrity.

With the integration of temperature monitoring (pluggable sensor), programmable temperature control, and continuous ice bath cooling, this protocol ensures a consistent and reliable dispersion process. These parameters enable high-quality suspensions suitable for applications in composites, nanomedicine, and electronic devices.

For further refinement, researchers can adjust surfactant concentrations and sonication times based on specific material characteristics and application needs.

Do you have questions or need help?
Ask our technical team by sending an email to info@hielscher.com!