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# Comparison of dispersion of nanoparticles ultrasonicated with probe and cup horn

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**Abstract:** This study aims to investigate the effects of a probe and a cup horn on the de-agglomeration efficiency in ultrasound vibration processes. $TiO_2$ and $Al_2O_3$ nanoparticle dispersions were prepared in distilled water at a concentration of 50.0 mg/mL followed by treatment with a dispersion stabilizer (100% FBS) and ultrasound vibration at 20 kHz and 35% amplitude for 10 min by a probe and a cup horn, respectively. The average sizes of dispersed $TiO_2$ and $Al_2O_3$ nanoparticles were measured by a dynamic light scattering device. Compared to dispersion with the probe sonicating, the average sizes of $TiO_2$ and $Al_2O_3$ particles sonicated by the cup horn are markedly smaller at time points of 30, 60, 120, and 180 min. The $TiO_2$ and $Al_2O_3$ particle size distributions of cup horn-treated suspensions were narrower than those of probe-treated suspensions at time points of 120 and 180 min. It is suggested that the cup horn has a higher efficiency than the probe in dispersing nanoparticles. The cup horn is better than the probe for processing multiple small sample vessels simultaneously. Indirect cup horn sonication is ideal for processing pathogenic and sterile samples.

**Key words:** nanoparticles; dispersion; ultrasonic devices

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Over the past decade, there has been a rapid development of research in the field of nanotechnology. Nanoparticles are defined as particles with a diameter of 100 nm or less at least in one structural dimension [1]. Compared to the same materials with micrometer scale dimensions, nanomaterials have specific properties, such as small size, large surface area, and special structure. Currently, nanotechnology is developing rapidly and nanomaterials are being involved in all aspects of life and commercial products including drug delivery, sensors, photodecomposition, pigment, catalysis, and thermal conductivity enhancement [2-8]. Nanoparticle dispersions in liquids are a critical requirement for a diversity of nanomaterial applications. Because of the large specific surface of nanoparticles, the interaction forces between the particles increase. Nanoparticles often form agglomerates in media. Additional external forces are required to overcome the adhesion forces of nanoparticles. Although mechanical vibration is often used to improve a homogeneous dispersion, it cannot prevent the nanoparticles from agglomerating. Ultrasonication is an efficient approach for dispersing nanoparticles homogeneously in solution[9-11]. The dispersing ability of ultrasonication arises from acoustic cavitation, which provides a unique interaction of energy and substance [12]. Cavitational collapse produces intense local heating and high pressures. This impairs the conjugation of nanoparticles and disperses nanoparticles in stabilization. The probe delivers direct sonication offering a high intensity sonication for processing most samples. The cup horn delivers indirect sonication with a high intensity ultrasonic bath, capable of processing multiple sample vessels simultaneously. The aim of this study is to investigate the influence of the probe and the cup horn on the de-agglomeration efficiency in ultrasound dispersing processes.

## 1 Material and Methods

### 1.1 Chemicals

Titanium dioxide (Degussa P25, primary particle size 21 nm, 80% anatase, 20% rutile, specific surface area (50 ± 15) $m^2/g$, Degussa Company, Germany) and aluminum oxide (primary particle size 13 nm, purity ≥ 99.6%, specific surface area (100 ± 10) $m^2/g$, Degussa, Germany) were commercially available and used for the experiments. Fetal bovine serum (FBS) was purchased from Hyclone (New Zealand).

### 1.2 Dispersion and characterization of nanoparticles

The dry powder of nanomaterials ($TiO_2$ and $Al_2O_3$) was weighed on an analytical mass balance (Sartorius BS210S, Goettingen, Germany), suspended in distilled water at a concentration of 50.0 mg/mL, and vibrated by vortex for 2 min (SK-1 Vortex, Hengfeng Instrument Factory, Jintan, China). To aid in preparing a homogeneous dispersion, suspensions were sonicated for 1 min at 35% amplitude (Sonicator ultrasonic processor, model S-4000, Misonix, Inc., Farmingdale, NY, USA.) by a cup horn. A dispersion stabilizer (100% FBS) was added to nanoparticle dispersions in order to obtain nanoparticle suspensions at a concentration of 0.5 mg/mL. Subsequently, the suspensions (2 mL) were vibrated for 2 min by vortex and sonicated for 10 min at 20 kHz and 35% amplitude by a cup horn. The average sizes of $TiO_2$ and $Al_2O_3$ particles in suspensions were determined by the method of dynamic light scattering (Zetasizer Nano ZS90, Malvern Instruments Ltd., Worces-

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tershire, UK) and analyzed by transmission electron microscopy (JEM-200CX, JEOL Ltd., Tokyo, Japan).

### 1.3 Determination of particle size

Before being used in experiments, the suspensions (2 mL) at a concentration of 0.5 mg/mL were vibrated for 2 min by vortex and sonicated for 30, 60, 120, and 180 min at 20 kHz and 35% amplitude by a probe and a cup horn, respectively. The average sizes of $TiO_2$ and $Al_2O_3$ particles in suspensions were determined using a dynamic light scattering device at each time point after sonication. The polydispersity index (PdI) describes the width of the particle size distribution.

### 1.4 Statistical analysis

Statistical analysis was performed with SPSS software. Data were presented as mean ± SD of at least three independent experiments. Statistical significant differences between groups were evaluated using Student's t-test. An $\alpha$ level of $P < 0.05$ was considered to be statistically significant. An asterisk * means that the cup horn-treated group has a significant difference from the probe-treated group.

## 2 Results

### 2.1 Particle characterization

The average sizes of $TiO_2$ and $Al_2O_3$ nanoparticles in suspensions measured by the dynamic light scattering analysis are (255.3 ± 6.8) nm and (176.8 ± 4.6) nm, respectively. TEM images of the $TiO_2$ and $Al_2O_3$ nanoparticles are shown in Fig. 1. The average size of particles measured by the dynamic light scattering analysis is larger than the particle size determined by TEM for the two types of particles. These results indicate that the nanoparticles formed agglomerates in the prepared suspensions.

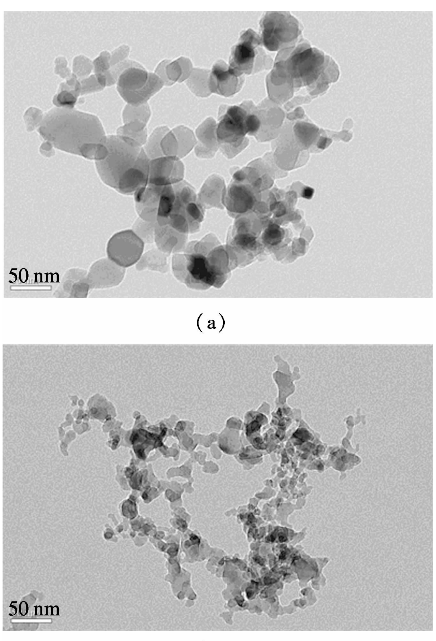


**Fig. 1** TEM analysis of nanomaterials (120 000 ×). (a) $TiO_2$; (b) $Al_2O_3$

### 2.2 Effects of probe and cup horn on $TiO_2$ nanoparticle dispersion

The average sizes of $TiO_2$ particles in the suspensions sonicated with the probe and the cup horn are shown in Fig. 2. The results of PdI for nano-$TiO_2$ suspensions are shown in Fig. 3.

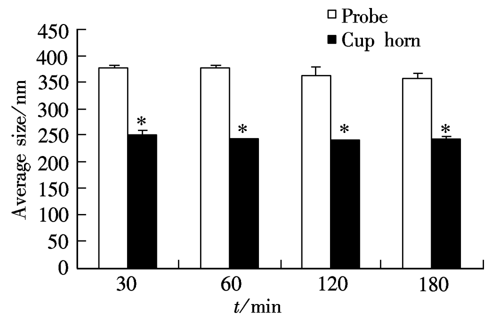


**Fig. 2** Particle size of nano-$TiO_2$ in suspension

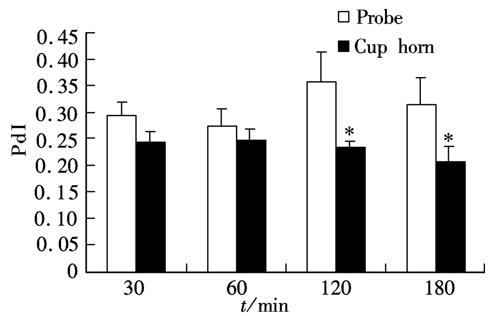


**Fig. 3** Particle size distribution of nano-$TiO_2$ in suspension

The results show that the average particle sizes for $TiO_2$ suspensions vibrated with the probe and the cup horn range from (378.9 ± 3.3) nm to (356.2 ± 8.2) nm and (251.2 ± 6.6) nm to (242.1 ± 3.5) nm in the time interval between 30 and 180 min, respectively. Compared to dispersion with the probe sonicating, the average sizes of $TiO_2$ particles sonicated by the cup horn are markedly smaller at time points of 30, 60, 120, and 180 min.

The PdI of particles in $TiO_2$ suspensions sonicated with the probe and the cup horn range from 0.296 ± 0.025 to 0.317 ± 0.051 and 0.246 ± 0.020 to 0.207 ± 0.030 in the time interval between 30 and 180 min, respectively. The $TiO_2$ particle size distributions of the cup horn-treated suspension are narrower than those of the probe-treated suspension at time points of 120 and 180 min.

### 2.3 Effects of probe and cup horn on $Al_2O_3$ nanoparticle dispersion

The average sizes of $Al_2O_3$ particles in the suspensions sonicated with the probe and the cup horn are shown in Fig. 4. The results of PdI for nano-$Al_2O_3$ suspensions are shown in Fig. 5.

The average particle sizes for nano-$Al_2O_3$ suspensions vibrated with the probe and the cup horn range from (248.1 ± 8.0) nm to (252.5 ± 13.7) nm and (175.8 ± 1.5) nm to (175.4 ± 1.8) nm in the time interval between 30 and 180 min, respectively. The average sizes of $Al_2O_3$ particles sonicated by the cup horn are significantly smaller than those of the probe-treated group at time points of 30, 60, 120, and 180 min.

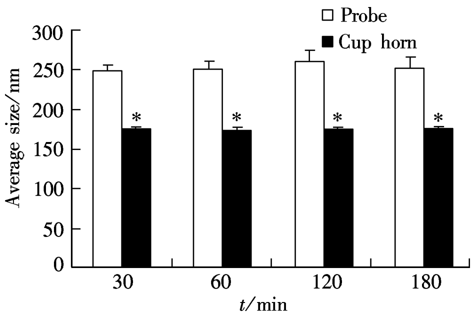


**Fig. 4** Particle size of nano-$Al_2O_3$ in suspensions

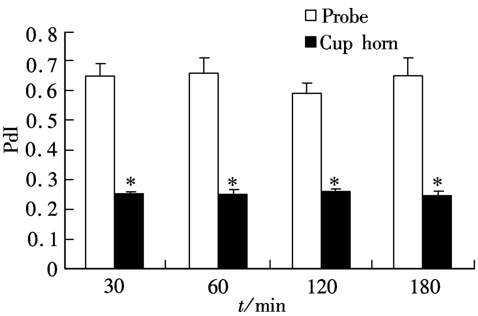


**Fig. 5** Particle size distribution of nano-$Al_2O_3$ in suspension

The PdI of particles in $Al_2O_3$ suspensions sonicated with the probe and the cup horn range from $0.648 \pm 0.043$ to $0.651 \pm 0.057$ and $0.251 \pm 0.006$ to $0.244 \pm 0.015$ in the time interval between 30 and 180 min, respectively. The $Al_2O_3$ particle size distributions of the cup horn-treated group are narrower than those of the probe-treated group at time points of 30, 60, 120, and 180 min.

## 3 Discussion

Ultrasound dispersion is an efficient method to prepare well-dispersed nanoparticle suspensions since cavitation is responsible for a high energy input. Due to the collapse of cavitation bubbles, energy gets released from collapse of the bubbles and is transferred to the suspensions, resulting in nanoparticle de-agglomeration. The horn serves as the second stage of acoustic amplification. In our study, $TiO_2$ and $Al_2O_3$ nanoparticle dispersions are treated with ultrasound vibration at 20 kHz and 35% amplitude by a probe and a cup horn for 10 min, respectively. We find that the average sizes of $TiO_2$ and $Al_2O_3$ particles treated with the cup horn are significantly smaller than those of the probe-treated group at the same time points. Also, the particle size distributions of $TiO_2$ and $Al_2O_3$ particles treated with the cup horn are narrower than those of the probe-treated group at the same time points as well. The probe tip diameters deliver high intensity sonication but the energy is focused in a small concentrated area. Large tip diameters can process large volumes, but offer lower intensity. In addition, to avoid damaging the probe, energy output is limited from the microtip in the ultrasonic machine. It is suggested that the dispersing efficiency of the cup horn is better than that of the probe in preparation of nanoparticle suspensions.

Serum is successfully used as a stabilizer in preparing nanoparticle suspensions and subsequent investigation of the biological behavior of nanoparticles[9,13]. Probe sonication offers high intensity, capable of processing most samples. Ultrasound vibration with the probe leads serum to produce large amounts of foam in suspension. However, this defect can be overcome in the cup horn-treated group. Indirect sonication is most effective for very small volume samples because foaming and sample loss are eliminated. The microtubes are placed within the water filled reservoir and the sonic energy is transferred into each individual well. Thus, the cup horn is more suitable for processing multiple small sample vessels.

Cup horn sonication eliminates the need for a probe to come in contact with samples. The contaminants of some other particles are prevented from entering suspensions in a cup horn. This method is suitable for processing pathogenic or sterile samples since aerosols and cross contamination are prevented. The cup horn supplies a high intensity ultrasonic bath, capable of avoiding sample contamination and processing multiple sample vessels at one time.

## 4 Conclusion

This study aims to investigate the effects of different ultrasonic horns on the dispersing efficiency in ultrasound vibration processes. The average sizes of dispersed nanoparticles are determined by using a dynamic light scattering device. Our results show that the cup horn has a higher efficiency than the probe in dispersing nanoparticles. The cup horn is better than the probe for processing multiple small sample vessels simultaneously. Indirect cup horn sonication is ideal for processing pathogenic and sterile samples.

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# 探针式和杯式超声探头对超声分散纳米颗粒的效果比较

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**摘要:**比较了探针式和杯式超声探头对纳米颗粒的超声分散效果. 用蒸馏水将纳米二氧化钛和纳米三氧化二铝配制成浓度为 50.0 mg/mL 的混悬液,随后加入分散稳定剂(100% 胎牛血清),分散液涡旋振荡 2 min 后备用. 分别采用探针式和杯式探头以 20 kHz,35% 振幅超声波将上述 2 种混悬液振荡 10 min,用动态光散射法分析二氧化钛和三氧化二铝纳米颗粒的粒径大小及其分布情况. 结果表明,杯式探头超声波振荡组在 30,60,120,180 min 时间点的二氧化钛和三氧化二铝平均粒径均明显小于探针式探头超声波振荡组. 同时,杯式探头超声波振荡组在 120 和 180 min 时间点的二氧化钛和三氧化二铝颗粒粒径分布范围均窄于探针式探头超声波振荡组. 结果说明杯式超声探头比探针式超声探头对纳米颗粒具有更好的分散效果. 杯式探头在同一时间能处理多个小容积样本且特别适合于制备有致病危险性的样品或无菌样品.

**关键词:**纳米颗粒;分散;超声设备

**中图分类号:**TB383