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High-gravity process intensification technology and application

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ABSTRACT

Process intensification has long been known to be a vital concept and is considered to be one of the fundamental pillars of the chemical engineering domain. This paper reviews the advances on the high-gravity (Higee) technology, including the theoretical studies on liquid flow, gas–liquid mass transfer, residence time distribution and micromixing, and the applications of Higee technology in absorption, stripping, nanoparticles preparation and other fields such as sulfonation, polymerization, synthesis of diphenyl-methane-diisocyanate and emulsification. The application of Higee in industry shows good adaptabilities of this technology and brings considerable profits.

1. Introduction

When we consider the world with its increasing need for space, safety, good environment, and economic growth, it is evident that all sectors of production have to critically assess their impact in these respects. For the chemical process industry, this implies that a decrease in size and an increase in efficiency are a must [1]. As one of the cutting-edge process intensification technologies, Higee has received considerable attention and extensively explored in absorptions, stripping, distillation, heat transfer, adsorption, extraction, nanoparticles preparation, reaction, and so on [2–15]. Higee technology, carried out in a rotating packed bed (RPB), was originally invented by Colin Ramshow and his co-workers in 1979 for the separation processes [16,17]. The height of transfer unit of the RPB is as low as 1–2 cm and the efficiency of mass transfer and micromixing can be up to 1–3 orders of magnitude larger than that in a conventional packed bed, exhibiting prominent process intensification characteristics.

The key point of the Higee technology relies on the simulated high-gravity environment created by the centrifugal force of the RPB. In the RPB, very thin liquid films and/or tiny droplets are generated and the interface between gas/liquid or liquid/liquid is renewed violently, leading to a significant intensification of mass transfer and micromixing.

2. Fundamentals

2.1. Liquid flow

Researchers [18–21] performed the visual study of liquid flow in the RPB and established a model to represent the flow patterns, which indicated that the liquid in the packing existed as films attached to the packing wire and filling the voids of the packing when the high gravity level was low ($<60g$), but as films on the packing wire, the droplets and small amounts of filaments in the voids of the packing when the high gravity level was high ($>100g$). Thickness of the liquid film was estimated as 8×10^{-5} m and 5×10^{-5} m at the rotational speed of 600 rpm and 1200 rpm, respectively [21,22]. Methods to calculate the droplet diameter, liquid holdup, flooding, and pressure drop were also presented [22–29].

2.2. Gas–liquid mass transfer

Ramshaw [16,17] reported the very first data in a Higee system for the absorption of oxygen in water. The results showed that the overall liquid-phase volumetric mass-transfer coefficients ($K_L a$) increased significantly with increasing rotational speed. Tung and Mah [30] derived a mass-transfer model for the RPB based on the penetration theory and obtained a correlation for k_L. They found that this correlation could fit Ramshaw's data well. Munjal et al. [27,28] performed the chemical absorption of CO_2 in NaOH solution in RPB and observed that both the gas–liquid interfacial area and the liquid-phase mass-transfer coefficient increased with rotational speed. Studies revealed that the liquid-side mass transfer coefficient of the Higee would be greatly increased (can be about

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Nomenclature

k_L	liquid-side mass transfer coefficient (m/s)
k_G	gas-side mass transfer coefficient (m/s)
K_La	overall liquid-phase volumetric mass transfer (s^{-1})
K_Ga	overall gas-phase volumetric mass transfer (s^{-1})
g	gravitational acceleration (9.8 m/s^2)
t_m	micromixing characteristic time (s)

Greek letter

τ	nucleation induction time (s)

Dimensionless quantities

Sc_L	liquid Schmidt number
Re_L	liquid Reynolds number
Gr_G	gas Grashof number

3–7 times) than that in the conventional columns under modest operation conditions [23].

Gas-phase mass transfer has also been extensively studied. Ramshaw and Mallinson [16] first reported NH_3 absorption in a Higee system and found that K_Ga for NH_3 absorption was 4–9 times higher in such systems than that in conventional packed beds. Sandilya [31] investigated a SO_2–NaOH solution system and found that the k_G values, estimated on the basis of the total surface area of the packing were lower than those of the conventional packed beds or in a range similar to those of conventional packed beds in view of the negligible tangential slip velocity of the gas. Guo [32] constructed a cross-flow RPB and performed experiments involving the absorption of NH_3 into water and the absorption of SO_2 into ammonium sulfite solution. Their results showed that K_Ga was not influenced by rotational speed when the centrifugal acceleration was above 15g.

2.3. Residence time distribution

Residence time distribution (RTD) in RPB, an essential operating parameter for design and scaling up, has been studied by Kenyvani and Gardner [33] based on data acquired from stationary sensor probes mounted at the opening of liquid nozzle and in the space close to the outer periphery of the rotor. Guo carried out the synchronous visual observations with the video camera mounted on the RPB to investigate the RTD. Two electro-conductivity sensors were mounted at the inner and outer edges of the packing to acquire signals in situ during operation. The inner sensor probe was moved radially to desired positions to acquire information on the liquid flow in the packing. Liquid holdup measurements were conducted at the same time. Under the synchronous visual observations, the film thicknesses were 20–80 μm with foam metal packing and about 10 μm with the wire mesh packing. The following conclusions were also reached: (1) the inner region of the packing of about 7–10 mm in radial thickness, called end effect zone, is where the liquid impinges, deforms and mixes most intensively in the whole packed bed. (2) In most part of the rotor, except the end effect zone, liquid flows in films on the surface of the packing and in the voids. (3) The average residence time of liquid in the RPB varies with liquid flow rate and rotational speed, and ranges from 200 ms to 800 ms. (4) Liquid backmixing in the RPB is equivalent to two continuous stirred tank reactors in series and occurs mainly in a narrow region near the inner edge of the packing. This ultra strong mixing region is unique for RPB and is highly important for mass transfer. (5) Liquid holdup does not exceed 5% of total bed volume under normal operating conditions.

Table 1
Comparison on the micromixing efficiency of different kinds of reactors.

Reactors	Micromixing time, t_m	Researchers
Stirred vessel	$t_m = 1–200$ ms	Guichardon [39]
Kenics static mixer	$t_m \geq 1$ ms	Fang and Lee [40]
Submerged circulative impinging stream reactor	$t_m = 87–192$ ms	Wu and Xiao [41]
Couette reactor	$t_m = 0.1–1$ ms	Liu and Lee [42]
Rotor-stator mixers	$t_m = 0.5–2$ ms	Bourne and Garic [43]
Ultrasound on micromixing	$t_m \geq 5$ ms	Monnier et al. [44]
	$t_m \geq 1$ ms	Monnier et al. [45]
Rotor-stator reactor	$t_m \geq 0.04–0.01$ ms	Chu et al. [46]
RPB	$t_m \geq 0.1–0.01$ ms	Chen et al. [47]

2.4. Micromixing

Micromixing (i.e., mixing at the molecular scale) is the last stage of turbulent mixing and consists of the viscous-convective deformation of fluid elements, followed by molecular diffusion. Micromixing is believed to play a very important role in the chemical industry when the time scale of the chemical reaction involved is at the same magnitude as or smaller than the time scale of the mixing process. Uniform spatial concentration distribution on the molecular scale can only be reached by intense micromixing. From the viewpoint of chemical reaction engineering, the reaction rate and subsequent nucleation in precipitation will be controlled by the intrinsic kinetics without the influence of micromixing in the region of $t_m < \tau$ and controlled or influenced by micromixing when $t_m > \tau$. Because of the very strong nonlinearity of homogeneous nucleation, intensification of micromixing to reach the region of

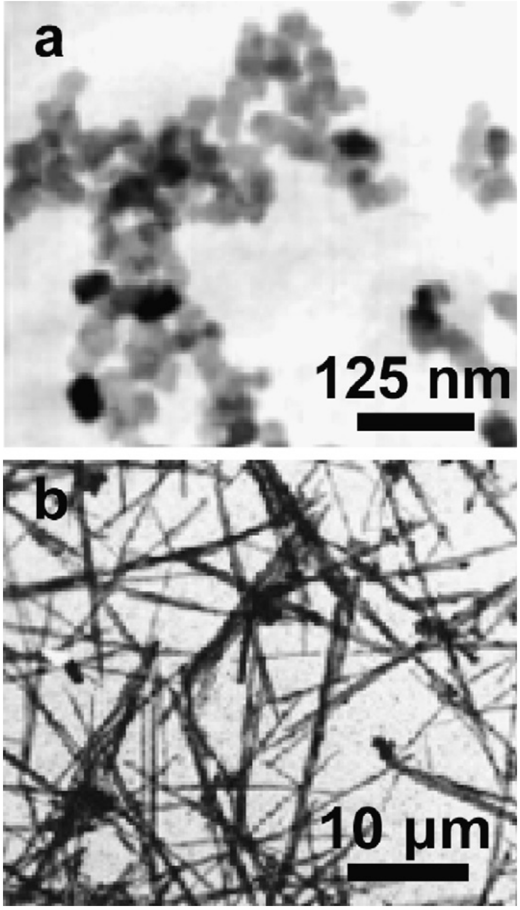

Fig. 1. TEM images of nano-$CaCO_3$ synthesized by Higee. (a) Cubic $CaCO_3$ particles and (b) acicular $CaCO_3$ particles.

$t_m < \tau$ should be taken so that the rates of nucleation at different locations in a precipitator will be nearly the same, and the particle size distribution can be controlled at a uniform level. The t_m in the RPB is evaluated to be about 10^{-4} s (by incorporation model) and smaller than τ, which is often on the order of 1 ms or less [34]. Yang has testified that the RPB is an appropriate reactor for preparing nanoparticles because the RPB possesses superior micromixing efficiency than many mixing devices (Table 1) [29,34–38].

3. Absorption and stripping processes by Higee technology

Gas absorption is one of the most common methods for treating waste gas in industrial processes, including coal gasification, synthetic gas production, natural gas processing, oil refining, and hydrogen manufacture. The RPB has been extensively studied for gas absorption due to the excellent mass transfer performance. In a typical RPB operation, liquid flowing through the packing is subjected to an acceleration of at least 300 m/s^2 tuned by the rotational speed. With this high-gravity environment, the RPB can be operated at higher gas and liquid flow rates, leading to a significant increase of production capacity and reduction of apparatus volume.

Absorption of CO_2 in RPB has been widely studied [27,28,48–52]. The absorbants investigated involve NaOH [51], K_2CO_3 [48], monoethanolamine [49,52,53], 2-amino-2-methyl-1-propanol [51,53] and the mixture, etc. [51]. Yi [48] has investigated CO_2 absorption with experiments and numerical modelling. The predicted values of the absorption efficiency by the model agreed well with the results from experiments at various liquid flow rates, gas flow rates, rotational speeds and temperatures. It was found that $K_G a$ decreased sharply from end effect zone to bulk packing zone. The model profiles revealed the end effect phenomena in the RPB, which confirmed that there is much better mass transfer efficiency in the inner region of the packing. Additionally, the effects of various operation parameters on the mass transfer efficiency in the RPB at higher gravity level were predicted reasonably by this model. This model may be used to simulate the removal of CO_2 in the RPB using other aqueous absorbents.

Li [54] has studied SO_2 absorption by the Higee technology in the sulfuric acid factory in Zibo of China, with the concentration of SO_2 in gas outlet reduced to less than 300 ppm or even as low as 50 ppm. Compared with a conventional tower, the investment, space, and energy consumption by the Higee technology decreased from about $130,000; 120 m^3; and 80 kW, to $85,000; 60 m^3; and 60 kW, respectively. Reddy [8] applied the Higee with split packing to SO_2 absorption and received good results. The removal of H_2S [55], ozone [7,13,56–58], volatile organic compounds (VOCs) [5,59–62], and seawater deaeration [63] was also studied, which revealed that the Higee is an enabling technology in the separation of specific compounds from gas or liquid streams.

4. Preparation of nanoparticles by Higee technology

Reactive precipitation is regarded as an effective method for mass production of nanoparticles because of low cost, and ease of scale-up. However, the conventional precipitation process is often carried out in a stirred tank or column reactor, and it is difficult to control the quality of the product and the morphology and size distribution of particles often change from one batch to another.

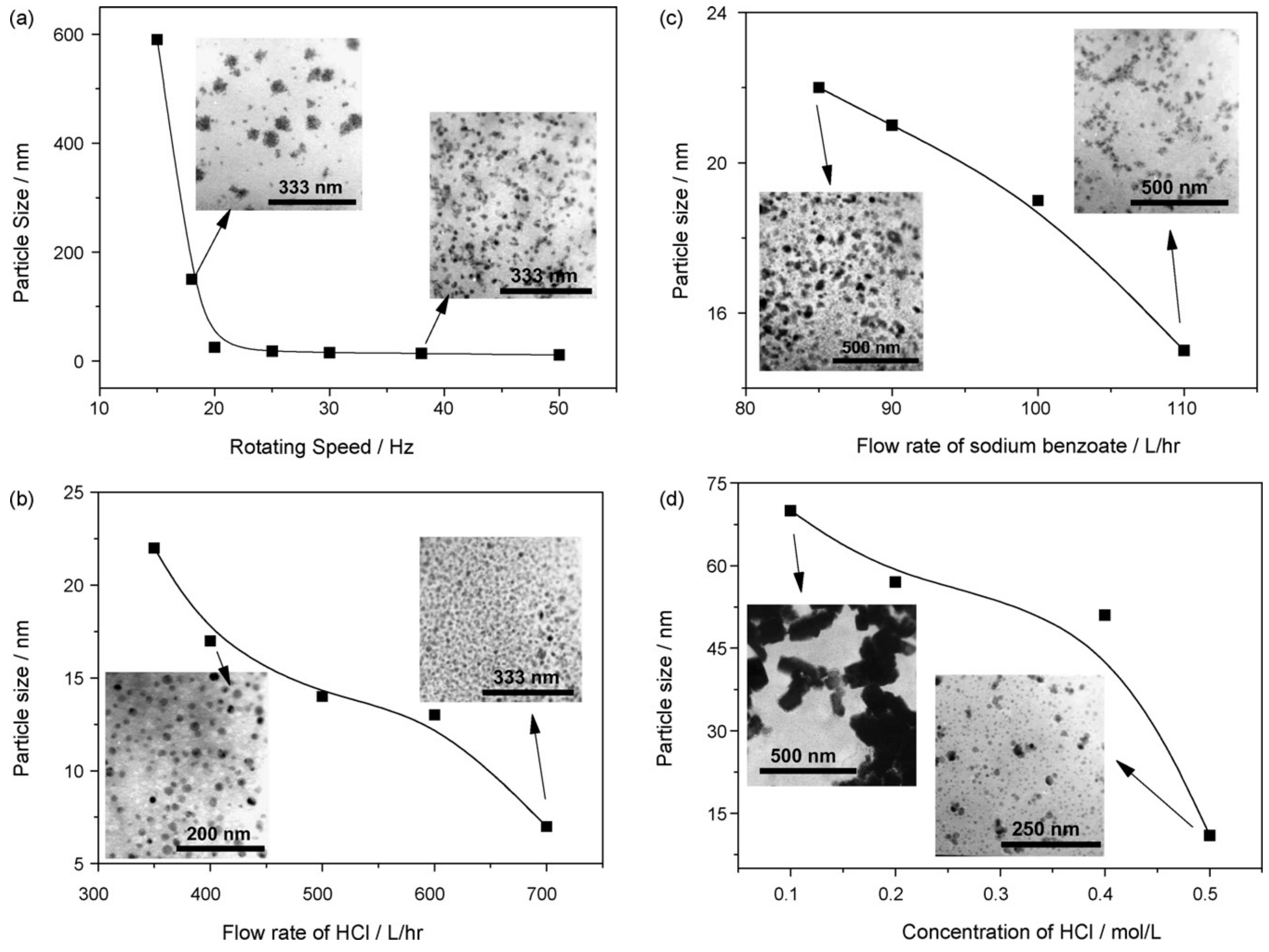

Fig. 2. Profiles of the change of particle size with (a) rotational speed, (b) flow rate of HCl, (c) flow rate of sodium benzoate, and (d) concentration of HCl.

Using the Higee as an innovative reactor to synthesize nanoparticles by precipitation process has become a promising technology thanks to the greatly enhanced mass transfer and micromixing efficiency.

4.1. Synthesis of inorganic nanoparticles by Higee

Inorganic nanoparticles are generally synthesized by using the high-gravity reactive precipitation (HGRP) process with gas–liquid–solid, gas–liquid, and liquid–liquid reactant systems. Take the preparation of $CaCO_3$ as an example, and the $CaCO_3$ particles can be synthesized by the reaction below:

$$Ca(OH)_2(\text{Solid}) + CO_2(\text{Gas}) + H_2O(\text{Liquid}) \rightarrow CaCO_3(\text{Solid}) + 2H_2O(\text{Liquid})$$

When this chemical process was performed in the RPB, the mean particle size of the $CaCO_3$ can be controlled in the range of 15–30 nm without any chemical additives. Moreover, by using surfactants, the nano-$CaCO_3$ can be tuned into different morphologies to fulfill various applications [29,37] (Fig. 1.).

Due to the high quality of the product and ease of commercial production, the production of nano-$CaCO_3$ by HGRP has been rapidly accepted by the $CaCO_3$ industry. The nano-$CaCO_3$ production lines using HGRP technology have been put into operation in five enterprises in China with overall production capacity reaching 36,000 t/a.

Other nanoparticles with well-defined size and morphology were also synthesized by HGRP technology, including $Al(OH)_3$ (1–10 nm of diameter, 50–300 nm in length), SiO_2 (30–50 nm), TiO_2 (20–30 nm), ZnO (20–60 nm), ZnS (20–30 nm), $SrCO_3$ (granular 30 nm), $BaCO_3$ (40–70 nm) and $BaTiO_3$ (stick-like). In situ synthesis of nano-$CaCO_3$ (20–40 nm) in the preparation of overbased petroleum calcium sulfonate by the Higee technology was also reported [64].

4.2. Synthesis of pharmaceutical nanoparticles by Higee

The effect of particle size reduction on the improvement of the bioavailability of hydrophobic pharmaceuticals has inspired scientists to turn to nanotechnology for solutions. Chen [38] has probed the feasibility of preparing pharmaceutical nanoparticles by HGRP method. Benzoic acid was employed as a model compound for the investigation of reactive precipitation of pharmaceuticals in the RPB. Benzoic acid particle size can be controlled by manipulating rotational speed, concentration and flow rate of the reactants (sodium benzoate and HCl) and benzoic acid nanoparticles as fine as 10 nm were obtained (Fig. 2). Nanoparticles of salbutamol sulfate and cephradine were synthesized by this HGRP method [65,66]. Subsequently, a high-gravity antisolvent precipitation (HGAP) method was developed, which utilizes the great difference of the solubility of the drugs in different solvents and the excellent micromixing performance of the RPB to produce nano-sized particles, such as cefuroxime axetile and danazol (Fig. 3) [36,67]. Compared with the commercially available particles, the nano-sized drug particles prepared by HGRP or HGAP demonstrated significantly improved bioactivities, such as faster dissolution rate [36,67], less need of complex solubilizers [68], higher fine particle fraction (FPF) [14,69] and so on.

5. Higee applications in other fields

5.1. Sulfonation

Petroleum sulfonate surfactant for enhanced oil recovery (EOR) is usually synthesized by sulfonation with petroleum fractions above 200 °C, liquid SO_3, $NH_3{\cdot}H_2O$, $C_2H_4Cl_2$, and composite aluminium salt catalysis. Sulfonation is an instant reaction. However, it is difficult to achieve a uniform reactant concentration distribution in conventional apparatuses before the sulfonation process, which probably leads to over sulfonation of the products. Therefore, special feeding method and good micromixing are required in preparing the petroleum sulfonate surfactant. The application of Higee technology to the preparation of petroleum sulfonate surfactant [70] satisfied these requirements and overcame the problem of the inability to sulfonate the non-aromatic hydrocarbon by conventional pathways. The oil–water interfacial tension of the product by Higee was lowered to the range of 10^{-3} mN/m, which is about 1/10 of that by conventional technology.

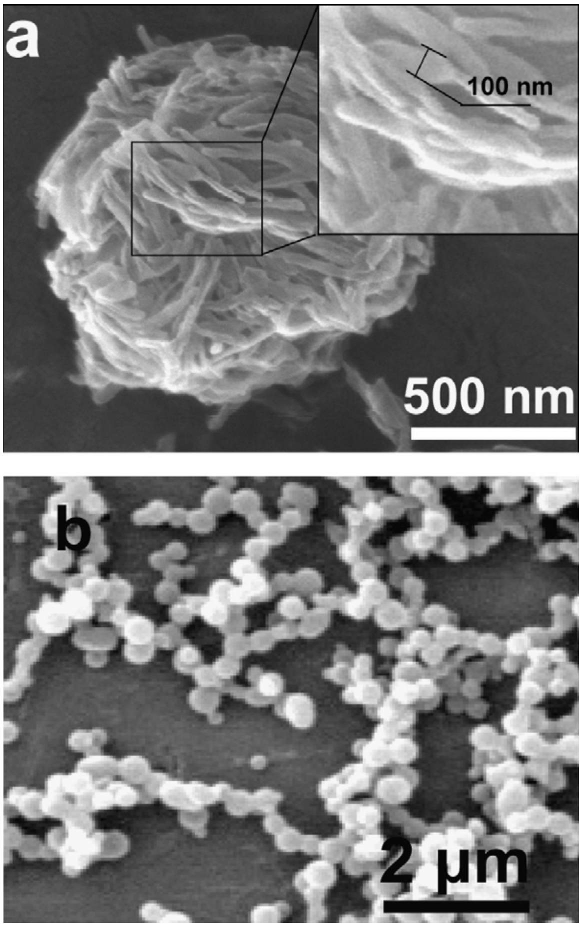

Fig. 3. SEM images of pharmaceutical nanoparticles prepared by Higee. (a) Spherical aggregates of salbutamol sulfate nanoparticles and (b) spherical cefuroxime axetil nanoparticles.

5.2. Polymerization

Butyl rubber synthesis by polymerization is a typical cationic polymerization, which is a rapid exothermic reaction. The polymerization degree of butyl rubber will increase with the decrease of the temperature, and reactors with excellent mass, heat transfer and micromixing properties are needed. It was revealed that butyl rubber prepared by the Higee technology had the molecular weight of 2.89×10^5, molecular weight distribution index of 1.99 and single-pass conversion of 30% at a rotational speed of 1200 rpm and −100 °C. In addition, the mean residence time of the product was less than 1 s, significantly shorter than 30–60 min in the tubular reactor. The production efficiency per unit volume of the RPB also

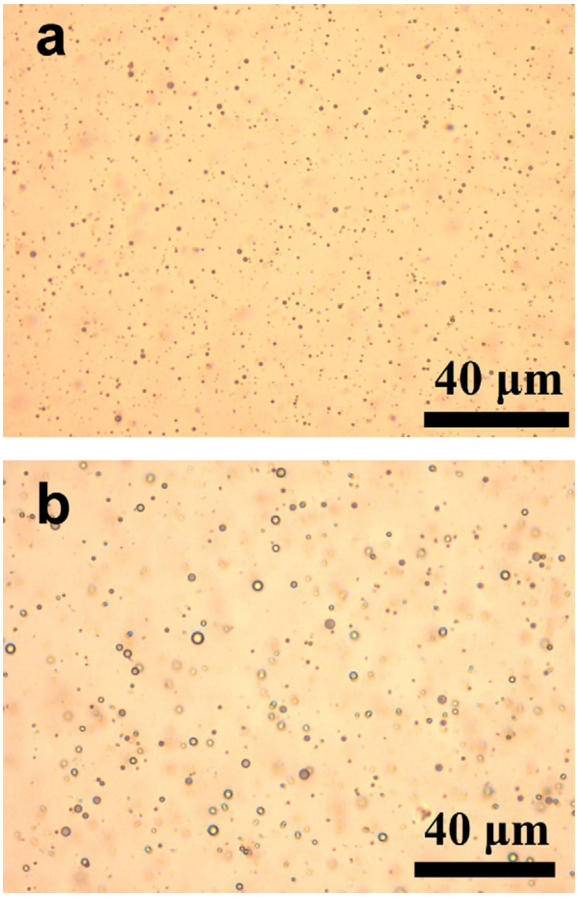

Fig. 4. Optical micrographs of the emulsions prepared by (a) Higee technology and (b) the conventional homogenizer.

increased by 2–3 orders of magnitude compared with the tubular reactor [71,72].

5.3. *Synthesis of diphenyl-methane-diisocyanate*

The application of Higee technology in the production of diphenyl-methane-diisocyanate (MDI) was also carried out [73]. In MDI production the phosgenation of COCl with RNH_2 is a rapid reaction process. It is difficult to avoid the side reaction due to the inefficient mixing and non-uniform spacial concentration distribution of the reactants in conventional reactors. The by-products usually result in low-quality product and blockage of the pipeline because of its high viscosity. The application of the RPB in this reaction reduced the side reactions significantly and boosted the product quality. In addition, with the implementation of the Higee technology the production capacity of MDI in Wanhua Co., China increased from 160,000 t/a to 240,000 t/a.

5.4. *Emulsification*

The application of Higee technology to emulsification, patented by Chen's group [74], took advantage of the high shearing force and high gravity field of the RPB to produce high-quality emulsion via the formation of ultrafine and uniform oil droplets (Fig. 4). This patent has been implemented in a 5000 t/a production line of a pharmaceutical enterprise in China. The previous batch production was upgraded by a continuous Higee process, and the 132 kW-homogenizer was replaced with a 10 kW-Higee apparatus, with markedly improved product quality and more than 75% of the energy-saving for the emulsification process.

6. Conclusions

Over 30 years' exploration and application has demonstrated that Higee is a promising and powerful process intensification technology, featuring miniaturization, high efficiency, high quality of the product and ease of scale-up. Higee may exhibit unique advantages in mass transfer limited reactions and the combination of several unit operations in one apparatus. The possibility of applying the Higee technology to absorption, stripping, adsorption, distillation, liquid–liquid extraction, crystallization, dedusting and heat transfer has been widely investigated, and several processes including deaeration of seawater, HOCl production and nanoparticles preparation have enjoyed commercial success.

However, extensive market acceptance of the Higee technology is yet to come due to concerns over reliability and energy cost of rotating equipment, which will be relieved by long-time running of RPBs to gain substantial commercial data and experiences. Applications of Higee technology in a large scale necessitate sound theoretical foundations which should be achieved by comprehensive fundamental researches on the RPB parameters like packing structure, fluid flow, mass/heat transfer, micromixing, pressure drop, residence time, flooding, power and their influence on reaction and separation processes. The scale-up of RPB is mostly empirically present. Fundamental performance characterizations and more generalized theoretical expressions for RPB parameters that accurately predict RPB performance will be needed to confidently scale-up this technology.

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