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Energy and Environmental Science (Communication)

Solid-Liquid Separation by Particle-Flow-Instability

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A robust separation strategy using novel particle-flow-instability physics is successfully developed for *difficult-to-separate suspension* in which there is some combination of a small density difference between solid and liquid, high viscosity, or small-sized particles.[1-3] The method we propose here requires no dilution for medium, high or extremely high viscosity slurries and can produce effective solid-liquid separation with low capital, and operational inputs. The results have relevance for many biotechnological and/or chemical processes. We anticipate that further optimizations could lead to more effective separation process with even lower energy input.

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Energy and Environmental Science (Communication)

1 Background and Motivation

Solid-liquid separation holds a dominant place in the chemical, mineral, pharmaceutical, food, water and waste-processing, and biotechnological industries. Around 50% of the total costs across the process industries are consumed in separation processes.[2] Since it is imperative for industry to increase product purity and reduce waste, it is important to develop more effective separation technologies. While there are myriad specific solid-liquid separation techniques, they broadly fall into two categories: particles move through a constrained liquid (e.g. sedimentation/flotation) or liquid moves through constrained particles (e.g. membrane filtration).

Finding an optimum technology to separate fine particles from viscous liquid in biorefineries is a challenge. Today, one or more solid-liquid separation steps (centrifugation, filtration or sedimentation[3]) are required in the recovery of biomass. Filtration is unsatisfactory for high-viscosity solid-liquid mixtures where fouling is the main challenge for membranes.[4] Centrifugation is energy-intensive and unsuitable for high viscosity systems.[3,5] In sedimentation, flocculants are typically required, which significantly increases the costs, so sedimentation is usually limited to the recovery of high-value products in the context of biotechnological processes.[3,6] There is a need for efficient, low-cost processes for streams of high-viscosity biomass slurries, particularly where large volumes must be processed, to produce a recyclable liquid stream and valuable solids streams.

In this paper, we propose a third category of solid-liquid separation physics where particles (with density $\rho_p \approx \rho_L$) can localize or cluster into small regions of viscous fluid in a stirred vessel. The paper will start with the introduction of a new physical phenomenon and, considering the impacts of fluid rheology and particle shape, results related to some non-ideal conditions will be presented. We also demonstrate solid-liquid separation for a difficult-to-separate suspension, where a conventional agitator is able to efficiently extract the purified liquid from the viscous solid-liquid suspension. The results have substantial implications for some biotechnological/chemical processes.

2 A New Approach to Concentrate Particles

2.1 Stirring in a Mechanical Agitated Vessel at low Reynolds numbers

The approach we propose here uses a standard agitated vessel equipped with a standard Ruston turbine (**Fig. 1A**), and the motivation is to separate fine particles from viscous liquid. One criterion for this approach is to operate the tank under laminar flow condition. The crucial difference between a turbulent tank flow and a laminar tank flow is that the quasi-random fluid pathlines of turbulence are replaced by pathlines forming a Lagrangian coherent structure. In this section, we will qualitatively explain how the coherent structure of a laminar tank flow is responsible for the clustering of inertial particles, with theoretical details are relegated to the supplementary material.

Mixing tanks have been in industrial use for a number of centuries; nevertheless, they are mainly used at high Reynolds number to turbulently disperse and mix materials into a homogeneous state.[7] However, flow tanks operated in the laminar flow regime can produce complex pathlines via chaotic advection. The passing of the impeller blades triggers the onset of chaos by introducing small perturbations in the flow while creating two coexisting confined mixing regions above and below the impellers. The confined mixing regions are two flow tori segregated by well-defined boundary layers.[8-10] These tori are known in dynamical systems parlance as Kolmogorov-Arnold-Moser (KAM) tubes.[11-13]

Fig. 1B shows that the structural properties of the tori can be complex with several filaments and a core torus.[10, 25, 27] This figure shows that the flow structure in the non-turbulent flow tank is a typical chaotic flow with coexisting KAM tubes and chaotic regions; this is the kinematic flow template that mediates inertial forces to influence the novel *clustering* instability, which will be discussed in the rest of this article. As the boundaries of KAM tubes are barriers for material exchange in fluid flow, these tubes create *separated flow regions*.[8-10, 27, 28]

2.2 Clustering Instability

Our main result is illustrated in **Fig. 2** using spherical polystyrene particles (with ρ_p/ρ_f = 0.80, a = 1.4mm). At a particular laminar Re, the particles were poured into the mixing tank and swiftly dispersed throughout the tank except into the separated flow region. Clustering begins almost immediately, and after a few minutes a large number of particles have moved into the KAM areas where they stay. In this case, particles in the KAM areas occupy ~10% of the total fluid volume. The right-most picture (at t~30mins) shows that eventually all particles are trapped in the tubes. A video of the spontaneous clustering is in the supplementary material. Similar experiments were conducted using other particles ($0.35 \leq \rho_p/\rho_f \leq 1.19$, 0.65mm ≤ a ≤ 7mm) as listed in **Table 1** and clustering was observed for all the particles.

We trace out the particle and fluid orbits in several ways. In this paper, we developed a technique that is particularly good at exposing the structure of individual particle orbits in the stirred tank. The visualisation approach involved placing light emitting diode inside transparent spherical capsules and then manually inserting them into the fluid. Long exposure photographs were then taken, such that colored pathlines were traced out in the resulting images due to the movement of the capsules in the flow. The photograph in **Fig. 3A** shows the orbits of the light capsules (essentially some large particles) from their introduction to the fluid surface near the tank wall (stars). The orbits of the three capsules spontaneously and rapidly move from the chaotic region into the KAM tubes. Once in the tubes the capsules move in helical orbits as do the passive fluid particles (**Fig. 3B**). These results show conclusively that particles can scatter from the chaotic flow regions and subsequently settle in the KAM tubes in a laminar stirred system. Next, we will discuss the necessary conditions for clustering.

2.3 Clustering Criteria

Why does this clustering phenomenon happen? Our explanation is that, when the inertial perturbations in the flow outside KAM regions, i.e. high strain areas in the chaotic flow region in our stirred tank (**Fig. 1B**), amplify particle trajectories away from the fluid trajectories, the particles can then move and settle into the nearby separated flow regions, i.e. KAM tubes. Particle clustering in stirred tanks is essentially due to the interplay of both attractors and repellors in the augmented dynamical system for motion of inertial particles.[15] Note that attractors and repellors are common features in fluid dynamical systems. In the stirred tank, chaotic mixing regions are considered to be repellors and KAM tubes are attractors.

More precisely, there are two necessary conditions for particle clustering: (1)the existence of repelling regions, or repellors, to convert particle trajectories from being nearly coincident with fluid trajectories (passive behavior) into particle trajectories that are independent of fluid trajectories (non-passive behavior); and, (2) separated flow regions, attractor, not far from repelling regions, into which particles can settle and that prevent particles encountering repelling regions again. KAM tubes amply fulfill the condition (2), as shown in **Fig. 3B**. Next, we focus on the first condition.

When a particle is sufficiently tiny, spherical and neutrally buoyant, it behaves as an infinitesimal fluid element and strictly follows the underlying fluid flow.[13] However, when these conditions are not satisfied, the inertia of the particle prevents it from instantaneously matching its movement to that of the surrounding fluid.[14] The misalignment comes purely from the finite-size effects when the particle will take a finite time to respond to flow acceleration and the flow stresses are averaged over the particle surface.[14] The significance of size and density of the particle is captured by an inertia number

$$\sigma = \frac{St}{R} \quad \text{with} \quad St = \frac{2}{9}\left(\frac{a}{L}\right)^2 Re, \quad R = \frac{1}{\frac{1}{2}+\frac{\rho_p}{\rho_L}} \qquad (1)$$

where a is the particle radius, L is the flow length scale (impeller diameter in our case), St is the Stokes number, Re is the Reynolds number and R is the density ratio. The inertial factor σ gives the ratio of the particle relaxation time and the typical time scale of the flow. For $\sigma << 1$, a particle moves passively, that is, it does not change the fluid velocity field and instantaneously matches its own velocity to that of the fluid. As σ increases, a particle is non-passive because inertia causes it to deviate from the fluid streamline.

It is generally assumed that particles with low enough inertia behave as part of the fluid, i.e. a particle released in a fluid with a slight velocity mismatch compared to the fluid velocity would adapt, after a short transient, to the local fluid velocity. While generally true, this assumption breaks down at the repelling regions, where particles can scatter from the fluid

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flow. Sapsis and Haller, expanding on earlier work[16,17], derived a criterion for repellors in the flow in terms of the eigenvalue of a tensor. [18, 19] With some manipulations (given in the supplementary material) it can be shown that repellors occur in the flow at locations where

$$\sigma\dot{\gamma} > 1 \qquad (2)$$

where $\dot{\gamma}$ is the local non-dimensional strain rate and $\sigma\dot{\gamma}$ is interpreted as inertial stress. It should be noted that the strain along streamline is proportional to the radius of curvature of the streamline. A particle can either follow a fluid streamline if it curves less, while deviates from the streamline when the streamline curves too much. Eq. (2) quantifies how much strain is 'too much' or, in other words, indicates how much strain is required to amplify particle trajectories away from fluid trajectories.

Eq. (2) is a local criterion on the fluid flow, because strain rate $\dot{\gamma}$ varies from place to place in the stirred system. In lieu of a measurement, we use correlations of $\dot{\gamma}$ as a function of Re and $\dot{\gamma} = kRe$ in Newtonian fluids, where $k=5$ is a constant specific to the Rushton impeller type.[21] It should be noted that k is available for many combinations of impeller type and fluid rheology. Substitute the correlation into Eq. (2), and this instability criteria become

$$k\sigma Re > 1 \qquad (3)$$

We emphasize that, Eq. (3) defines the global instability boundary, rather than a local criterion. We then substitute $Re = \rho_f N L^2/\mu$ into Eq. (3) (N is the impeller speed), and after the rearrangement, find the minimum rotational rate (N_{min}) required for particle clustering:

$$N_{min} = \frac{\mu}{aL\rho_f}\sqrt{\frac{9}{2}\frac{R}{k}} \qquad (4)$$

This equation can predict the critical impeller speed for clustering for any given particle and fluid properties. For example, **Fig. 4** shows the theoretical critical speeds required to cluster

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particles with ρ_p/ρ_f=*0.5, 1* and *2*, respectively in the tank. The universal trend is that smaller particles tend to migrate into the tube at higher rotation rate.

To compare with theory, we monitored the signs of clustering at different speeds. **Fig. 5** shows the critical (i.e. minimum) speeds required for clustering different particles. At $N > N_{min}$, we observed localization of particles, regardless of their size and density, confirming that the data agrees well with the theory. Localization of particles in the stirred tank could take place when the impeller speed reaches a critical value that depends on the particle and fluid characteristics. Eq. (4) also suggests that the minimum speed required for particle clustering is linearly proportional to *1/L,* suggesting that clustering could take place in a larger tank with lower speed requirement. For example, we can concentrate 650μm particles at ~700rpm in our L = 0.2m small-scale tank whilst in theory, the same particles can be made to concentrate at just ~70rpm in a L = 2m full-scale tank. Our theory thus implies that clustering also happens in industrial-scale tanks. The dependence of clustering on Reynolds number is also shown in Fig. 13 (Supplementary materials).

3. Non-Ideal Systems

The previous sections focused on clustering of spherical particles in ideal Newtonian fluids. However, in a biorefinery, the carrier fluid is often non-Newtonian and the biomass involved is non-spherical. In this section, we show experiments extending applicability of our methodology to non-ideal systems.

3.1 Non-Newtonian Carrier Fluid

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High-viscosity fluids are hardly ever Newtonian in biorefineries or in minerals, chemicals or food industries. Non-Newtonian fluids are far more common.[22] Shear-thinning fluids (viscosity decreases with shear rate) are usually encountered. Here, we use a high-viscosity carboxymethyl cellulose (CMC) fluid in a large-scale mixing tank (L = 0.6m) to mimic the low-Reynolds-number processes in the biorefineries. The rheology of our working fluid is well described by a power law model: $\mu = m\dot{\gamma}^{n-1}\ and, \dot{\gamma} = k_s N$ where μ is the effective viscosity, $\dot{\gamma}$ is the shear rate, N is the impeller speed, Metzner-Otto constant k_s =12,[16] m = 10.6 Pa.s and n = 0.63 for the CMC solution we used in our experiment.

First particles were dispersed uniformly throughout the tank at a very high impeller rotation rate in the first instance followed by operating the tank under in laminar flow (Re=ρND^2/μ=14). From **Fig. 6** it is clear that particle localization takes place inside the ‘pseudo-cavern’, where fluid velocities are usually order of magnitudes larger than those found outside the ‘pseudo-cavern’. It should be noted that a, ‘pseduo-cavern’ is formed when shear-thinning fluids or even highly-viscous Newtonian fluids are agitated, where only inside a pseudo-cavern the fluid is in *active* motion.[23] The reduced shear-rate away from the impeller results in a significant drop in the fluid motion; therefore it is not surprising to see that the velocities of particles outside the cavern boundary are much smaller. In this respect, the highly inhomogeneous distribution of fluid velocities in non-Newtonian fluid is mainly responsible for the *incomplete* focusing of the particles. To achieve the complete focusing, a method using multiple impellers can be used to maximize the active flow region.

3.2 Irregular Particles

So far, all our results have been restricted to *ideal* particles, that is, spherical particles. A trial run was conducted to validate if clustering could take place for non-uniform particles, which are usually found in biorefineries. **Fig. 7** shows that we are also able to *concentrate* the non-uniform wood chips into the KAM regions. The wood chips had random shapes and sizes (a = 1.5±1mm). Second-generation biofuels are usually made of wood chips and other agricultural waste. It should be noted that the separation of biomass residue from the second-generation biofuels still presents a practical challenge in biorefineres. Here, our result strongly suggested that a conventional mixing tank could be adopted to resolve this problem.

4 Solid-Liquid Separation

To achieve the actual solid-liquid separation, we need to avoid impacts of pumping facility on the underlying flow structure (to maintain the KAM tubes), and a more concentrated particle roll will be preferred. **Fig. 8A-B** show that it is possible to concentrate all the particles into a single *tube* when the impeller is placed close to the tank bottom. The phenomenon is related to the formation of a single KAM region in the mixing vessel.[9] In this case, the tank-bottom effect eliminates any KAM tube below the impeller. The concentrated particle-tube potentially makes the collection of particles easier via pumping. On the other hand, the large portion of purified liquid above the particle-tube can also be easily drained out. **Fig. 8B-D** shows our preliminary result that removal of *~75%* purified liquid is achievable using a very simple setup, consisting of a mixing tank and a liquid pump. We speculate that draining holes on the side wall could be used to replace the pump. In this way, we can use the gravity force to drain out the viscous liquid, while significantly reduce the impact of the pumping facility on the underlying flow. We anticipate that impeller optimizations could further enhance the separation as well. A slurry pump can be adopted to suck out the remaining particles in the tank.

5 Discussion and Future Work

Like other separation methodologies,[26] the separation technique we demonstrated here can be used for separating solid particles of various sizes from medium/high-viscous liquids, biomass from liquids of biotechnological processes, *without* any dilution, whereby the solid particles of various sizes cluster into the specific regions. We anticipate that our separation technique will find utility in many fields where viscous liquids are encountered, as for example, in the production of: celluloid, fats and oils, cellulosic fibers, cellulose ethers, gelatine and starch.[24]

In practice, the tanks can be connected in series to ensure a high-throughput separation, while parallel connection can ensure a continuous separation (illustrated in **Fig. 9**). The proposed design was based on the following considerations: 1) The design is applicable to separating solid particles from viscous liquid for *difficult-to-separate suspensions*; 2) The multi-stage design enhances the particle/liquid recovery; 3) Utilizing a single-tube minimizes the impact of additional pumping on the underlying flow pattern, making the particle-tube more stable; 4) The single-tube also maximizes the volume of purified liquid, ensuring an easier collection. Further experiments are required to gauge the benefits of using multi-stage tanks.

The versatility of agitated tanks makes them the most popular mixing equipments in the chemical, biological, petrochemical and biotechnological processes. The clustering of particles that we found in stirred tanks is rather surprising. These tanks have been in constant use over a number of centuries but this particle-clustering phenomenon has never been reported. While the main intention of this article is to propose a novel separation technique based on a new physical phenomenon, we also urge engineers, biotechnologists and operators to pay particular attention to the potential consequences of such phenomenon in tanks being used for mixing purposes. It should be noted that the requirements to generate this solid-liquid separation are easily satisfied. The highly inhomogeneous particle distribution due to the clustering effect appearing in the mixing tank could result in the production of large amounts of undesired by-products, leading to adverse process economics. The lack of material exchange between the concentrated particle-tubes and the remaining fluid/slurry would certainly lead to poor effectiveness of an agitated reactor.

The work is obviously still at the preliminary stage. *The focusing of particles* is dependent on many factors, such as the geometrical design (impeller type, vessel shape and size, and impeller location, number of impellers), operating conditions (impeller speed) and feed slurries (particle size, particle concentration and slurry rheology, slurry viscosity, residence time). Further experimental and theoretical studies must be conducted to find the optimal geometrical conditions for the separation taking into account both slurry conditions and residence time requirements. It should also be noted that our method has been demonstrated to be successful in concentrating all small particles in a highly viscous fluid (~85 Pa.s) in our preliminary studies. We anticipate that making an optimized impeller could compensate for smaller particle inertia, thus we speculate that the clustering of extremely small particles (e.g. ~few microns) in viscous fluids may also be possible by using this novel mechanism.

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More experiments are also required to test our methodology in the more complex systems encountered in biorefineries. In particular, the solubility of components on separation theory will need to be validated. The intention of this communication is to propose a new methodology for solid-liquid separation. The detailed studies on impacts of slurry properties and operational conditions on separation will be explored in a separate paper.

6 Concluding remarks

In this article, we proposed new physical mechanism for solid-liquid separation in ideal and non-ideal stirred systems. We found that, regardless of flow rheology and particle shape, under quite general but controllable circumstances, some regions in the laminar tank can be made to concentrate particles. Our preliminary results in solid-liquid separation using this novel approach exhibit great potential in a number of areas, particularly in biotechnological processes, where solid-liquid separation is often difficult. We anticipate that engineering approaches along the lines we suggest can greatly enhance the efficiency and effectiveness of bio separations.

Materials and Methods

The mixing system consists of a cylindrical vessel (without baffles) and a Rushton turbine. The specifications of the impeller and tank are shown in **Fig. 1A**. Glycerin (>99.7%) was used as the working fluid and its viscosity was maintained in the range: 0.80 to 0.85 Pa.s at room temperature. As shown in Table 1 we used six types of particles with $0.70 \leq \rho_p/\rho_f \leq 1.19$, $0.65 \leq a \leq 7$mm, where ρ_p is the particle density, ρ_f is the fluid density and a is the particle diameter. Laminar flow was maintained (*Re* < 150), and we produced the data in **Fig. 4** with $10^{-3} \leq \sigma \leq 10^{-2}$, where σ is defined in Eq. (1).

Supplementary Video

Clustering Instability is triggered in the vessel.

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Table 1
Spherical particles used in the study; clustering effect was found for all the particles listed below

Particle type	**Particle size (mm)**	**Particle Density (kg/m^3)**	**ρ_p/ρ_f**
Polystyrene	1.4±0.4	1000	0.80
Polystyrene	2.4±0.4	1000	0.80
Ion exchange resin	0.65±0.05	1220	0.97
PMMA	3.18±0.05	1180	0.94
Urea resin	5±0.05	1500	1.19
Rubber	7±0.02	888	0.70
Plastic Capsule	27±0.05	900	0.71
Wood chips	1.5±1	440	0.35

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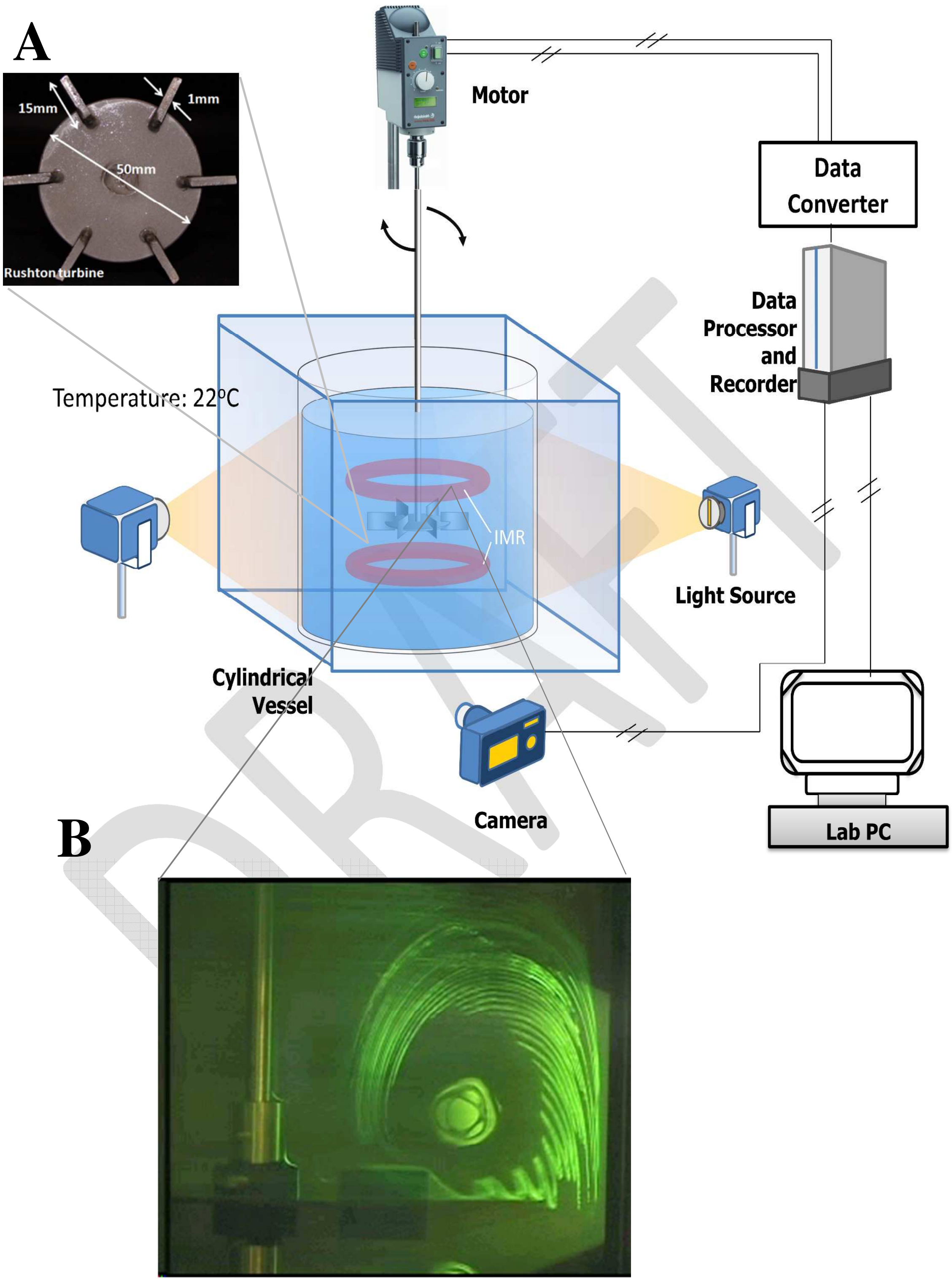

Figure 1 Schematic illustration of experimental apparatus. This figure consists of A, the rig, impeller specification; B: the flow template in the stirred tank, chaotic flow regions, surrounding the *KAM* tubes[25].

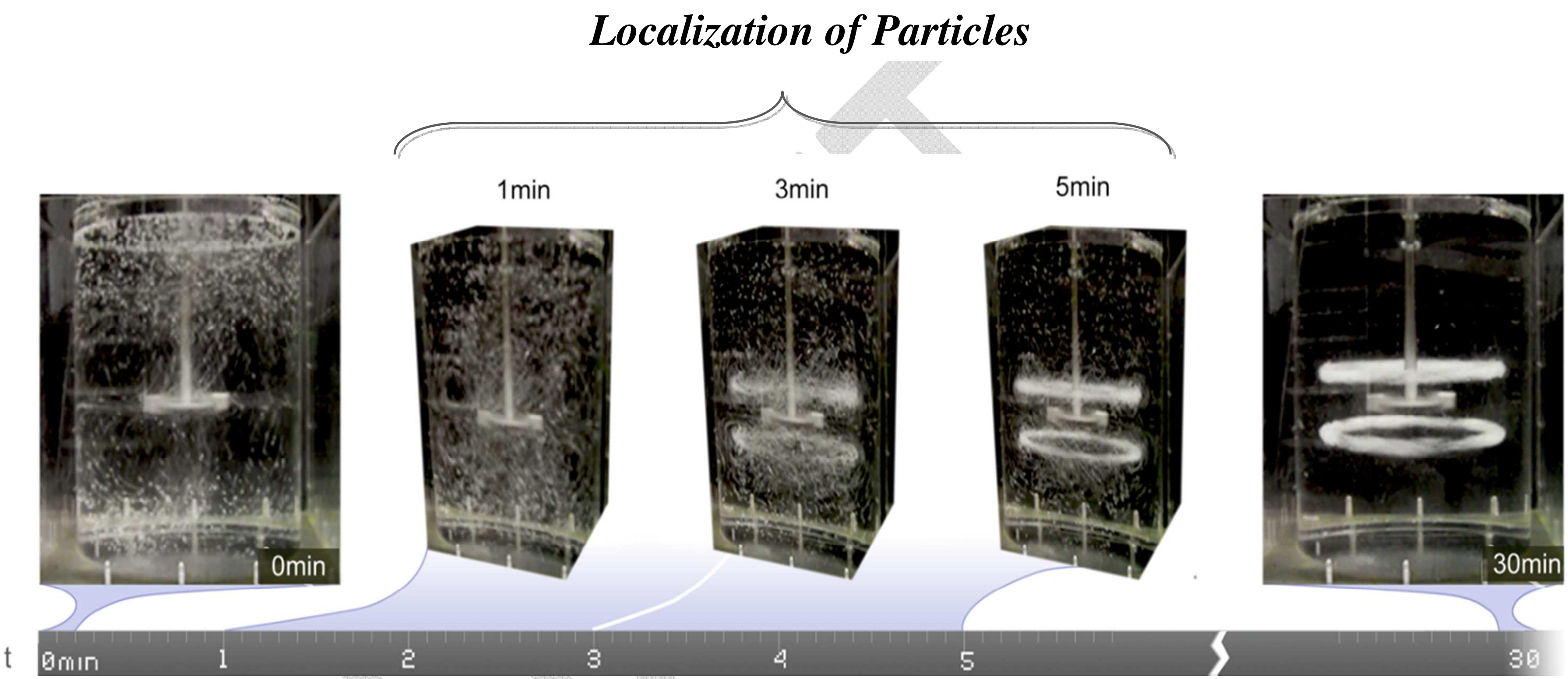

Figure 2 Localization of inertial particles in a mixing tank.

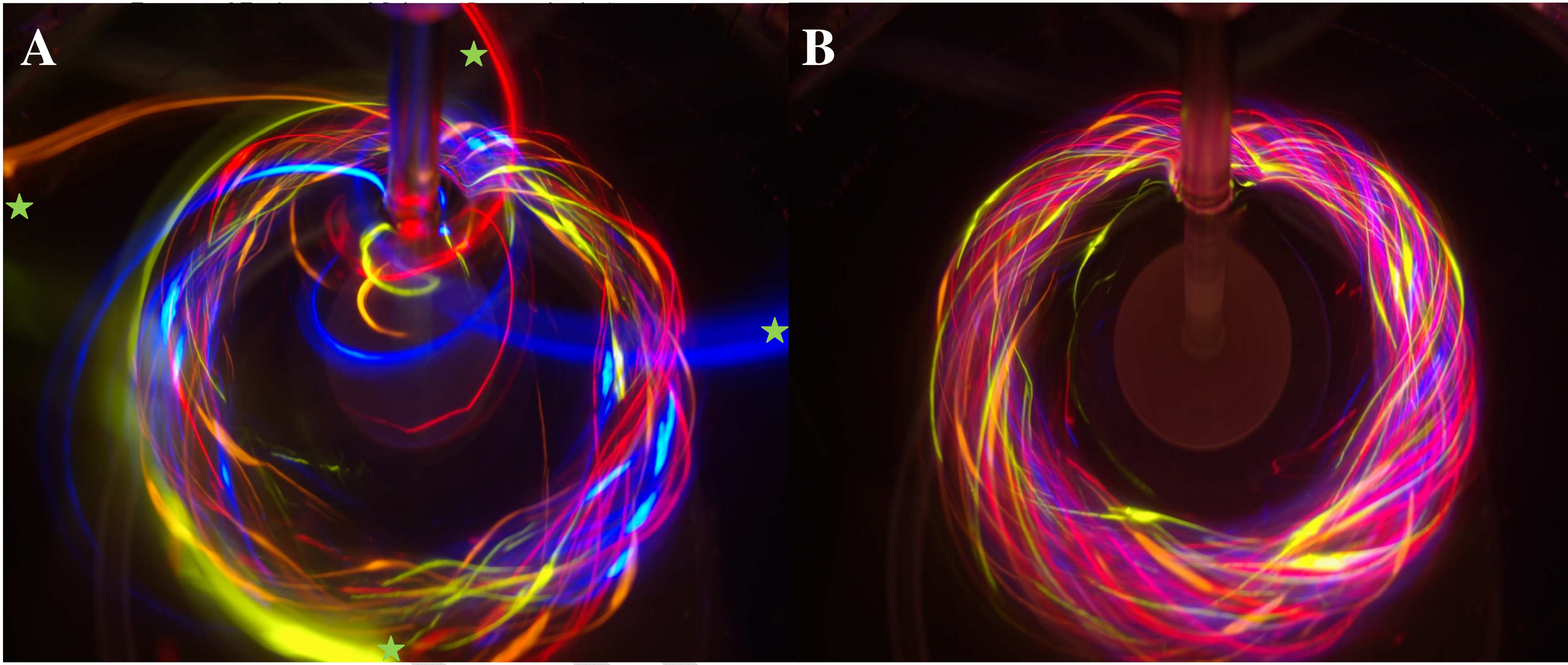

Figure 3 Visualization of particle trajectories. **A** localization of particles, inserted at the liquid surface, exposure time=30s, N =700rpm, Re=85, St=2.152. Stars indicate where the particles were released at the liquid surface. **B** Particle trajectories inside the *separated flow region*, exposure time=30s, N =700rpm, Re=85, St=2.152

Energy and Environmental Science (Communication)

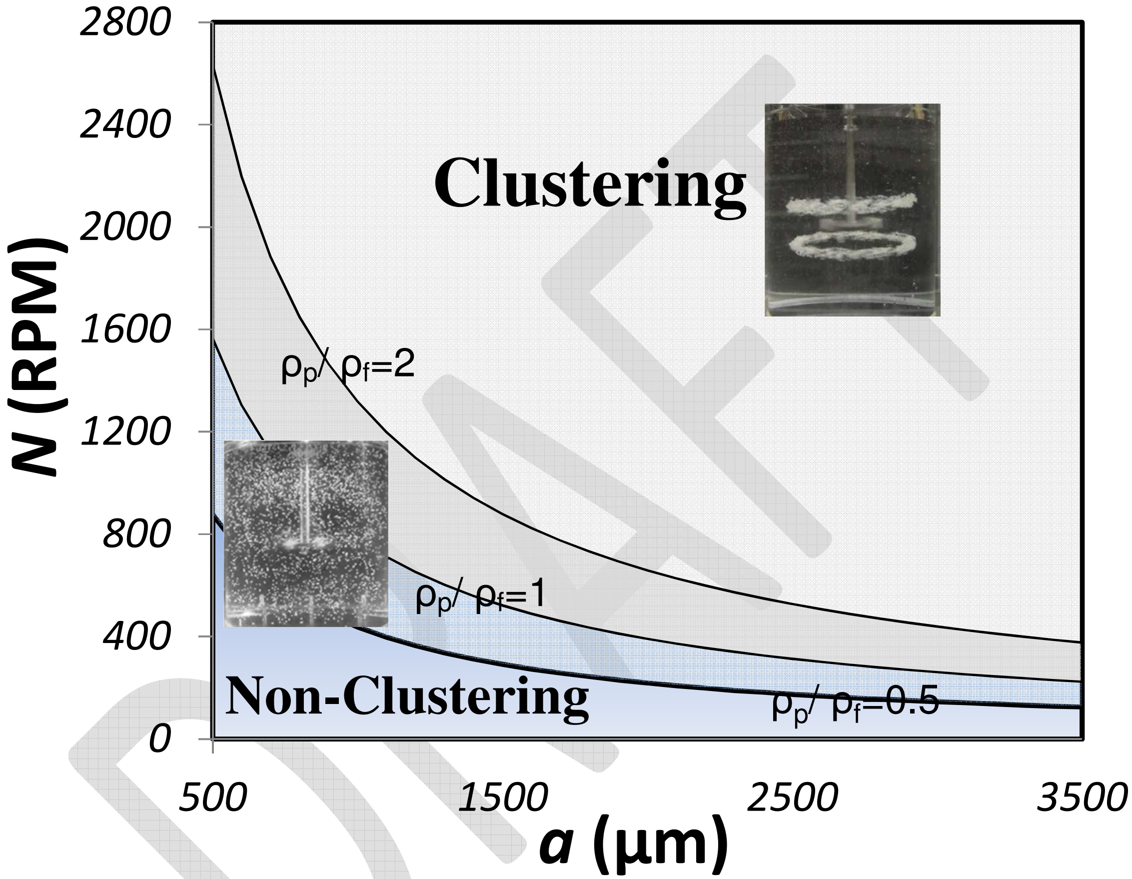

Figure 4 Theoretical critical speed required for particle clustering. (Tank diameter=0.20m, viscosity=0.85 Pa.s.)

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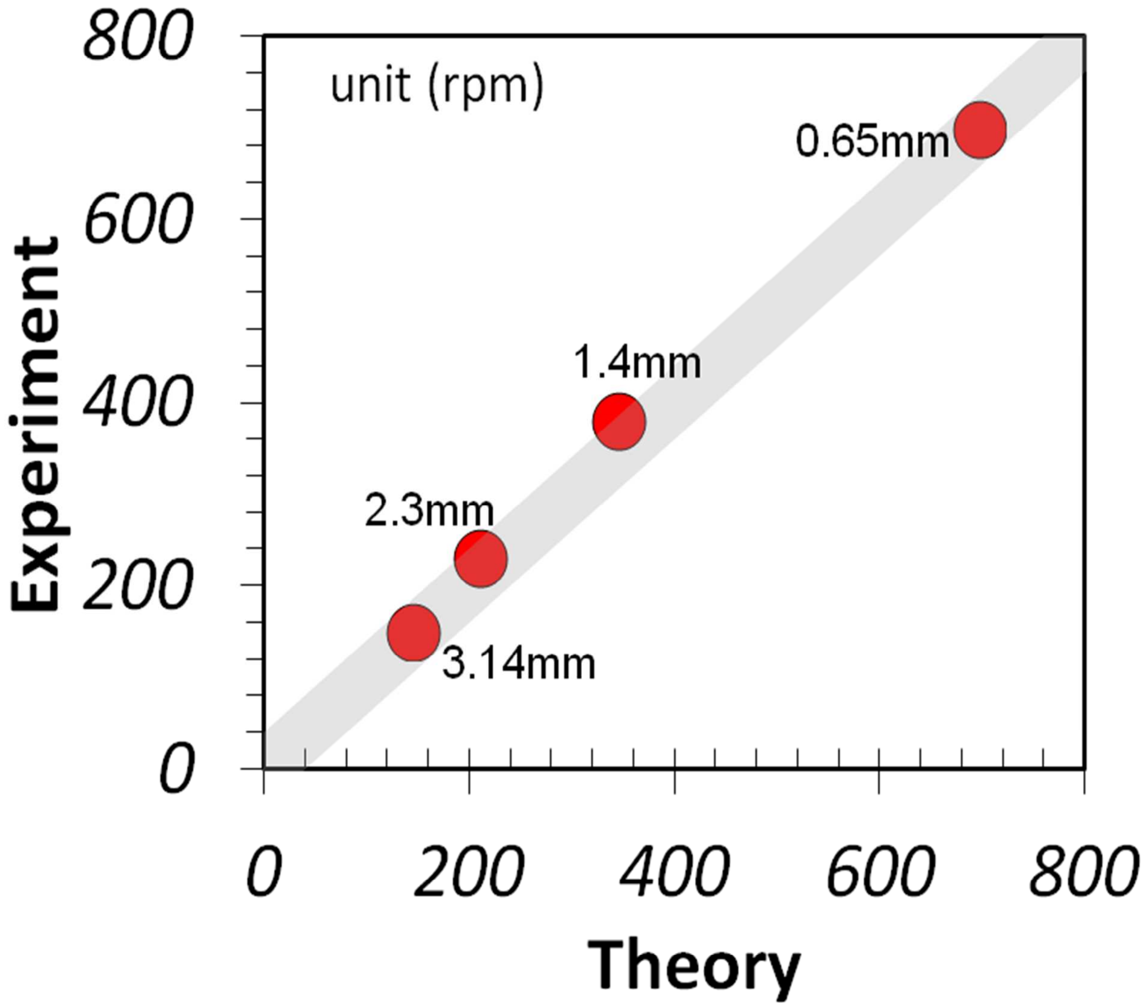

Figure 5 Theory vs. experiment. (Critical impeller speed requirement, N_{min}, unit: rpm.), the densities of the particles are given in Table 1.

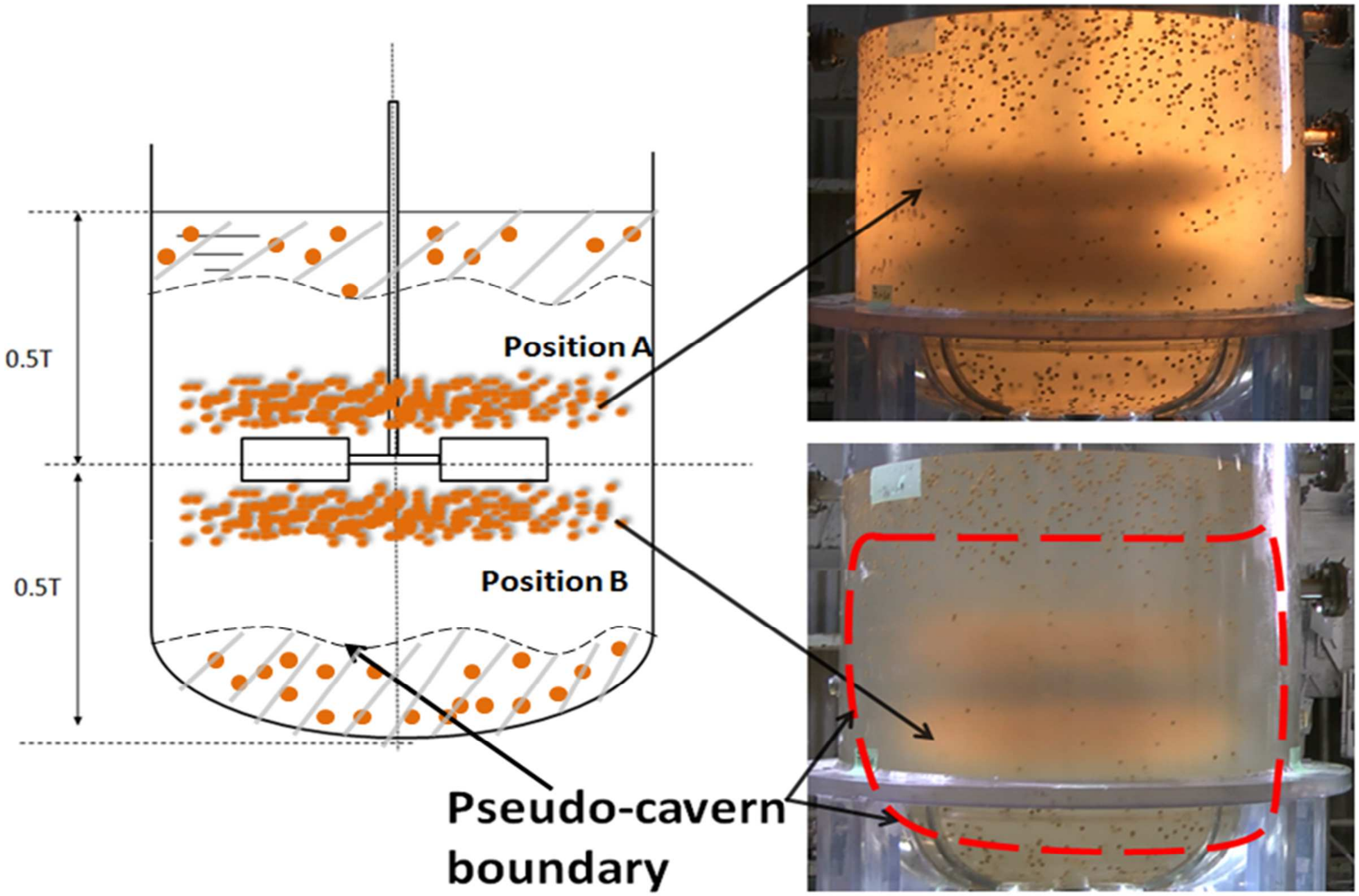

Figure 6 Clustering of particles in viscous shear-thinning fluid, in a large agitation vessel (L = 0.60m). Red dash line in the photo shows the pseudo-cavern boundary. Liquid density: 1001kg/m^3. N=60rpm, *Re*=14. D/T=0.40. Particle size: 6mm, particle density: 1039 kg/m^3. (A large impeller can potentially localize all the particles in this situation)

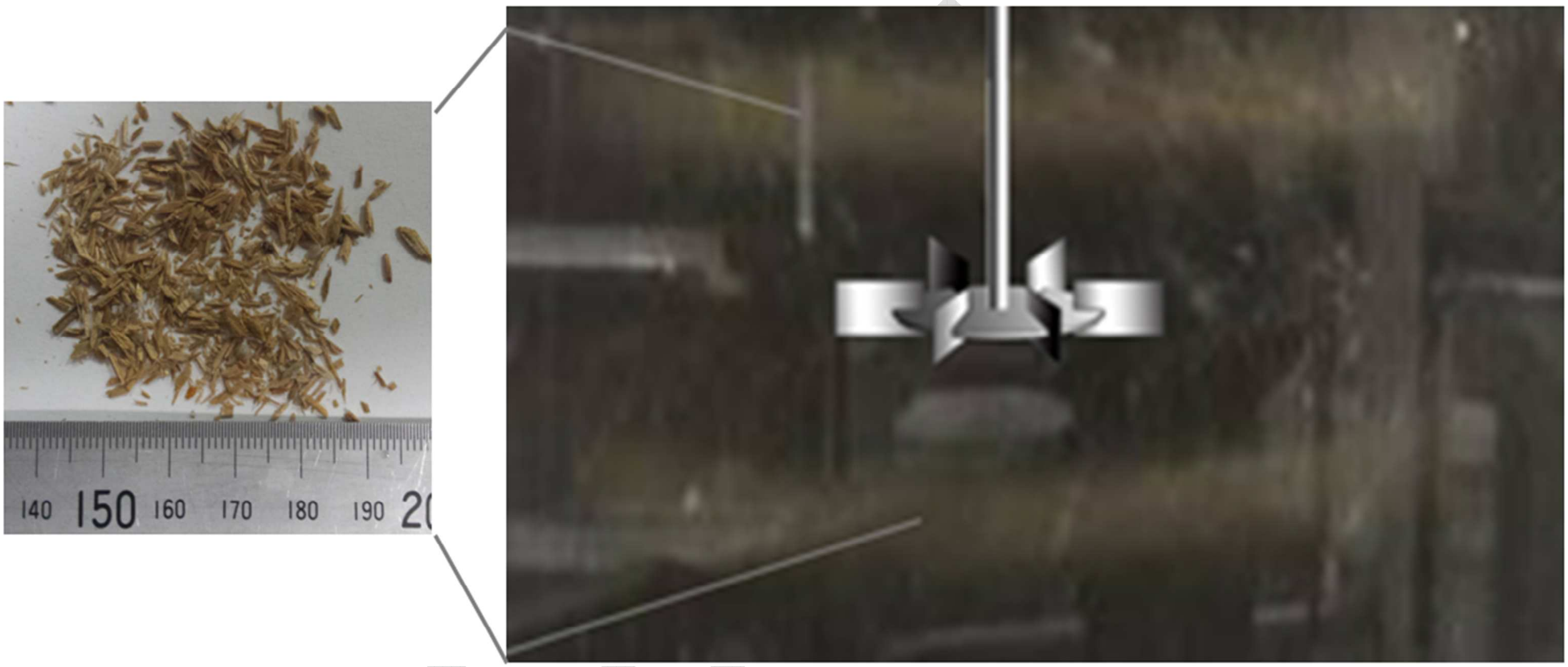

Figure 7 Clustering of non-spherical wood chips for second generation biofuels. Viscosity of working Fluid: 0.85 Pa.s. Impeller speed: 1100rpm. Re=133.

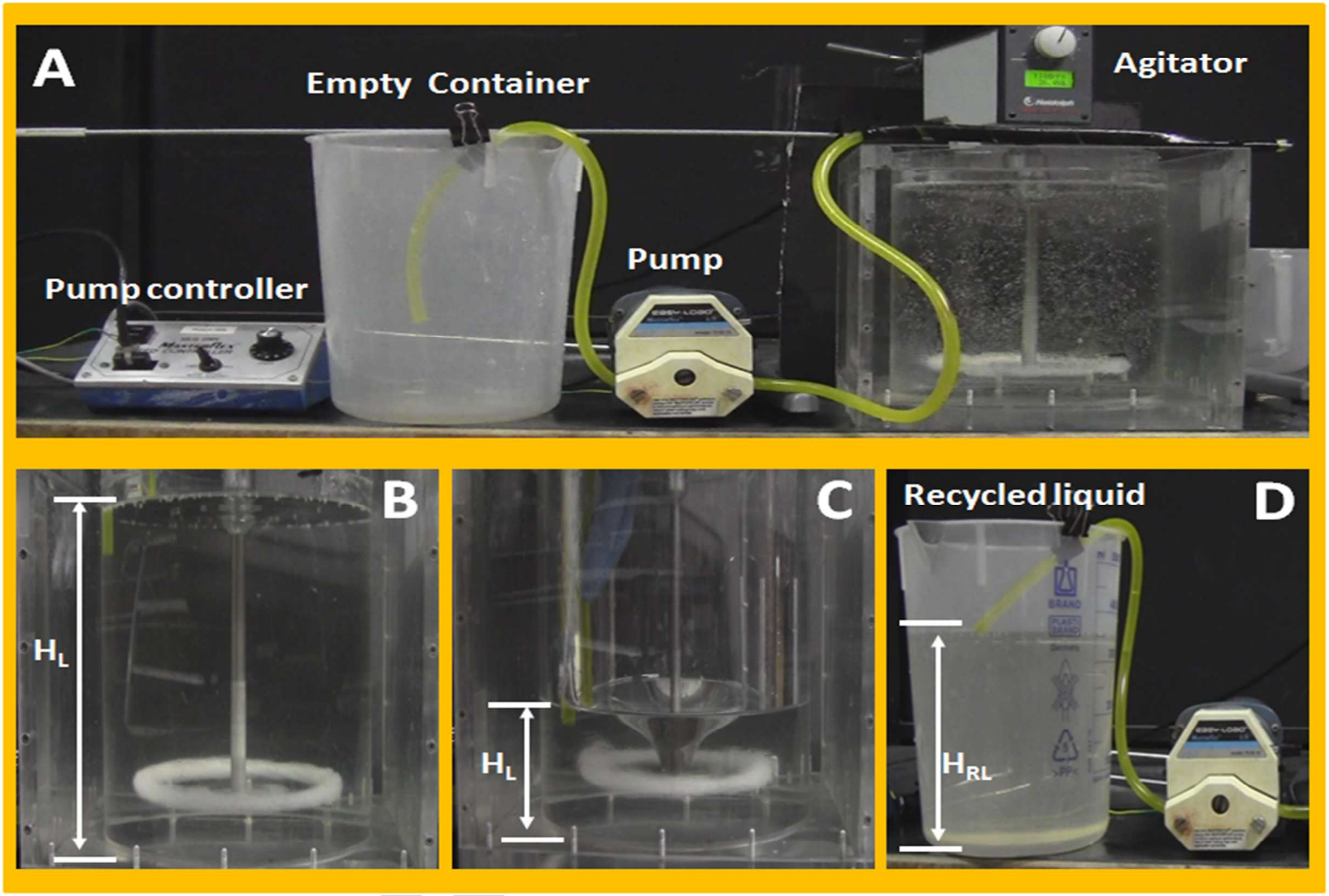

Figure 8 Removal of viscous liquid from the solid-liquid suspension. ~ 75% (v/v) purified liquid was attained in the experiment. A: experimental setup; B: concentration of *all* particles; C: removal of liquid from the system; D: recycled (clean) liquid stored in the container. H_L: liquid height in the agitator; H_{RL}: recycled liquid height.

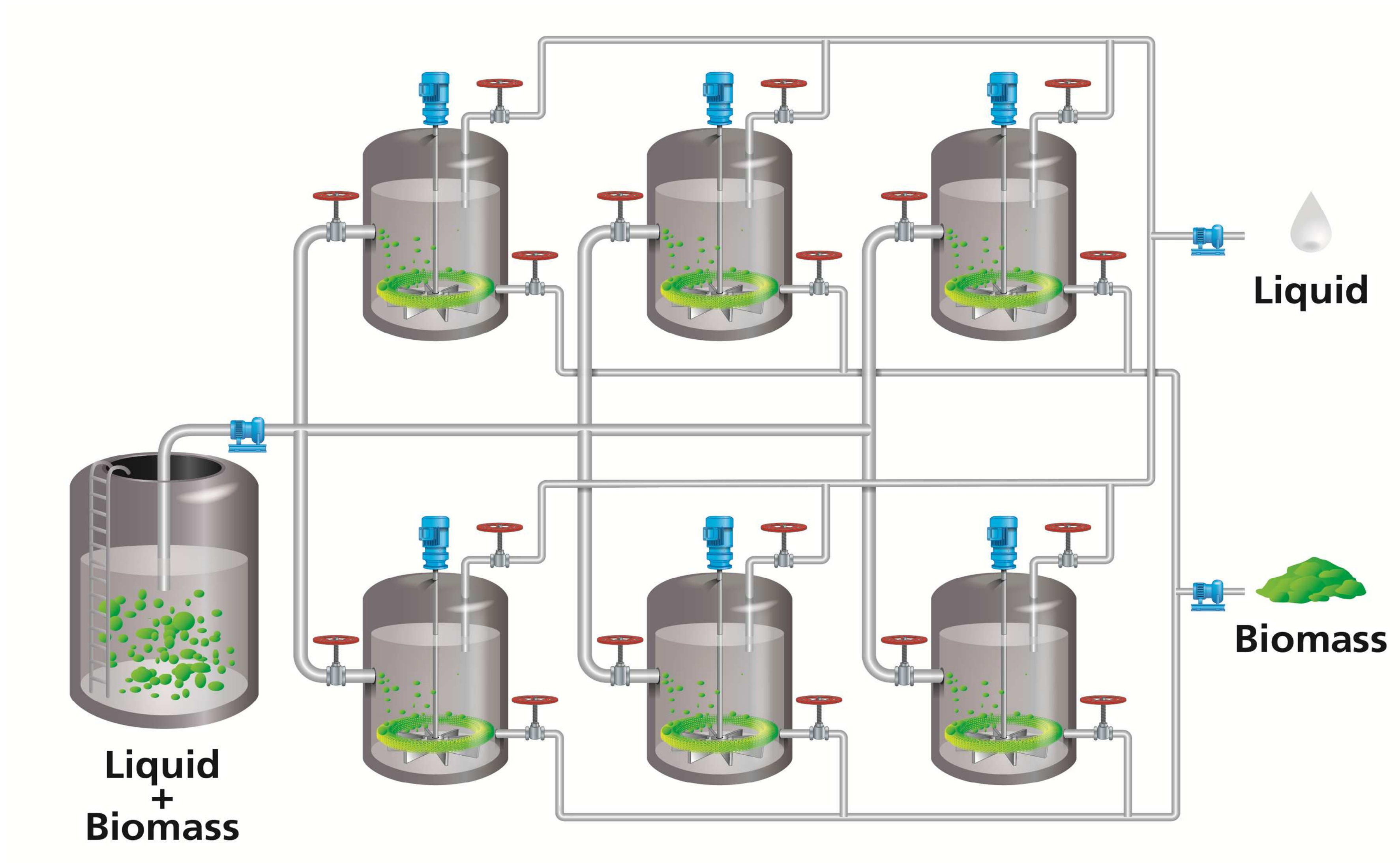

Figure 9 A conceptual design for biomass-liquid separation applied in the biorefinery

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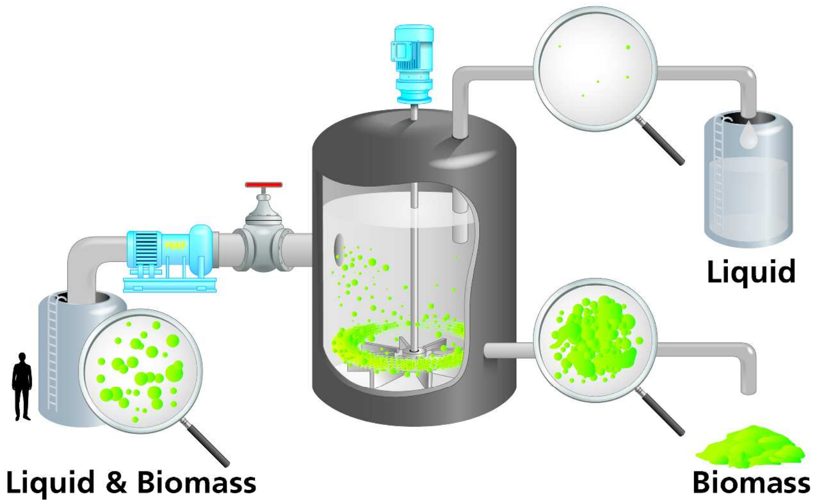

particle-flow-instability is manipulated for solid-liquid seperation
297x209mm (300 x 300 DPI)