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Transient Localized Rotating Structures in a Suspension of Highly Thermophilic Nanoparticles

Marina Carpineti, Stefano Castellini, Andrea Pogliani and Alberto Vailati*

Dipartimento di Fisica "A. Pontremoli", Università degli Studi di Milano, Milano, Italy

A thermophilic suspension of nanoparticles heated from below exhibits a complex stability diagram determined by the competition between the stabilizing flux of nanoparticles induced by thermophoresis and the destabilizing flux determined by thermal convection. We investigate Rayleigh-Bénard convection in a suspension of highly thermophilic nanoparticles with large negative separation ratio $\psi = -3.5$ heated from below. We show that transient localized states appear in the range of Rayleigh numbers $2200 < \mathrm{Ra} < 3000$. These states rotate rapidly around their axis and gradually shrink in size until the system returns in a purely conductive state. We discuss how these states are originated by the travelling waves arising from the competition between Rayleigh-Bénard convection and the solutal stabilisation of the sample.

Keywords: thermophilic nanoparticles, Rayleigh-Bénard convection, travelling waves, localized structures, convectons

1 INTRODUCTION

In the presence of a density stratification, a layer of fluid exhibits a rich phenomenology determined by the action of gravity that can lead to oscillations and wave propagation [1, 2]. A typical case is represented by a single component fluid heated from below, where the stratification is determined by the thermal dilation of the fluid, which can transfer heat either in a conductive or in a convective regime. The stability of the system is parametrized by the dimensionless Rayleigh number $\mathrm{Ra} = \frac{\alpha g \Delta T h^3}{\nu \kappa}$, which quantifies the applied thermal stress. Here α is the thermal expansion coefficient, g the acceleration of gravity, ΔT the temperature difference, h the thickness of the layer, ν the kinematic viscosity, and κ the thermal diffusivity. The transfer of heat is parametrized by the Nusselt number Nu, which represents the ratio between the heat transferred by the fluid and the one transferred by conduction only. For a layer of fluid of infinite aspect ratio $r = \Phi/h$, where Φ is the diameter of the layer of fluid, the transfer of heat is conductive (Nu = 1) when the Rayleigh number is below the threshold value $\mathrm{Ra}_c = 1708$, while it occurs by convection (Nu > 1) above the threshold (**Figure 1**). The addition of a second component to the fluid dramatically alters the stability of the layer. More in detail, the temperature gradient imposed to the fluid gives rise to a non-equilibrium mass flux determined by the Ludwig-Soret effect: $j = -\rho D [\nabla c - c (1 - c) S_T \nabla T]$, where ρ is the density of the sample, D the diffusion coefficient, c the weight fraction concentration, and S_T the Soret coefficient. As a result, the density profile inside the sample becomes affected both by the vertical temperature and concentration profiles of the fluid. The case of a suspension of thermophilic particles heated from below is particularly interesting, because in this case the stabilizing flux of nanoparticles induced by thermophoresis competes with the destabilizing flux determined by thermal convection. The relative weight of these two contributions is expressed by the separation ratio $\psi = \Delta \rho_s / \Delta \rho_T$, which represents the ratio between the density differences $\Delta \rho_s$

FIGURE 1 | Stability diagram. Nusselt number as a function of Rayleigh number for a single component fluid (liquid Helium, triangles) and LUDOX^{TM} colloidal silica nanoparticles in water at a concentration of 4.0% w/w (circles). The colloidal sample exhibits bistability, represented by the co-existence of a stable convective branch (full circles) and a stable conductive branch (open circles). The dashed horizontal line represents the purely conductive regime (Nu = 1), while the vertical dotted line is the threshold Ra_c = 1708 for Rayleigh-Bénard convection in a single component fluid. Transient rotating structures are observed in the range of Rayleigh numbers 2000 < Ra < 4000.

and $\Delta\rho_T$ determined by solutal and thermal expansion, respectively. In the case of a separation ratio in the range $-1 < \psi < 0$ this competition determines the onset of travelling waves, accompanied by the presence of localized pulses of convection, localized states and convectons [3–14]. The case $\psi < -1$ is particularly interesting, because in this case the stabilizing effect determined by the Soret flux dominates, and the transfer of heat occurs in the conductive regime, irrespectively of the magnitude of the imposed temperature gradient. However, experiments performed on suspensions of strongly thermophilic nanoparticles with $\psi = -3.5$ and $\psi = -7.5$ have shown that the behaviour of the system is strongly affected by the initial conditions [15, 16]. When the temperature gradient is imposed gradually, nanoparticles migrate towards the bottom of the fluid and give rise to a density profile that completely stabilizes the layer of fluid against Rayleigh-Bénard convection. Under this condition, the transfer of heat occurs by conduction. Conversely, when the temperature gradient is imposed rapidly and the particles are initially dispersed uniformly, convective motions set in and, provided that the Rayleigh number is large enough, keep the particles dispersed and convection lasts indefinitely. The theoretical investigation of the stability of a strongly thermophilic colloidal suspension with large negative separation ratio has shown that the competition between Rayleigh-Bénard convection and the stabilizing effect determined by the Soret effect leads to a transient oscillatory instability [17]. The analysis of the role of the settling of the nanoparticles showed that the presence of a sedimentation profile leads to the oscillatory onset of convection [18] and to the development of travelling waves [19, 20]. Under these conditions, an additional parameter affecting the stability of the system is represented by the sedimentation length $l_g = k_B T/(\Delta\rho V g)$, which characterizes the typical length scale of variation of the concentration profile (here $\Delta\rho$ is the density

mismatch between the particle and the carrier fluid, and V is the volume of the particle). The investigation of experimental conditions where the settling of particles is not strong and the sedimentation length l_g is comparable or larger than the sample thickness showed that a decrease of l_g is accompanied by a decrease of the convection threshold and of the frequency of neutral oscillations [21]. The analysis of the interplay between the combined effects of sedimentation, thermophoresis with negative separation ratio and convection led to the discovery of a new travelling wave solution, characterized by an anharmonic distribution of the vertical velocity across the sample layer [22]. Two dimensional simulations performed under conditions mirroring those adopted in experiments on a Hyflon MFA colloidal suspension [15], characterized by $\psi = -7.5$, showed recently that including the effect of gravitational sedimentation allows to achieve a quantitative estimate of the lifetime of the oscillatory flow as a function of Rayleigh number [20].

In this work we focus on the investigation of transient Rayleigh-Bénard convection in a suspension of highly thermophilic nanoparticles with large negative separation ratio $\psi = -3.5$ heated from below, under experimental conditions similar to those employed in theoretical studies [20–22]. We show that transient localized states appear in the range of Rayleigh numbers 2200 < Ra < 3000 after the sudden imposition of a thermal gradient to a suspension of uniformly distributed nanoparticles. These states rotate rapidly around their axis, and gradually shrink in size, until they disappear and the system returns in a purely conductive state. We provide a quantitative characterization of the wave number and angular velocity of the localized states and we discuss our results in comparison with previous theoretical models and simulations summarized above.

The study of spatially localized states is of great interest in the field of pattern formation, as they appear in a great variety of physical and biological systems [23, 24]. In the case of localized stationary convective states, they take the name of *convectons*, and there is a growing number of works studying them from a theoretical, computational and experimental point of view [10, 23, 25–27].

2 METHODS

2.1 The Sample

The sample chosen for the experiment is a colloidal suspension made by distilled water and LUDOX^{TM} TMA, a commercial colloid made of silica nanoparticle with an average diameter of 22 nm. In this work we have used concentrations of 4.0% w/w. The thermophysical properties are detailed in **Table 1**. This sample has been selected as a model system for the investigation of thermophoresis at the mesoscopic scale in the absence of gravity within the framework of the Giant Fluctuations and TechNES space projects of the European Space Agency [28, 29]. Within these projects, a series of experiments will be performed on the International Space Station to investigate the non-equilibrium fluctuations determined by the thermophoretic

TABLE 1 | LUDOX^TM TMA thermo-physical properties.

Quantity	Diameter (nm)	D	D_T	α	ν	β	S_T
Value	22	2.2e-7cm²/s	1.52e-3cm²/s	2.97e-4K⁻¹	8.18e-3cm²/s	0.57	−4.7e-2K⁻¹

process. Preliminary studies performed on Earth under isothermal conditions showed the presence of a peculiar relaxation dynamics of non-equilibrium concentration fluctuations, characterized by anomalous diffusion at large wave vectors [30]. For these reasons, it is very important to achieve a full understanding of the stability of this colloidal suspension in the presence of gravity.

2.2 Experimental System

The central element of the apparatus is a thermal gradient cell, consisting in a layer of fluid confined by two sapphire plates through which a thermal gradient can be applied. The heating elements are two ring-shaped Thermo Electric Devices (TED) thermally coupled to the sapphire plates. The other side of both the TED elements is in contact with a thermal reservoir, consisting of two annular aluminium chambers, inside which there is a steady flow of water at constant temperature. The purpose of this reservoir is to remove the excess heat. The cell is mounted on an optical bench in the configuration where the sample layer is horizontal, and is illuminated by a superluminous diode (Superlum SLD-261) with central wavelength 670 nm. The limited temporal coherence of the source avoids interference determined by multiple reflections of the beam on the surfaces of the cell windows. The sensor employed is a monochrome CCD Camera (JAI CV-M300) with a resolution of 576×768 pixels and a depth of 8 bit, operating at a frame rate of 10 images per second. An achromatic doublet with focal length of 30 cm, placed between the sample cell and the camera images a plane at distance $z = -90.6 \pm 0.5$ cm from the sample in a shadowgraph configuration that allows to visualize the self-organized structures generated by the convective motions. The camera is controlled remotely by a computer through a National Instrument PCI-1407 frame-grabber.

3 RESULTS

Heat conduction in fluids is greatly affected by the presence of colloidal particles that exhibit a thermophilic behaviour. In particular, the presence of even a small amount of thermophilic particles has a stabilizing effect when the suspension is heated from below and, as a result, the sample can transfer heat either in a conductive or in a convective regime, depending on the initial condition (**Figure 1**) [15, 16]. The conductive regime can be accessed by imposing slowly a temperature difference to the sample heating from below, so that the thermophilic particles accumulate at the bottom of the cell and stabilize the sample against Rayleigh-Bénard convection. The convective regime can be accessed by first heating the sample from above to determine the accumulation of nanoparticles at the top of the cell, and then suddenly reversing the temperature

gradient by heating from below. Under these conditions a Rayleigh-Bénard instability starts and the accumulation of the thermophilic particles at the bottom of the cell is prevented by the convective flow that keeps the particles mixed. Conversely, if the temperature gradient is reversed by slowly heating from below thermophoresis determines the rapid formation of a stable boundary layer at the bottom of the cell and the system ends up in the conductive regime.

The bistable heat transfer is currently a well established feature of strongly thermophilic nanoparticles [15, 16], but bistability is apparent only for Rayleigh numbers Ra > 4000. Below this value of the Rayleigh number and above the threshold for Rayleigh-Bénard convection Ra$_c$ = 1708 the system exhibits transient convective behaviour when heated from below, but eventually the stabilization determined by the particles dominates and the system enters into a purely conductive regime.

In order to study the transient convective regime in this range of Rayleigh numbers we have performed experiments according to the following procedure: i) The sample is heated from *above* for 1 h, applying a temperature difference $\Delta T = 10.5K$. This ensures that, because of the strong thermophilic behaviour of LUDOX^TM TMA, the colloidal particles diffuse towards the upper plate, and the sample enters a regime of solutal convection [31–33]. The time needed to enter this solutal convective regime has been determined empirically. Physically, it corresponds to the diffusive time $\tau_\delta = \delta^2/D$ needed for the formation of a thin—unstable—boundary layer of thickness δ at the top of the cell, and is affected by the size of the nanoparticles through their diffusion coefficient D. ii) The temperature gradient is abruptly reverted to a negative value. In our tests we used temperature differences of $\Delta T = -3.00$ K, $- 3.38$ K, $- 3.75$ K, $- 4.13$ K. iii) shadowgraph images of the sample are recorded with a frame rate of, approximatively, 10 images/second.

Under these conditions, when the sample is first heated from above at the beginning of the experiment, solutal convection destabilizes the suspension (**Figure 2A**) so that, when the gradient is inverted, a convective instability develops in the sample, even when the sample is below the threshold needed for the development of stationary Rayleigh-Bénard convection in the nanofluid (**Figure 2**). At the beginning, the convective patterns are the usual convective rolls that characterize Rayleigh-Bénard convection, spanning all the area of the cell. After some time, the patterns begin to rotate around the vertical axis (clockwise or anticlockwise, indifferently). Subsequently, convection begins to die out starting from the borders of the cell, giving rise to the formation of a single rotating localized pattern. Eventually, the rotating pattern shrinks in size, until it disappears completely and the sample reaches the stationary conductive state. The duration of the entire phenomenon, measured from the inversion of the gradient, is of the order of
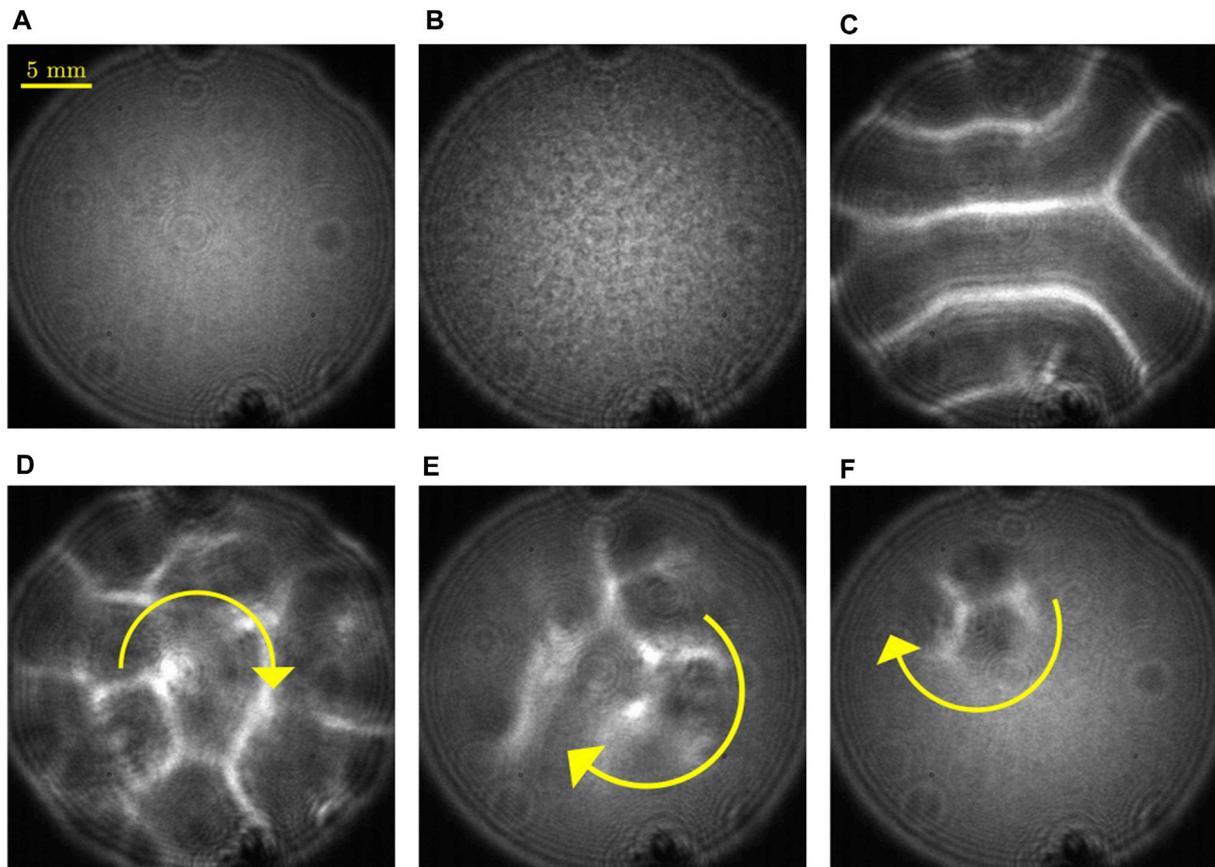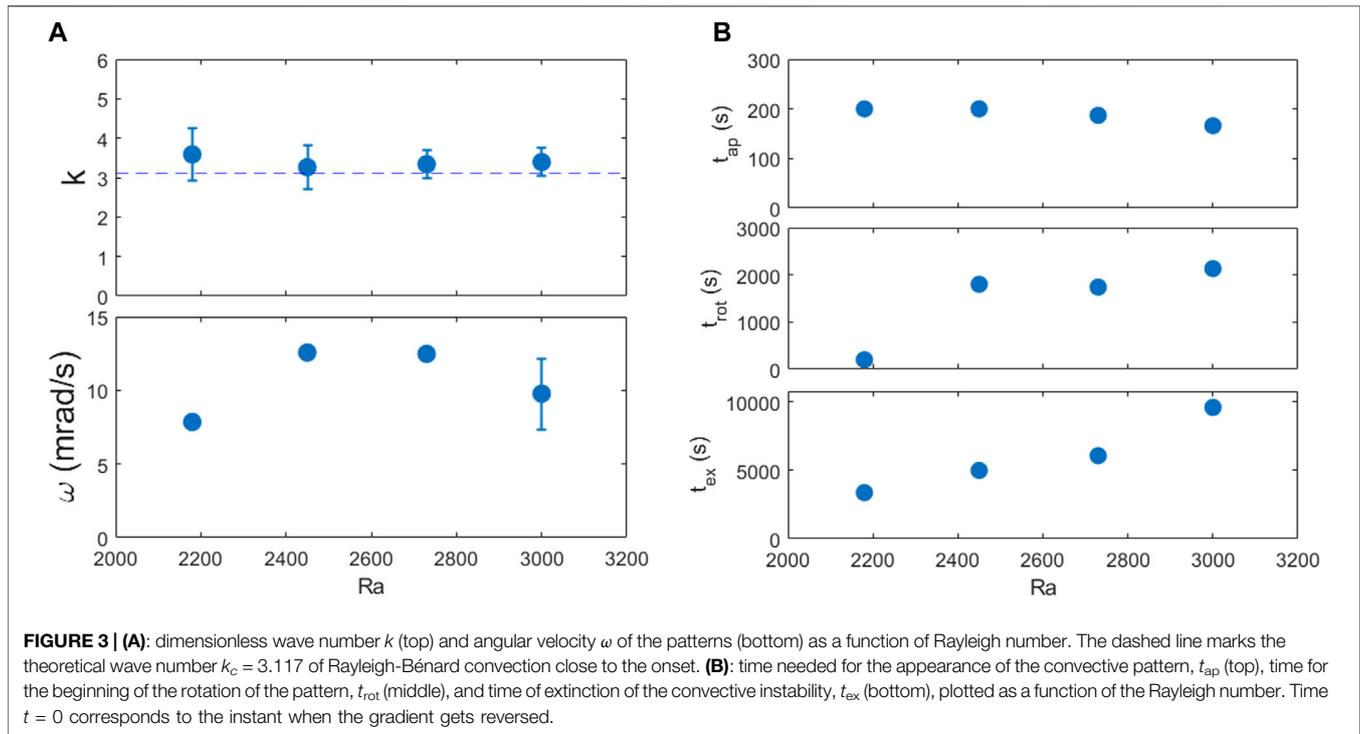
FIGURE 2 | Image sequence representing the evolution of the convective instability in a 3.1 mm thick layer of LUDOX^{TM} TMA 4% w/w. **(A)** at the beginning, heating from above with a temperature difference of 10.5K, no macroscopic motions are present; **(B)** after a time of the order of tens of minutes solutal convection develops in the cell; **(C)** after 1 h, the temperature difference is inverted and set to -4.13 K. In, approximately 3 min thermal convective rolls appear in the sample; **(D)** the rolls, after a time that depends on the Rayleigh number, start to rotate; **(E)** they die out starting from the borders; **(F)** they gradually shrink in size, until they finally disappear.

1–3 h. The same phenomenology is observed for all temperature differences studied.

By looking at sequences of images, we measured some characteristic parameters of the localized states as a function of Rayleigh number. The interesting quantities are the wave vector k of the patterns and their angular velocity ω, and the typical timescales t_{ap} needed for the appearance of the convective structure, t_{rot} needed for the rotation of the pattern to start, and t_{ex} needed for the patterns to disappear. The results are summarized in **Figure 3**.

The characteristic wave vector k is compatible with the value 3.117 predicted for Rayleigh-Bénard convection close to the threshold. The angular velocity of the patterns has been determined from the visual observation of a large number of complete revolutions of the localized pattern (typically 10–20 revolutions). In selecting the maximum number of revolutions to consider for a reliable determination of the angular velocity we relied on the fact that the structure of the rotating pattern needed to be preserved, so that the motion of the spatial features of the patterns could be followed during their rotation. This method proved to be more effective and immune to errors than the automatic processing of the angular correlations.

The rotation of the patterns is very slow, a complete revolution taking a time in the range 500–800 s. The time t_{ap} needed for the appearance of the structures is of the order of 200 s, and slightly decreases as the Rayleigh number increases. The behaviour of the time t_{rot} needed for the rotation to start is of major interest, because it exhibits a variation of a factor 10 when the Rayleigh number is increased from 2180 to 3000, indicating that the range explored is extremely important and revealing. Indeed, in this range, as already noted, there is a competition between the Rayleigh-Bénard thermal convection and the stabilizing effect of the colloidal particles. This competition leads to the formation of travelling waves patterns, which, in a circular cell, become rotating patterns, due to the confinement determined by the lateral boundary. However, in the regime of subcritical Rayleigh numbers explored by us Rayleigh-Bénard convection prevails, and the stabilizing effect due to the accumulation of the colloid on the bottom of the cell is hampered by the continuous remixing of the particles. Under these conditions, the sedimentation of the particles is slowed down, and so are the appearance of travelling waves and the beginning of the rotation. The time needed for convection to disappear also exhibits a marked dependence on the Rayleigh number, and is in qualitative agreement with the

FIGURE 3 | (A): dimensionless wave number k (top) and angular velocity ω of the patterns (bottom) as a function of Rayleigh number. The dashed line marks the theoretical wave number $k_c = 3.117$ of Rayleigh-Bénard convection close to the onset. **(B)**: time needed for the appearance of the convective pattern, t_{ap} (top), time for the beginning of the rotation of the pattern, t_{rot} (middle), and time of extinction of the convective instability, t_{ex} (bottom), plotted as a function of the Rayleigh number. Time $t = 0$ corresponds to the instant when the gradient gets reversed.

results of simulations on suspensions of thermophilic nanoparticles with large negative separation ratio heated from below [20].

4 DISCUSSION

Our system is different from most of those that, to our knowledge, have been previously characterized experimentally or theoretically and by means of simulations. In fact, a large number of studies has been devoted to the investigation of water-ethanol mixtures heated form below. Depending on the concentration of ethanol, this system can exhibit a negative separation ratio $\psi > -1$, leading to the oscillatory onset of a convective instability when the fluid is heated from below. Water-ethanol mixtures exhibit a multistable heat transfer at sufficiently small supercritical Rayleigh numbers [3, 5, 6, 8, 11, 12, 14], where depending on the initial condition and on the history of the system the transfer of heat can either occur by conduction or by steady overturning convection, similarly to the bistable behaviour reported by us. The investigation of transient behaviour during the onset of convection shows the development of localized pulses of travelling-wave convection in 1D [9, 23] and 2D geometries [10]. Under supercritical conditions, localized rotating structures develop, and gradually expand until they reach the boundaries of the cell [10, 27]. Conversely, bringing back the system in a subcritical condition after the onset of convection determines the formation of rotating structures that gradually shrink in time until they disappear [13, 34].

All these works deal with binary fluids with a negative separation ratio $-1 < \psi < 0$, a condition where the thermal contribution to the density variation is larger than the solutal one. At variance, our system is a colloidal suspension of nanoparticles and has a separation ratio $\psi = -3.5$, so that the solutal contribution to the density profile largely dominates the thermal one. Indeed, many of the solutions studied in the cited works are travelling waves that don't die out, whereas the instability that we observed in the end disappears, notwithstanding the fact that the system is under supercritical conditions, due to the stabilization determined by the highly thermophilic particles.

The case of thermal convection in a suspension of thermophilic nanoparticles with a large negative separation ratio $\psi = -10$ has been investigated theoretically by Ryskin and Pleiner [17]. They studied the linear and nonlinear behaviour starting from a suspension with an initially uniform distribution of nanoparticles. They found that at small supercritical Rayleigh numbers Ra < 1840 the fluid exhibits a transient oscillatory instability, while at higher Rayleigh numbers the system exhibits bistability, characterized by the presence of a stationary instability and of a stable conductive state. The phenomenology reported is qualitatively very similar to the one reported by us, but the threshold for the transition from a transient oscillatory instability to a stationary one is much larger in our experiments.

Cherepanov and Smorodin have performed a detailed theoretical investigation of the stability of a colloidal suspension heated from below and with negative separation ratio $\psi = -0.8$, taking into account not only the Soret effect but also the effect of sedimentation induced by gravity [21,22]. The sedimentation length l_g becomes in this case an important parameter: for $l_g \ll h$ the colloid gets almost entirely accumulated at the bottom boundary, and the system behaves as a single component fluid. When $l_g \geq h$, the

threshold for Rayleigh-Benard convection gradually increases to values of the order of several time the threshold for a single component fluid. While the parameters adopted in these studies partially mirror the ones of our sample, the separation ratio $\psi = -0.8$ adopted by Cherepanov and Smorodin corresponds to the condition where the Rayleigh-Bénard convection dominates over the solutal stabilization. By contrast, in the case investigated by us the sedimentation length is moderately high, $l_g/h = 20$, but the strong negative separation ratio $\psi = -3.5$ determines an accumulation of particles at the bottom boundary dominated by the Soret effect and affected only marginally by sedimentation.

Recent experimental studies have outlined the potentiality of the bistability of nanofluids with large negative separation ratio, like the one we studied in this work, to actively control heat transfer by switching between the conductive regime and the convective one (and vice-versa) exploiting the thermophilic behaviour of the nanoparticles [15, 16]. Cherepanov and Smorodin performed 2-dimensional simulations of thermophilic nanofluids with a separation ratio $\psi = -7.5$ heated from below mirroring the one used in these experiments. This study has been able to reproduce accurately the following quantitative aspects observed in experiments performed on a Hyflon MFA suspension of nanoparticles at a concentration of 4.0% w/w [15]: i) The presence of transient and stationary travelling wave regimes separated by a threshold Rayleigh number $Ra^* \approx 3400$; ii) the modifications determined by the presence of nanoparticles on the bifurcation diagram of the Nusselt number as a function of Rayleigh Number; iii) the diagram of the lifetime of travelling waves in the transient regime as a function of Rayleigh number.

Simulations along the same lines of the ones performed by Cherepanov and Smorodin on suspensions of Hyflon MFA nanoparticles should be able to describe effectively the experimental results provided by us in this publication, in particular the bifurcation diagram (**Figure 1**), and the lifetime of transient convection (**Figure 3**) in a suspension of Ludox nanoparticles.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

AV conceived the study and wrote the paper. AP performed measurements. MC and AV coordinated research. All the authors analysed results and contributed to the manuscript.

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