Advection kinetics induced self-assembly of colloidal nanoflakes into microscale floral structures

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Abstract

The article explores the governing role of the internal soluto-thermal hydrodynamics and advective transport within sessile colloidal droplets on the self-assembly of nanostructures to form floral patterns. Water–acetone mixture and Bi$_2$O$_3$ nanoflakes based complex fluids are used as the experimental liquids. Micro-liter sessile droplets are allowed to vaporize and the dry-out patterns are examined using scanning electron microscopy. The presence of distributed self-assembled rose-like structures is observed and is postulated to be formed by the hydrodynamic interactions within the drying droplet. The population density, structure and shape of the floral structures are noted to be dependent on the binary fluid composition and nanomaterial concentration. Detailed microscopic particle image velocimetry and infrared thermography analysis is undertaken to qualitatively and quantitatively describe the solutal Marangoni advection within the evaporating droplets. It has been shown that the kinetics, regime and spatial distribution of the internal flows are dominantly responsible factors towards the advection influenced clustering, aggregation and self-assembly of the nanoflakes. In addition, the size of the nanostructures and the viscous character of the complex fluid are also noted to play dominant roles. The resulting interplay of hydrodynamic behavior, adhesion and cohesion forces during the droplet dry-out phase, and thermodynamic energy minimization leads to formation of such floral structures. The findings may
have strong implications towards modulating micro-hydrodynamics induced self-assembly in complex fluids.

(Supplementary material for this article is available online)

Keywords: Sessile droplet, Marangoni advection, micro-hydrodynamics, self-assembly, pattern formation, complex fluids

(Some figures may appear in colour only in the online journal)

1. Introduction

Evaporative self-assembly and pattern formation in colloidal complex fluid droplets has been an area of academic interest and applications, especially in modern day nanoscale technologies and engineering. These include design and development of superlattices (Chen et al. 2011, Romano and Sciortino 2011), engineering building proteins of life (Tkachenko 2002), nano-biotechnology (Ng et al. 2013), nanoscale origami and structure modulation (Biancaniello et al. 2005, Ahn et al. 2010), crystal engineering for optics and photonics (Zheng et al. 2007), supra-molecular drugs (Ariga et al. 2011, Drummond and Fong 1999), nanoscale lithography (Ozin and Yang 2001), nematic crystals (Mušević et al. 2006), and so on. Despite being an area innately associated with physical chemistry and chemical physics, the incontrovertible role played by fluid dynamics and thermo-solutal transport processes is often overshadowed in the studies. The pattern and structure assembly is typically governed by micro-hydrodynamics forces in conjunction with physico-chemical interactions, such as adhesion-cohesion (Ye et al. 2001), electro-magneto-thermal interactions (Demirörs et al. 2010, Swan et al. 2014) and/or chemical reactions at the microscale (Kang et al. 2007). Consequently, the role of micro-hydrodynamics and advection kinetics within such complex fluid droplets and the subsequent dry-out behavior that leads to such assembly requires thorough understanding. Recently, some aspects of evaporative transport (Harikrishnan et al. 2018), internal hydrodynamics (Jaiswal et al. 2018b) and Marangoni advection (Dhar et al. 2019) in complex colloidal droplets, and their roles on interfacial processes, have been explored. The modulation of such micro-hydrodynamics employing opto-thermal (Harikrishnan and Dhar 2018), electric (Jaiswal et al. 2018c, Jaiswal and Dhar 2019) and magnetic (Jaiswal et al. 2018a, Chattopadhyay et al. 2020) fields have also been discussed in literature.

However, the idea that the internal hydrodynamics within evaporating colloidal droplets may ultimately decide the nature of the deposition pattern and self-assembly, is often overlooked. The present article discusses the role of the internal solutal Marangoni advection and its kinetics in modulating the physical self-assembly of nanostructures within evaporating sessile droplets. Binary fluid compositions (Zhang et al. 2011) of water and acetone are employed to modulate the characteristics of the solutal advection hydrodynamics during evaporation. Bismuth oxide (Bi$_2$O$_3$) nanoflakes are used as the colloidal phase and their self-assembly is studied using high resolution scanning electron microscopy (HRSEM). Flake shaped nanostructures have been used as these have greater chances to form self-assembled morphologies than spherical nanoparticles. Physical clustering, agglomeration and packing assembly into microscale floral (rose) shapes are noted under different conditions. The number density and conformational characteristics of the flowers are explained based on the internal hydrodynamics. Microscopic particle image velocimetry (PIV) and infrared thermography is used to qualitatively and quantitatively understand the Marangoni advection kinetics. The strength
of micro-hydrodynamics, droplet lifetime and colloidal concentration are explored and self-assembly behavior is explained. The study may have strong implications on developing micro-scale soluto-thermo-hydrodynamics modulated colloidal patterning and assembly strategies.

2. Experimental materials and methodologies

The experimental details are described briefly and the detailed description on similar setup is available in reports by the author (Dhar et al 2018, 2019). Since the study involves HRSEM imaging, aluminum (Al) foil cut-outs (10 mm × 10 mm) are used as the electrically conducting substrates for the droplet evaporation process. The foils are cut with great caution (to prevent wrinkling and crumbling, which may lead to local patterning during the colloidal evaporation), cleaned with isopropanol and oven dried overnight. Similarly processed sterile glass cover-slips are used as the substrates for the PIV. The wetting behavior of both processed substrates are very similar (contact angle for water droplet range from 32° to 37°, the advancing contact angles range from 53° to 59°, and the receding contact angles range from 13° to 17°) and evaporating droplet lifetime for water is within 5%–7% and acetone droplets are within 2%–3%. Deionized water and acetone (>99.5% pure, AR, Sigma Aldrich) are used as test fluids. The Bi$_2$O$_3$ nanoflakes (size distribution in figure S1 (available online at http://stacks.iop.org/FDR/52/065506/mmedia), supporting information SI, Alfa Aesar, India, flake aspect ratio of ∼1–5, as per supplier) are dispersed in the base fluids by ultrasonic disruption (Oscar Ultrasomics, India). Bi$_2$O$_3$ concentrations of 0.005 wt%, 0.01 wt%, 0.025 wt%, 0.05 wt% and 0.1 wt% are studied. The base fluids are different proportions of water (W) and acetone (A), viz. W100 A0, W80 A20, W60 A40, W40 A60, W20 A80, and W0 A100 (all wt/wt%).

The nanocolloids are ultra-sonicated for 30 min, using a 750 W disruptor. The disruptor is programmed to operate at on-off cycles of 15 s and 15 s to prevent heating up, and evaporation of the colloids. In addition, the fluid vials are specially covered to prevent vaporization of the colloids. The fluid vials are also kept in cold water baths to prevent heating of the samples which may lead to excessive vaporization of the acetone during sonication. Since the colloids are in the dilute regime, the shelf-life stability is noted to be 5–7 d via visual inspection. This largely exceeds the evaporation time scales (~60 s) of the micro liter-droplets and hence it is safely assumed that aggregation and flocculation behavior do not play major roles towards the experimental observations. Since the water and acetone system is highly miscible; hydrodynamics due to localized density variations may be ruled out safely. A precision microliter chromatography syringe (Holmarc Opto-mechatronics, India) is used to dispense a 10 µl droplet on the foils, and allowed to evaporate within an enclosed acrylic chamber (temperature and relative humidity conditions were noted to be 30 ± 2 °C and 48 ± 4% for all experiments). The evaporation process is recorded at 20 fps using a CCD camera (Holmarc Opto-mechatronics, India) with long-distance-microscope lens, and the droplet life-times are assessed from image processing. The nanomaterial deposits on the Al foils are imaged using HRSEM and the assembled structures are counted per unit area.

For quantification of the internal advection, the droplets are placed on glass cover-slips and observed under a CMOS camera (60 fps, 5 megapixel resolution) integrated digital microscope (Holmarc Opto-mechatronics, India). In the present context the evaporation is not associated with heating the substrates, the glass cover-slips behave similar to the Al foils, since the equilibrium contact angles are noted to be similar on the two surfaces. The fluids are seeded with hydrophilic, 450–600 nm carbon particles (density ~ 1.6–1.8 gm cm$^{-3}$) for the PIV. The microscope is focused approximately at the horizontal mid-plane of the droplet and the
internal flow patterns are imaged at 30 fps with bright-field illumination. The images are post-processed using the open-source software ImageJ (Rueden et al. 2017), where initially the images are converted to color inverted binary, which transforms the seed particles to white pixels and the fluid regions to black. The velocimetry analysis is done in the open source code PIV-lab (Thielicke and Stamhuis 2014), where 1000 consecutive images are processed using four pass cross-correlation algorithm (consisting of 64, 32, 16 and 8 pixel interrogation windows). Conventional noise suppression and contrast enhancement algorithms are used to obtain better peak matching and high SNR. Typical resolution of the velocimetry is $\sim 150$ pixels $\text{mm}^{-1}$. Detailed description of the PIV methodology followed has been reported by the author (Dhar et al. 2018). The infrared thermography of the evaporating droplets is performed using an infrared camera (FLIR T650sc) at $640 \times 480$ resolution; at 30 fps and accuracy of $\sim 3\%$ of the thermal range of the experiments ($\sim 0.3 \degree\text{C}-0.4 \degree\text{C}$ in the present context).

3. Results and discussions

The colloidal patterns formed on Al foils are examined using HRSEM, and dried bed of distributed nano-flakes is observed. Under certain conditions (discussed subsequently), this dry-bed pattern is characterized by numerous localized distribution of microscale floral assemblies, typically composed of the larger nanoflakes stacked against one another. An illustration of a typical self-assembled ‘rose’ pattern is shown in figure 1. The smaller scale flakes form the bed of the deposition, whereas the larger flakes adhere by cohesion to form the floral structures. The bed deposition (where the flakes are deposited randomly and heterogeneously) is formed by the 40–60 nm flakes (refer figure S1, supporting information) and the floral patterns are formed by the 200–400 nm flakes. The mean overall size (base diameter) of such self-assembled flowers ranges between 500 nm and 1000 nm (figures 1 and 2(a)). The content of the acetone in the binary fluid colloid is noted to govern the symmetry of the assembled structures. Near the observed optimal water-acetone proportion (discussed later), the rose formations are observed to possess good symmetry (figure 1). On the contrary, acetone content far from the determined optimal point is noted to yield rose structures with mostly asymmetric topology (figure 2(a)), with additional clustering or fusion of two or more flowers. Such changes in morphology of colloidal deposit patterns due to changes in concentration have been reported in literature (Deegan 2000).

In the SEM analysis, the whole droplet deposit is scanned visually at a magnification of $1 \mu\text{m}$. The floral structures are visible at this magnification as small button structures. The typical number of such structures for different cases is noted and the number density per unit area is determined. In majority of the cases, the floral structures are individual and are counted accordingly. Each experiment set is repeated five times, and the structures are counted using microscopy of the deposits. The typical variation among the number density of floral shapes for each sample has also been noted. The height, morphology and size of the floral structures may vary; however, that does not form the focus of this present study. Such physics has already been reported in previous literature (Popov 2005, Pease III et al. 2010). The focus of this article is kept on the nature of the internal advection and its role vis-à-vis the propensity of formation of the well-defined floral structures from the dispersed nano flakes. The formation of the flowers is due to the interplay of hydrodynamic and cohesive-adhesive forces between the larger nanoflakes and the base fluid during evaporative drying. This forms weakly structured flakes (figure 2(b)), however, the cohesive forces between the flakes is strong enough to withstand damage to the structural integrity of the flowers during the gold film deposition before HRSEM imaging. The physical layering of the large flakes to form the floral structures
Figure 1. HRSEM image of a typical microscale rose structure by individual nano-flakes formed post evaporation of a water-acetone-$\text{Bi}_2\text{O}_3$ (40% water, 60% acetone, and 0.025 wt% nanomaterials) colloid droplet.

Figure 2. (a) HRSEM image of a microscale rose structure from a binary colloid (60% water, 40% acetone, and 0.025 wt% $\text{Bi}_2\text{O}_3$) droplet (here two rose structures seem to overlap one another) (b) zoomed view within the structure showing the physical layering of the individual flakes (c) formation of random layers of nanoflakes for only water or acetone based colloids.
Figure 3. (a) Number density (per 0.25 mm$^2$ area) of the rose structures for different evaporating droplet of colloidal binary fluid compositions (at 0.025 wt% Bi$_2$O$_3$) (b) number density (per 0.25 mm$^2$) of the rose structures for different Bi$_2$O$_3$ concentrations at 60–40 water acetone composition (W60 A40). The error bars represent the maximum and minimum of the observations in each case, with respect to the average value shown in the figure.

can be observed from figure 2. It is also important to note that the binary complex fluids are responsible for the aggregation behavior, as only acetone and water based colloids do not show such floral structures, but only randomly deposited nanoflakes on the substrate (figure 2(c)).

The experiments were designed using proper parametric variation to establish facts from artefacts. First, at constant Bi$_2$O$_3$ concentration, the role of the binary fluid composition was studied. Next, the role of the Bi$_2$O$_3$ concentration was studied at the optimum binary fluid composition. The results of the parametric experimentation are illustrated in figure 3. The dried patterns from both pure water (W100 A0) and acetone (W0 A100) based colloids (irrespective of nanomaterial concentration) do not show any sign of floral pattern formation (figure 3(a)), and the SEM images only show uniformly distributed bed of the nanostructures (figure 2(c)). However, in the case of binary colloids, such floral pattern structures are always present, although their number density is a function of the binary fluid composition. This clearly indicates that the self-assembly phenomena is driven by the internal advection generated within the binary fluid droplet. This further brings to the forefront that the unequal evaporation rate generated concentration and/or thermal gradient driven advection within the binary colloid droplet (Harikrishnan et al. 2018, Jaiswal et al. 2018b, Dhar et al. 2019) is the major governing phenomenon for the formation of the observed structures. Based on the population count of such structures from microscopy (over a $\sim$ 0.25 mm$^2$ scan area), the number density plot has been shown in figure 3(a).

The consistently highest number density of the floral structures is observed for the W60 A40 binary colloid, and majority of the structures possess good symmetry. The immediate neighboring compositions (W40 A60 and W80 A20) also yield such structures, albeit reduced in number density, and asymmetric defects are innate in such structures (figure 2(a)). From the nature of the plot it is evident that presence of acetone in water is a governing criterion that induces the self-assembly of the larger flakes. However, beyond certain acetone content, reversal of trend is consistently prominent, which furthers the proposition of optimal thermosolutal Marangoni advection as a governing mechanism. Figure 3(b) illustrates the influence of the nanoflakes concentration on the number density of the floral structures. Measurements of the viscosity of the colloids (refer supporting information, measured using capillary viscometry (Dhar et al. 2013a, 2013b), Anton Paar) illustrate that the viscosity (for all the binary...
fluids) enhances suddenly beyond the $\sim$0.025 wt% Bi$_2$O$_3$ concentration. Consequently, a proposition can be made that in the dilute regime (<0.025 wt%), the number density of the flakes is not sufficient to aggregate into well defined and numerous floral structures, whereas beyond the optimum concentration, the enhanced viscosity of the colloid opposes the internal advection hydrodynamics, which plausibly leads to reduction in formation of self-assembled microstructures. Consequently, the trend in figure 3(b) can be explained based on the viscosity of the colloids.

With evidence that the internal soluto-thermal advection results in the self-assembly, the kinetics of the advection and the observations in figure 3(a) needs to be correlated. The temporally averaged advection velocity during the initial phases of evaporation (within few seconds of depositing the droplet and after the droplet has stopped its spreading phase) and that during the droplet half-life period (refer figure 4(b) for the non-dimensional droplet life-times) are quantified using microscopic PIV. The velocimetry imaging is done to capture 1000 consecutive images (of a quadrant of the droplet, from the top view), and the spatial velocity fields are determined. The temporal average of such flow fields gives the mean flow field within the droplet during the velocimetry. The spatially averaged velocity of the mean flow field gives the spatio-temporally averaged mean velocity within the droplet. The flow patterns within the droplets may not always be very axisymmetric, and will change as the drying of the droplets progresses. Further, asymmetric flows within the droplet will be induced by wettability gradients of the underlying substrate, which cannot be always eliminated. However, since the fluids used (water and acetone) are highly miscible, it may be assumed that flows arising out of differential solubility within the droplet is not possible. Thereby, it is assumed that the flows are fairly symmetric with respect to the centre of the droplet, and one quadrant of the droplet has been employed for the velocimetry exercise. In order to take into account the role of the drying process on the hydrodynamics, both the initial regime velocity and late regime (at the droplet half-life) velocity have been considered. In fact, the very foundation of the manuscript rests on showing that the drying process induces differences in hydrodynamics, which in turn leads to the different nanoscale aggregates. This shall be subsequently discussed.

The velocities of internal advection are illustrated in figure 4(a). The PIV data is post-processed to obtain the spatio-temporally averaged velocities at the horizontal mid-planes of the droplets. The velocities are obtained at the initial regime (within seconds of placing the
Figure 5. Post-processed velocity contours and vector field at the droplet mid-plane (horizontal) (a1) W60 A40 sample at initial regime (a2) W60 A40 sample at late regime (b1) W0 A100 sample at initial regime (b2) W0 A100 sample at late regime. The contours and velocity fields correspond to the temporal average of 1000 velocimetry frames (experimental frame rate is 30). Each sub-part in this figure represents the top view of a quadrant of the sessile droplet.

droplet) and at the later regime (corresponding to the half-life of the droplet). The initial regime velocities are observed to enhance with addition of acetone to water, and is due to solutal Marangoni convection and solute gradient advection (Jaiswal et al 2018b, Kaushal et al 2020) within the binary fluid droplet, brought forth by the rapid evaporation rate of acetone compared to water. Since acetone vaporizes faster compared to the water phase (figure 4(b)), the solutal advection kinetics is a function of time and decaying species concentration within the droplet. The advection velocity trends at the half-life (figure 4(a)) shows that the velocities decay significantly, except for the case of pure acetone and W20 A80. The velocity at half-life for the optimal case of W60 A40 is interestingly similar to the water case, despite the initial regime being largely dominated by advection. Hence, the cases where the floral structure population is higher, shows a stark decrease in the internal circulation velocity at the half-life regime from the initial regime.

Hence, the initial and late regime advection characteristics both govern the floral pattern formation. A picture of the internal kinetics can now be proposed. The 40–60 nm flakes behave
as perfect Stokesian bodies (Fischer 2002) and follow the advection behavior, and are deposited evenly along the substrate during the dry-out process (Jung et al 2009). The larger flakes however, due to their greater adhesion-cohesion interplay, are deposited in agglomerates, as local structures or clusters during the dry-out phase. While the initial regime advection currents (figure 5(b1)) allow the clustering of the larger flakes, the fast advection and mixing motion in the W0 A100 and W20 A80 even in the late regime (figure 5(b2)) prevents the clusters from stabilizing structurally during the dry-out (Hu and Larson 2006), thereby not allowing floral aggregations to form. On the contrary, the initial advection in the W60 A40 (figure 5(a1)) is potent enough to support the localized clustering and aggregation. The later advection decays substantially (figure 5(a2)), which permits for the aggregates to stabilize structurally under the balance of adhesion-cohesion during the dry-out phase, and form the rose like shapes, to achieve thermodynamically favorable conformations and structures (Zhang et al 2012, Ouyang et al 2012, Ibenskas and Tornau 2012). In the low acetone concentrations, the initial advection itself is weak and cannot induce the favorable clustering and aggregation. The results thus illustrate how the Marangoni advection, which is well documented in literature (Tam et al 2009, Dhar 2019), may lead to such intricate physical assembly of colloidal flakes in binary fluid systems. Further details on the thermal states of internal advection regimes and periods have been discussed in figure S3 (refer supporting information).

4. Conclusions

To infer, the article explores the role played by internal Marangoni advection and the hydrodynamic characteristics within sessile droplets of complex fluids on formation of microscale floral structures. Bi2O3 nanoflakes based water-acetone binary fluids are tested. Evaporation induced clustering and self-assembly of the flakes to form rose shaped structures is noted from HRSEM. It is seen that the water-acetone proportions acutely govern the population density of such structures. Microscopic PIV is done to understand the kinetics of the internal hydrodynamics and Marangoni advection, and it is revealed that the strength, nature and spatial extent of the advection are the governing factor. Likewise, the size of the nanoflakes and viscosity of the colloid also play dominant roles. It is shown that both the initial regime and late regime advection behavior is important for the structure formation. High values of initial regime advection velocity lead to clustering and agglomeration of the larger sized nanoflakes. Further, low values of late regime velocities allow the clusters and aggregates to stabilize and be deposited as floral shapes and structures. This alliance of the hydrodynamic behavior of the two regimes is the necessary and sufficient condition for the aggregation of the larger flakes to form the rose shaped structures, and any deviation from these conditions lead to absence of such structures. The findings are first of its kind and directly correlates the internal microhydrodynamics within a droplet to the observed colloidal self-assembly and structuring.

Data Availability statement

The data that supports the findings of this study are available within the article (and its supplementary material).

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Conflict of interest
The author has no conflicts of interest with any individual or agency.

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