

Evaporation of Ethanol Drops on a Heated Substrate Under Microgravity Conditions

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Abstract We present in this paper results obtained from a parabolic flight campaign regarding ethanol sessile drop evaporation under reduced gravity conditions. Drops are created using a syringe pump by means of injection through a PTFE (polytetrafluoroethylene) substrate. The drops are recorded using a video camera and an infrared camera to observe the thermal motion inside the drop and on the heating substrate. The experimental set-up presented in this paper enables the simultaneous visualization and access to the heat flux density that is transferred to the drop using a heat flux meter placed between the heating block and the PTFE substrate. We evidence original thermal spreading phenomena during the ethanol drop creation on a heated PTFE substrate. The drop exhibits specific behaviour which is discussed here. This work is performed in the frame of a French-Chinese collaboration (project IMPACT) for future experiments in a Chinese scientific satellite.

Keywords Infrared visualization · Sessile drop · Liquid spreading · Capillary forces · Drop evaporation

Introduction

The evaporation phenomena can generate temperature variations in the vicinity of the solid surface and along the drop interface. The Marangoni effect can produce instabilities at the interface. The variations of the contact angle and dimensions of the meniscus (profile and perimeter of contact) as well as the heat gradient within the interface are studied in the literature according to temperature differences between the solid surface and the gas phase. Drop evaporation phenomena have been studied with experiments and numerical simulations for several configurations. An important area to take into account in the analysis is the contact line. This line represents the location where the three interfaces exist. In this zone, an intense evaporation takes place. Numerous research studies have focused on the droplet evaporation in order to highlight the main physical mechanisms, which govern the evaporation kinetic (see Table 1). Since 2002, the research on this topic became very productive, and is still active nowadays. Indeed, the droplet evaporation appears in a number of fabrication or deposition processes and in various heat transfer applications. According to the literature, the main physical mechanisms governing the drop evaporation process are:

- the conduction heat transfer into the wall,
- the convective heat transfer induced by the surface tension gradients and the natural convection due to the temperature gradients in the liquid drop,
- the liquid/wall molecular interactions, coupled with the substrate surface roughness, which tend to

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Table 1 Publications related to sessile drop evaporation [numerical simulation (num.), experiments (exp.) or theoretical investigations (th.)]

First author	Year	Approach	Fluid	Substrate
Hegseth (Hegseth et al. 1996)	1996	Exp.	Ethanol, polystyrene particles	–
Chandra (Chandra et al. 1996)	1996	Exp. and th.	Water with surfactant	Stainless steel
Bernardin (Bernardin et al. 1997)	1997	Exp.	Water	Aluminum
Zhang (Zhang and Chao 2001)	2001	Exp.	Silicon oil, R113, ethanol	Anodized glass
Erbil (Erbil et al. 2002)	2002	Exp.	N-butanol, toluene, n-nonane	PTFE
Crafton (Crafton and Black 2004)	2004	Exp.	Water, n-heptane	Copper, aluminum
Savino (Savino and Fico 2004)	2004	Exp. and num.	Silicon oil, hydrocarbones, water	Several metals
Grandas (Grandas et al. 2005)	2005	Exp.	Water	PTFE, aluminum
David (David et al. 2006)	2006	Exp.	Water, methanol, acetone	PTFE, Macor, Ti, Al
Sodtke (Sodtke et al. 2007)	2007	Exp. and th.	Water with saturated vapour	Stainless steel
Tarozzi (Tarozzi et al. 2007)	2007	Exp.	Water	Black paint on IR materials
Sefiane (Sefiane et al. 2008)	2008	Exp.	Ethanol, methanol, FC-72	PTFE, ceramic, titanium, copper

- modify the droplet wettability, and also, the wetting surface area for identical drop volume,
- the vapour mass diffusion around the drop.

Despite active research, the dominant mechanisms acting on the drop evaporation kinetic are not well understood due to the strong couplings between the various phenomena. In the drop evaporation studies two approaches may be distinguished: the drop evaporation with and without heating the wall (Bernardin et al. 1997; Erbil et al. 2002; Grandas et al. 2005). For drop evaporation in a non-saturated gas and without any wall super-heating, the vapour mass diffusion in the gas surrounding the droplet may become the limiting phenomena on the evaporation kinetic. According to the experiments of Shahidzadeh-Bonn et al. (2006) the evaporation rate is affected by the fact that water vapour is lighter than air and that the vapour of other liquids is more dense. In a purely diffusing case, the vapour of water does not accumulate at the drop interface which would enable a higher evaporation rate. In the case of drops posed on a heated wall, the interactions between the liquid and the wall play a fundamental role in the evaporation kinetics. The large temperature difference between the wall and the liquid is the source of thermo-convective instabilities, induced by both Marangoni and buoyancy forces. These instabilities largely depend on the thermo-physical properties of the liquid and of the substrate, but also on the drop shape, which is correlated to the liquid/substrate interactions.

Various experimental studies attempt to explain the role of the particular mechanisms on the drop evaporation. In most test set-ups, a drop of controlled volume is deposited on the substrate from a syringe. The global evolutions of the drop shape (drop base radius,

height and contact angle) are usually deduced from the drop visualization with a CCD camera. Thus, the drop behaviours for various couples of liquid/substrate have been analysed (Bernardin et al. 1997; David et al. 2006). During the drop evaporation, two main drop shape evolutions have been observed: a constant drop base diameter and a constant drop contact angle. As an example, Crafton and Black (2004) observed that, for a water drop, the drop base radius remains constant during the evaporation, whilst for a heptane drop, the contact angle remains constant. Other global observations on the drop evaporation rate (based on drop volume or weight measurement) indicate a linear increase of the evaporation rate with an increase in the drop base diameter. Analysis of such results leads to an assumption that evaporation mostly occurs at the triple line.

Although global observations are useful in giving an overview of drop behaviours for specific conditions, such an experimental approach is not sufficient for the prediction of the evaporation kinetics depending on the coupling of liquid/substrate for various substrate super-heating. There are a few experimental studies that attempt to give further understanding of the drop evaporation using specific techniques. Hegseth et al. (1996) and Kang et al. (2003) have used PIV to visualize the fluid flow inside the drop, by charging the ethanol drop with polystyrene particles. Whilst Zhang and Chao (2001) have used a laser shadowgraphy method to visualize the fluid flow. An investigation of Marangoni and buoyancy convection in a hanging drop of silicone oil and hydrocarbons using infra-red thermography have been tested by Savino and Fico (2004). The wall temperature distribution below the drop has also been measured using thermo-chromic liquid crystals (Sodtke

et al. 2007) and with infra-red thermography through an IR-transparent substrate (Tarozzi et al. 2007).

The experimental method that is often used to make non intrusive thermal measurements during drop evaporation is infrared thermography (Savino and Fico 2004; Tarozzi et al. 2007; Tartarini et al. 2008; Tuckermann et al. 2005; Wulsten and Lee 2008). The non-contact advantage of this technique enables the observation of the thermal phenomenon without any disturbance. However, the difficulty is then in correctly interpreting the infrared flux measured by the camera with respect to the radiative properties of the observed fluid (see Fig. 1). Indeed, the liquid sample may be semi-transparent in some spectral measurement ranges, making it difficult to convert the measured radiance into surface temperature. Some authors (Tarozzi et al. 2007; Tartarini et al. 2008) observe the rear face of the plate on which the drop creates a layer. The ‘drop side’ of this transparent BaF₂ plate is coated with an opaque

emissive paint, making it possible to measure its surface temperature which is also the temperature of the drop base. In other publications, authors observe directly the fluid (Savino and Fico 2004; Tuckermann et al. 2005; Wulsten and Lee 2008). Assuming the opacity of the various observed fluids (water, ethanol, n-pentane) in the long wave spectral range of the camera (around 9–10 μm) they only need to know the fluid emissivity to deduce the surface temperature of the liquid. To our knowledge, no article has been published on work dealing with the IR observation of heated liquid being semi-transparent in the spectral range of the camera. This kind of study is already done for polymers (Cuccurullo and Berardi 2002), where the authors try to infer the temperature of an isothermal semi-transparent slab from the radiometer signal and may also try to treat the non-isothermal case. Finally, some authors such as Liu (2000) propose to solve the inverse radiation problem of the simultaneous identification of temperature profile and absorption coefficient in a semi-transparent medium, in one dimension.

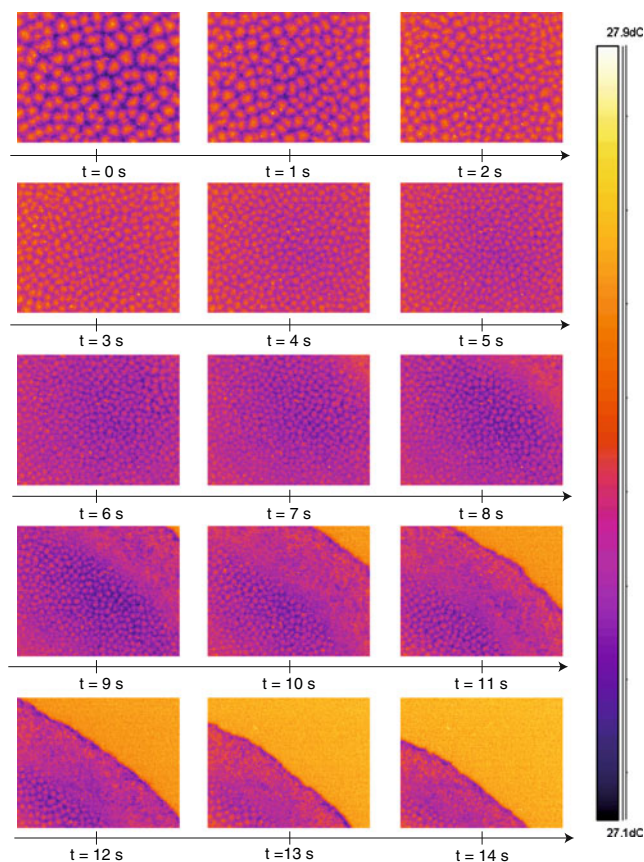


Fig. 1 Example of the performance of an infrared camera: observation of convection cells in a 1 mm-thick starting FC-72 layer under natural evaporation on PTFE, [$T_{\text{room}} \sim 28^\circ\text{C}$, $T_{\text{substrate}} \sim 28^\circ\text{C}$, $\Delta T_{\text{image}} = 0.8^\circ\text{C}$]

Experimental Set-up

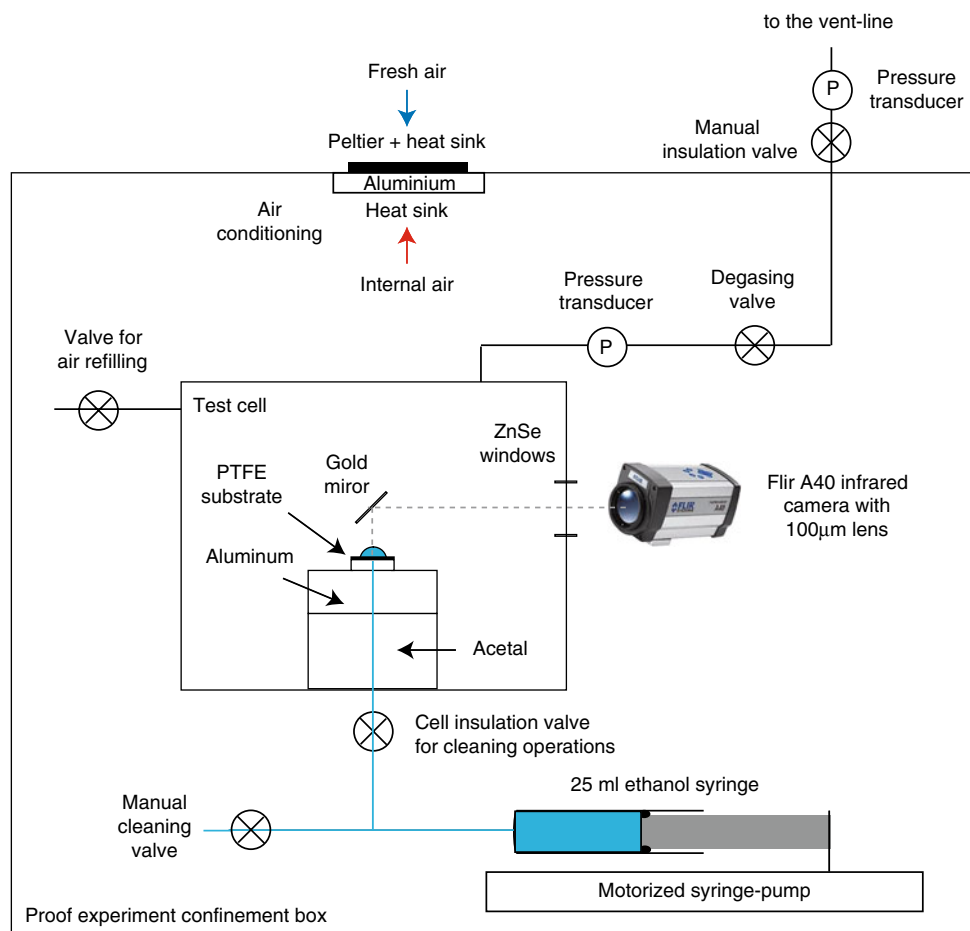
The experimental set-up is designed to enable the creation of an ethanol sessile drop to and to observe its subsequent evaporation under reduced gravity conditions. A test cell, located inside a confinement box, is used to create a drop in a thermally controlled environment. The experiments were performed during a parabolic flight campaign at Novespace, France during the 79th parabolic flight campaign organized by CNES.

Rack and the Test Cell

The rack is 1.8 m \times 0.80 m \times 0.75 m and weighs 125 kg. It is made out of Bosch aluminium bars. A confinement box enables the storage of the fluid test cell, in which the drops will evaporate. The rack is equipped with two computers, power supplies and a heating and cooling system to control the confinement box temperature during the flight. Regarding the test cell and the heating substrate: a 700 μm diameter hole is performed through the heating surface of 10 mm diameter to create the drop by injection at a low mass flow rate, as shown in Fig. 2.

The injection mass flow rate is a parameter which has been determined experimentally in microgravity during a previous campaign (Brutin et al. 2009), to avoid jet formation at a high mass flow rate. If the mass flow

Fig. 2 Confined volume and its equipment used for ethanol sessile drop injection and evaporation on PTFE: a test cell which contains a heating substrate and a gold mirror, a one way syringe-pump and an infra-red camera



rate is too low, the drop creation time will be important in comparison to the microgravity time (20 s). The substrate temperature can be modified using heating cartridges. The surface temperature can be increased by +10°C, +15°C, +20°C, +30°C and +35°C from the cell air temperature, which for all experiments is between 15–20°C.

The drops are created under microgravity conditions to measure and observe the static and dynamic contact angles. Experiments were performed to confirm the sessile drop creation feasibility in microgravity even for very low surface tension fluids (FC-72 and HFE-7100) in a previous campaign (Brutin et al. 2009). Here, only pure ethanol at 99.9% is used. Under terrestrial gravity, the drop during the creation is maintained on the surface by gravity. Under microgravity, the drop is maintained on the surface mainly due to surface tension forces and is slightly influenced by the gravity fluctuations. We previously confirmed that under microgravity conditions for low surface tension fluids, the drop does not detach from the surface even if the vibration level is high (Brutin et al. 2009). The

experiments are performed at the plane cabin pressure which is regulated at 835 ± 2 mbar.

Visualization

The experimental rack is equipped with two digital cameras:

- a visible camera Pike AVT (800 by 600 pixels) is used to record the drop creation using a side view; a microscope lens VZM100i is used to reach a spatial resolution of 15 μm . The acquisition frequency is 7.5 images per second to enable a continuous recording during the 90 s (20 s before the parabola, 60 s of parabola and 10 s after the parabola).
- an infrared camera FLIR A40 (320 by 256 pixels) is used to record the thermal behaviour of the drop and the substrate using a top view (a commercial gold mirror of 20 mm in diameter is used); the spatial resolution is 100 μm using a dedicated lens; the acquisition frequency is 25 images per second (Fig. 3).

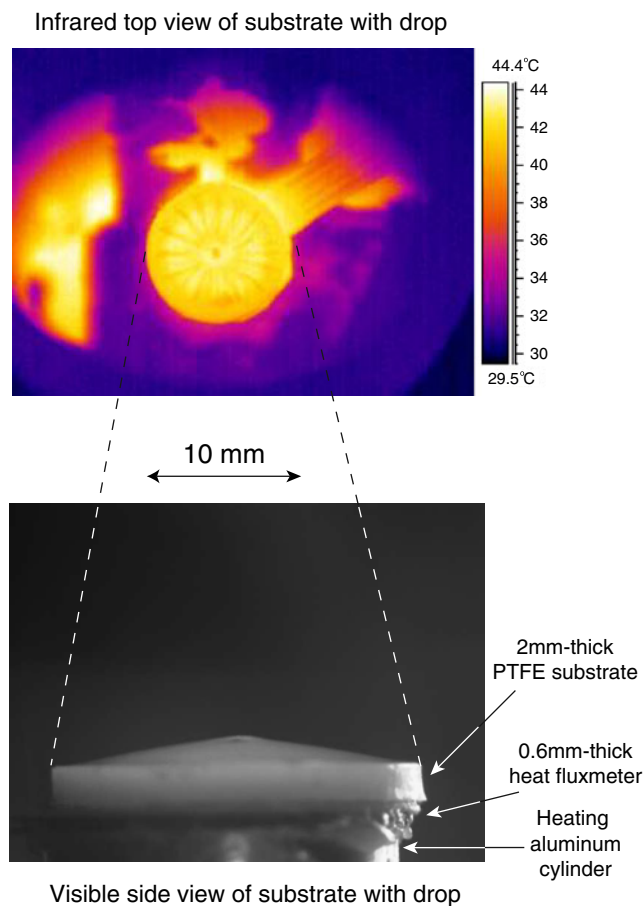


Fig. 3 Top image is a top view using the infrared camera with a field of view of 32 mm by 25.6 mm. Bottom image is a side view using the visible camera on the same drop with a field of view of 13 mm by 9.75 mm

A heat flux-meter (CAPTEC) is used and stuck between the heating block and the PTFE substrate. A 54 μm adhesive tape is used to adhere all these elements together. The flux-meter has a diameter of 10 mm and a thickness of 0.6 mm. It is made out of copper which reduces the heat resistance from the aluminium block to the drop. The PTFE substrate is 2.0 mm thick.

Data Acquisition

The test cell is regulated for the temperature and pressure, which are recorded using K-type thermocouples and a differential pressure sensor respectively. The

heating substrate temperature is regulated using a PT-100 sensor with a PID regulator at $\pm 0.1^\circ\text{C}$. The aluminium block is heated by two heating cartridges of 15 W each. The entire experimental set-up can be found in full detail in Brutin et al. (2009).

Working Fluid and Wettability Issues

The ethanol properties used in this paper are presented in Table 2. Using this fluid physical properties, it is possible to determine the capillary length for the three gravity levels encountered. The capillary length is used to determine the critical diameter between the gravity deformable drop and the non-deformable drop. The critical diameter is about two times the capillary length given in Table 2. When the drop is very small (droplet) the surface tension is mainly dominating the drop interface curvature. The gravity in this situation has almost no effect on the drop interface. However, when the drop diameter is bigger than the capillary length, the drop interface is strongly influenced by gravity effects. Ethanol is used for its relative high surface tension compared to low enthalpy fluids such as HFEs or FCs. Ethanol also has a low saturation temperature and phase change enthalpy which are useful for evaporation. Ethanol is also semi-transparent in the infra-red wavelength of the infra-red camera.

To easily access the contact angle by image processing, we need to obtain a picture with a high diameter/height ratio. When the diameter is much larger than the height, the contact angle is difficult to determine due to the difficult determination of the drop interface. For ethanol drop wettability, the liquid/surface wettability is low, which corresponds to a wetting situation observed previously for FC-72 and HFE-7100, the liquid spreads on the surface, so that the contact angles will be below 10 to 20°.

Infrared Properties of Ethanol

Ethanol transmittivity in the 7.5 to 13 μm range is low for a sample fluid with a thickness of 0.5 mm (Fig. 4). Consequently, the global emissivity for a sample of ethanol with a thickness of 0.5 mm is 0.89 (Fig. 5). For fluid thicknesses below 3 mm, emissivity varies exponentially with fluid thickness until a null

Table 2 Liquid physical properties and capillary lengths at 25°C and 0.835 atm

	ρ_L kg.m^{-3}	ρ_V kg.m^{-3}	C_p $\text{J.kg}^{-1}.\text{K}^{-1}$	L_v kJ.kg^{-1}	λ $\text{W.m}^2.\text{K}^{-1}$	μ mPa.s	σ mN.m^{-1}	L_c (μg) mm	L_c (1g) mm	L_c (1.8 g) mm
Water	997	0.60	4,180	2,449	0.606	0.891	72.7	12.2	2.7	2.0
Ethanol	789	1.55	2,845	841	0.140	1.095	22.0	7.5	1.7	1.3

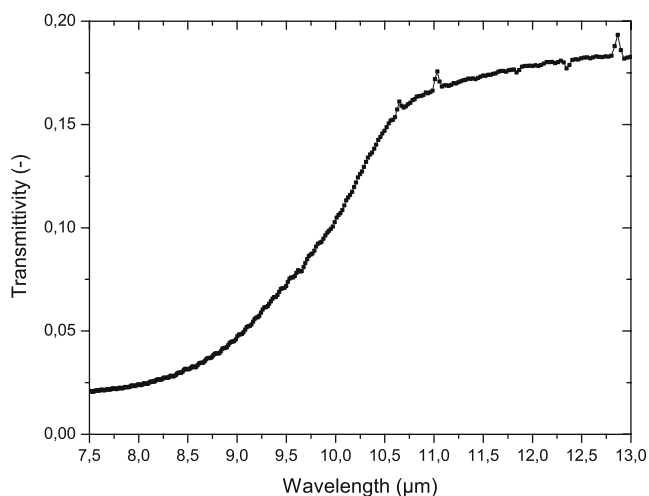


Fig. 4 Spectral transmittivity of ethanol as a function of the wavelength for an optical thickness of 0.5 mm

emissivity for a null fluid thickness. Thus, ethanol can be used for infra-red measurement by transmission up to 3 mm and have a transmission of 2% (Fig. 5).

Experimental Protocol

To perform the experiments within a few minutes between two parabolas, we used the following procedure. During normal gravity, the test cell is cleaned using the vent-line as a vacuum cleaner. The remaining liquid is evaporated by the sudden decrease of pressure from 835 mbar to 350 mbar (vent-line pressure). Then fresh air is taken from the confinement box to refill the test cell of air at the cabin pressure and temperature. The

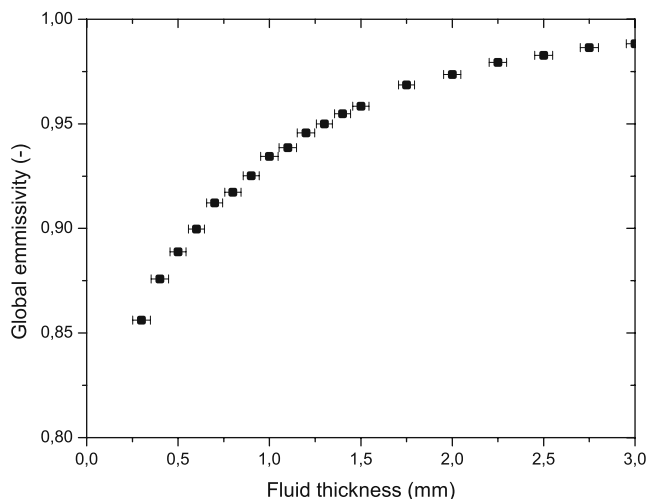


Fig. 5 Global emissivity of ethanol as a function of total optical thickness in the range: 7.5 to 13 μm

flush is performed several times to ensure identical starting conditions. The syringe pump is set to a low mass flow rate value. Ethanol is injected at this low mass flow rate in the test cell to ensure that a liquid level very close to the substrate level is obtained. A small quantity of ethanol vapour created is evacuated by a one or two last flushes. The mass flow rate is set between 10 to 40 μL/s for the drop creation. There are no more actions performed 30 s before the parabola starts to ensure stable thermal conditions. During the first second of the microgravity, the syringe pump is switched on. The drop is created, and evaporates during the microgravity. All the data and video recording are started 20 s before the parabola. All acquisition devices are stopped 10 s after the end of the parabola. Then, the test cell is cleaned using the vent-line to prepare for the next experiment.

Data Analysis

In the two following subsections, we present the results obtained regarding the thermal behaviour observed during ethanol drop injection in microgravity. Then we detail the typical results obtained using a heat fluxmeter during drop evaporation in microgravity.

Ethanol Spreading at Injection

When ethanol is injected through the substrate, the liquid spreads due to the low surface tension with PTFE. A thin layer of ethanol quickly propagates on the heating substrate to reach the substrate periphery. A bump of the drop interface is clearly visible at the apex. This bump is due to the injection mass flow rate which deforms the drop interface in microgravity near the hole exit. On Fig. 6, we put a sequence of side images taken when a drop is injected during microgravity conditions at 20 μL/s.

The side view does not enable us to clearly see the drop spreading behaviour. This is possible using the top view provided by the infra-red camera. On Fig. 7, we present the same experiment but recorded with the infra-red camera. The time gap between two images is 40 ms. Ethanol reaches the substrate periphery 560 ms after the drop injection starts. During the liquid injection, axis-symmetric thermal patterns can be observed. These patterns are clearly due to the ethanol injection and spreading on the substrate in microgravity; then they vanish during the next steps of the injection; cold fluid coming from below the substrate is injected and propagates into the drop. The injection hole drilled

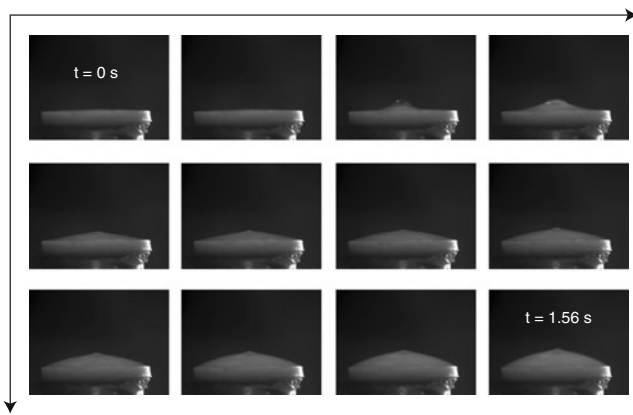


Fig. 6 Visualization of ethanol injection under reduced gravity conditions (130 ms between each frame, substrate temperature is 45°C)

into the heating substrate is 0.7 mm diameter and 20 mm long which represents a volume of warm fluid at the substrate temperature of 8 μ L. When the ethanol injection starts, first warm fluid at the substrate temperature is injected, then colder fluid is used to create the drop which is clearly evidenced on Fig. 7. This infrared sequence evidences that the duration to reach a thermally homogeneous drop is about 3 s. The evaporation analysis begins only after this transition period.

Heat Flux Transferred to the Drop

The description of the typical evaporation phenomenon studied is based on Fig. 8. The CAPTEC flux-meter is used to measure the heat flux density provided to the drop during microgravity. The objective is quantify the influence of the gravity level on the evaporation phenomenon. The gravity level and the flux-meter data are acquired at 133 Hz; however due to the electrical noise at low pass filter is applied at 1 Hz. The four phases described in accordance with Fig. 8 are the following:

- Phase 0 [Terrestrial gravity]: the substrate is dry; the heat flux decreases from the previous experiment to the natural convection and radiative heat flux transferred from the PTFE substrate to the surrounding air. The temperature rises to reach a stabilized value.
- Phase A [Hypergravity]: the substrate is still dry; the parabola begins with an increase of the gravity level, the heat flux and the substrate temperature are almost stabilized.
- Phase B [Microgravity]: the microgravity begins then ethanol is injected through the substrate. The heat flux transferred to the drop strongly increases

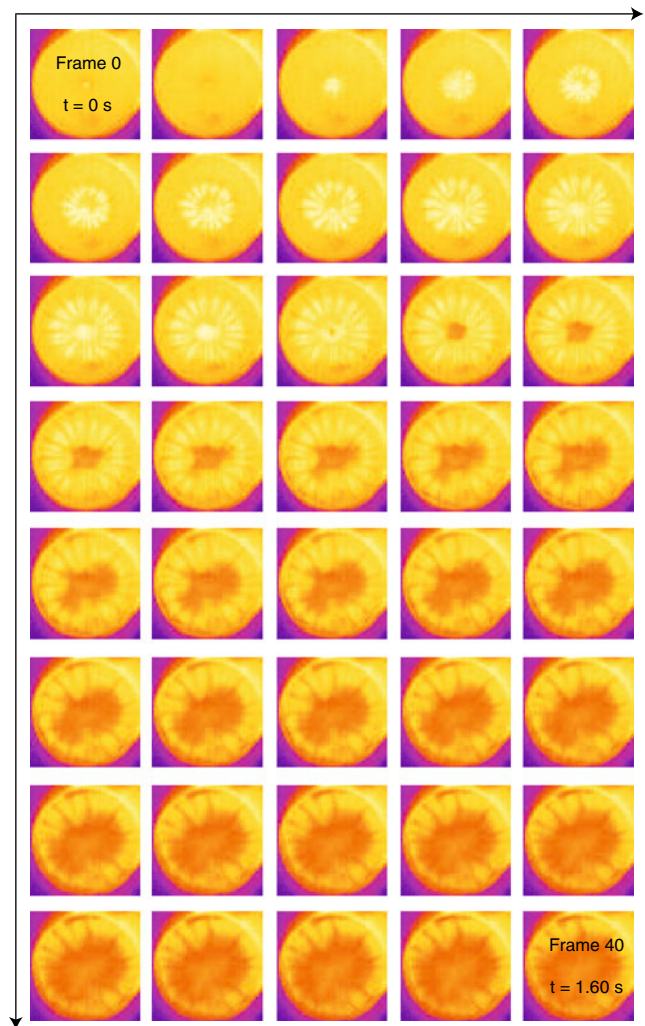


Fig. 7 Infrared visualization of ethanol injection under reduced gravity conditions (40 ms between each frame, substrate temperature is 45°C)

- Phase C [Hypergravity]: the microgravity end while the drop is not yet completely evaporated. The appearance of hypergravity induces a sharp increase of heat flux transfer to the drop. This strong increase is coherent with the existence of convection cells. Flow motion inside the drop is mainly driven by the buoyancy forces which increase due to the

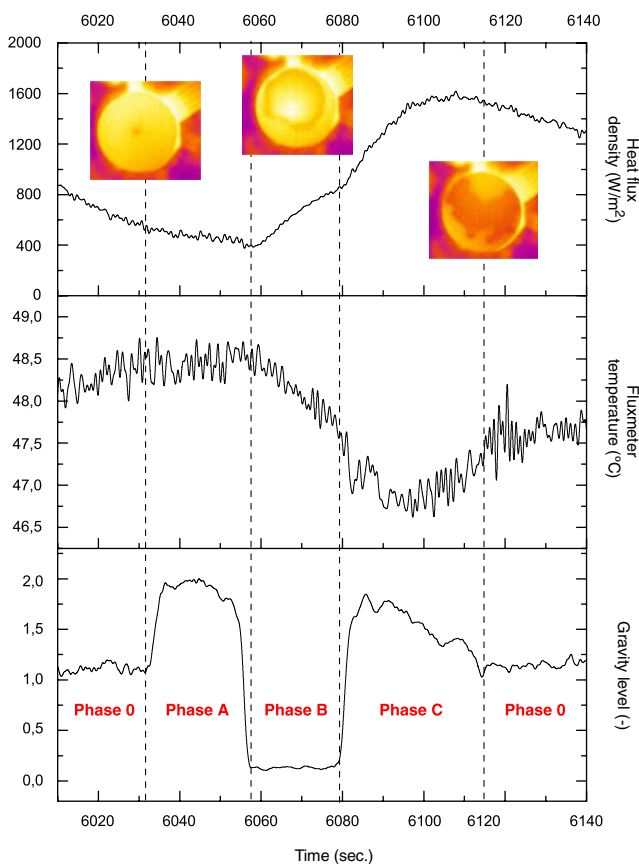


Fig. 8 Evolution of the heat flux transferred to the drop provided by the flux-meter (*top graph*), substrate temperature given by the flux-meter (*middle graph*) and gravity level provided by the experiment accelerometer (*bottom graph*) for an ethanol drop creation during microgravity on a substrate at 48.5°C (PID regulation at 50°C)

hypergravity. As soon as the drop is completely evaporated, the heat flux decreases and the substrate temperature increases.

- Phase 0 [Terrestrial gravity]: the gravity level is back at the terrestrial one, the substrate is dry, the heat flux and temperature return to the starting values. The test cell is cleaned.

The PTFE substrate inertia appears to influence the heat flux and substrate temperature. The layer of PTFE used is 2 mm which induces a thermal resistance between the drop and the heat flux-meter. For the next campaign, a PTFE substrate of a few hundred microns or a thin coating will be used, to enable a good wettability with less thermal resistance.

Using this heat flux-meter technique, we evidenced the feasibility of heat transfer measurements with sessile drops in microgravity. The next step is to have a longer experiment time and better microgravity condi-

tions, hence enabling better infra-red and visible visualisation, coupled with the heat flux measurements.

Conclusions and On-going-work

We presented results obtained during a parabolic flight campaign observing ethanol sessile drop evaporation under microgravity conditions. Drops were created using a syringe pump by injection through a substrate. The drops were recorded using a video camera and an infra-red camera. The experimental set-up presented in the paper enables the simultaneous visualization in the infrared and the visible wavelengths and also allows access to the heat-flux density transferred to the drop using a heat flux meter. We evidenced the existence of an increasing heat flux encountered during microgravity. The absence of gravity does not stop the evaporation which still occurs. The short duration of the microgravity does not enable us to obtain a stationary case. Also, we evidenced original thermal spreading phenomenon during the drop creation on a heated PTFE substrate. The drop evaporation is also characterized by the heat transfer observed during the microgravity phase with a sharp increase of the heat flux used by the drop for evaporation. However the thermal resistance of the PTFE substrate does not enable us at this time to access the steady state measurements expected.

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