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<td>TÍTULO PROYECTO</td>
<td>STATICS AND DYNAMICS OF GRANULAR INTERFACES</td>
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<td>INVESTIGADOR(A) RESPONSABLE</td>
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### OBJETIVOS

Cumplimiento de los Objetivos planteados en la etapa final, o pendientes de cumplir. Recuerde que en esta sección debe referirse a objetivos desarrollados, NO listar actividades desarrolladas.

<table>
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<th>Nº</th>
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<th>CUMPLIMIENTO</th>
<th>FUNDAMENTO</th>
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<td>1</td>
<td>Developing methods of caracterization of a granular interface</td>
<td>TOTAL</td>
<td>Langmuir blodgett-through was first tested during the first year and shown inadapted due to side effects and ambiguities of the method of measurement for such an interface. At the end of the second year the evaporating droplet method was developed, allowing both an homogeneous, isotropic compression, and allowing an hypothesese-free measurement of the tensions in the interfaces.</td>
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<td>2</td>
<td>Caracterization of the wrinkling transition of a granular interface</td>
<td>TOTAL</td>
<td>simultaneous analysis of the shape of the drop and imaging the grain network showed that the wrinkling correspond to the random close packing (frictionless J-point) of the granular interface, defined by a specific compacity and number of contacts.</td>
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<td>3</td>
<td>Caracterization of the mechanical properties befor the wrinking.</td>
<td>TOTAL</td>
<td>Simultaneous mechanical measurement and network imaging in the drop evaporation experiment evidenced three distint mecanical regimes of constant compressibility, we named L,S1 and S2 regimes respectively, (by increaing compressibility)</td>
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<td>4</td>
<td>Measurement of the heigth of a granular interface under compression using ftp technique</td>
<td>PARCIAL</td>
<td>A Fourier transform profilometry system was built in the first year of the project, but not applied directly to the project due to the change of experimental system from a langmuir blodgett through to an evaporating liquid marble. Nevertheless, this system is now in use for other experiment in the laboratory.</td>
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Two experiments were done:

First, a vibrating liquid marble experiment showed that the effective pressure between grains in a granular interface decreases when the interface is shaken.

Secondly, a shearing drop experiment was done in collaboration with Navier Laboratory (Marnes la Vallée, France) to study the response of a granular system to shearing.

The fact that previous measurements of granular interfaces were irrelevant was published by Bormashenko & al in early 2013 (Colloids and Surfaces A: Physicochem. Eng. Aspects 425 (2013) 15–23). It both reinforced the relevance of this project and made us adapt our study in a late stage of the project to answer in depth to the questions raised by Bormashenko, delaying the writing of the first paper from early 2013 to September 2013.
RESULTS OBTAINED:

For each specific goal, describe or summarize the results obtained. Relate each one to work already published and/or manuscripts submitted. In the Annex section include additional information deemed pertinent and relevant to the evaluation process.

The maximum length for this section is 5 pages. (Arial or Verdana, font size 10).

Context.

This project aims at the study of the properties of the granular interfaces, formed of a monolayer of micro or nano particles at the water surface. Such interfaces possess remarkable properties that have been shown recently but their fundamental mechanisms remained elusive.

The first year of my project was devoted to the statics of these interfaces and more precisely the following questions:
- what is the correct method to investigate such an interface?
- Are the jamming and the buckling related or linked to two distinct mechanisms?
- How does the grain network local structure is modified when under constraint?
- What is the mechanism of the elasticity of these interfaces?

In the second year, we studied systematically the statics using the experimental tools developed in the first year, and started the analysis of dynamics. Both shear response and oscillatory response were investigated.

I. Statics

Methods.

Two setups were built:

1/ A Langmuir trough equipped with a planar pressure sensor and a Profilometry measurement system (FTP profilometry).
Due to the frictional effects of the sides of the trough we found that it is not the ideal system for approaching the basics properties of granular interfaces.

2/ An evaporating liquid marble (drop coated with grains) which has the advantage of the absence of side friction and still allow mechanical and structural measurements by image analysis of the drop.

Local stresses of the interfaces are obtained by the profil measurement using a side view. Structural measurements of the grain network are performed simultaneously by taking images from top with a microscope(Fig.2).

An example of millimetric liquid marbles is shown on fig.1. were from right to left, are presented three drop states : capillary (L), jammed (S1&S2) and buckled (W)

Fig.1 example of liquid marbles

Fig.2. Grain network image (the grains are of 0.025 mm diameter)
Results
We evidenced three distinct regimes of mechanical response of a liquid marble. These are shown in Fig 3. The Surface modulus K increases by jumps, defining the L regime (softest, at low surface fraction) as well as S1 and S2 regimes, of increasing rigidity.

![Image of typical side view of an evaporating liquid marble](image1)

*Fig.3 a) Typical side view of an evaporating liquid marble from a liquid-like shape (left) to a wrinkled shape. b) Mean pressure π in the granular shell as a function of Φ. In logarithmic scale for Φ, the slope is directly the surface modulus K. We named the three different regions of constant slopes L (liquid shell), S1 (solid shell 1) and S2 (solid shell 2). Top views of the grain network for the respective states. Right insert: The π curve rectified by the slope in the S1 region shows a high definition in the location of transitions.*

We characterized each of the each of the transition between regions
The L-S1 transition (Fig 4. a1&a2) correspond to the apparition of a global shear in the network
The S1-S2 transition (Fig 4. b) is the maximum of entropy of the network.
The S2-W transition (wrinkling) is the geometric jamming of the network

![Image of side view of typical droplets](image2)

*Fig 4. a1) Side view of typical droplets with a red line highlighting its profile used to compute stresses and isoperimetric quotient. a2) Deviatoric stress δπ in the shell  b) Distribution of coordination numbers of grains as a function of Φ. The lines indicate the best fits. Insert Left: evolution of the variance of the contact numbers. Insert Right: difference between the surface fraction of the S1-S2 transition and the surface fraction of the maximum variance. c1) Isoperimetric quotient Q as a function of time of an evaporating liquid marble
The W-point indicates the onset of wrinkling. c2) Coordination number (average of the Z distribution for a trial) and Φ at the W-transition.*
Finally, the mechanical properties are shown to be determined by the compacity of grains when the drop was created.

For districts experiments, we observed a large variability of the surface fractions and pressures at which occurs each transition. Careful control of the drop preparation method allowed us to understand the hidden parameter organizing this variability: all our experiments are well organized as a function of the initial surface fraction as can be seen in figure 5.

Fig 5. Granular shell state as a function of $\Phi_0$.
   a) In the surface fraction plane. Points indicate L-S1 transition and S1-S2 transition respectively. Vertical dotted line provides the boundary of stable droplets, (wet the solid surface). Inclined dotted line $\Phi=\Phi_0$.
   b) The average pressure $\pi$ at transitions. Dotted lines indicate best linear regressions.

Conclusion
We showed a direct intrication of the mechanical properties of the granular interfaces with the grains network: Three mechanical transition were evidenced (Jamming, Maximal entropy, and wrinkling) that all correspond to changes of compressibility coupled to structural changes of the grains network.

This work is considered as finished, and an article is close to be submitted, whose version to date is joined in annex.
2. Dynamics.

Two experiments were devised to explore the dynamics properties of granular interfaces in response to a deformation. While still under analysis, the results are sufficiently promising to allow writing of two papers in the next months. Two kind of deformation were explored:

a. Shear
For exploring shear response, an experiment was designed in collaboration with E.Lorenceau of Navier Institute (université de Marnes la Vallée, France). And this collaboration lead to a 6 month internship of a PhD student of the non linear physics laboratory from USACH (A.Rescaglio), financed thanks to “Beca de Pasantía Doctoral en el extranjero Becas Chile, Convocatoria 2012, n°75130074”.

The experiment consists of an horizontal liquid bridge between two horizontal capillars, on which micron sized hydrophobic grains were disposed. The bridge is linked to a pressure sensor, and a camera allows the measurement of the mean curvature. The ratio between both values is an effective surface tension measurement. Shear can be imposed by rotating in opposite direction both capillars. Early results show a large difference of behavior between the static case and the shear case as shown in figure 6.

![curvature vs pressure](image)

**Fig. 6 curvature $\kappa^{-1}$ of granular-liquid bridge as a function of the pressure $\pi$ in the bridge, for distinct shear velocity.**

b. Oscillation
The resonance of a liquid marble, vibrated vertically by a piezoelectric shaker was also investigated. It allows the study of the dynamic effective modulus of the capillary interface. Oscillation amplitude was
The main result is that the resonance frequency decreases when the agitation is more intense (as shown in figure 7) which indicates lower modulus and lower pressure between grains.

![Resonance curves](image)

**Fig. 7 Resonance curves of a liquid marble of fixed number of grains for distinct vibration amplitudes $A_{App}$**
OTHER ACHIEVEMENTS OF THE PROJECT:
- Research visit(s) to other institution(s).
- Outreach activities related to the project’s main topic.
- Any other contribution, not addressed elsewhere, that you consider important.

The maximum length for this section is 1 page. (Arial or Verdana, font size 10).

During this project, I visited specialists of granular interfaces during my travel to Europe:
D. Vella (OCCAM, oxford, UK)
M. Abkarian (université de Montpellier, France)
E. Lorenceau (université de Marnes La Vallée, France)
In the two latter cases I gave a laboratory seminar presenting the results of this project.

During the course of this project, a new collaboration between the non linear physics laboratory (F. Melo) and the Navier Laboratory (université de Marnes la vallée, france) (E. Lorenceau) build upon ideas which were developed during the project.
It lead to a 6 month internship of a PhD student of the non linear physics laboratory (A. Rescaglio), financed thanks to Beca de Pasantía Doctoral en el extranjero Becas Chile, Convocatoria 2012, nº75130074.

I also build the FTP (Fourier Transform Profilometry) system of surface height measurement in the USACH, that while not used directly in this project led was used in other thematics (dry granular matter and elasticity of thin sheets).
This method was also successfully adapted as a laboratory experiment for teaching at Licence level.
El Trabajo del Dr. Lagubeau se desarrolló de acuerdo con lo programado en la propuesta inicial. Se abordaron íntegramente los estudios propuestos y nos encontramos en etapa de redacción y publicación de resultados. Este último aspecto no ha tenido una celeridad óptima debido a las dificultades usuales de proyectos experimentales. Además, publicaciones recientes nos obligaron a profundizar nuestras investigaciones en algunos aspectos no considerados originalmente. A pesar de esto, contamos con todos los resultados y los análisis que nos permitirán contar con al menos dos artículos antes de fines de 2013. Se dispone de un preprint que será sometido en los más breves plazos.

En el detalle, el Dr. Lagubeau logró profundos avances en la comprensión de los estados “jammed” en “liquid marbles”. Abordó tres estudios complementarios entre sí:

1.-Estudio de gotas recubiertas con partículas hidrófobicas “liquid marbles”.
   En una configuración simple pero elegante, el Dr. Lagubeau logró identificar por primera vez estados bloqueados de la superficie granular y caracterizar su respuesta mecánica. La novedad y calidad de este estudio, nos inducen a pensar que estos resultados representan un avance significativo en el área. El grueso de este trabajo se encuentra descrito en el preprint que se anexa.

2.-Dinámica de superficies granulares:
   Para examinar la respuesta al cizalle de las interfaces granulares se desarrollaron novedosos métodos experimentales en colaboración con E. Lorenceau del Instituto Navier en Francia, de esta colaboración también participo nuestra alumna de Doctorado la Srta. Rescaglio quien dedicó seis meses (en Francia) al desarrollo del aparataje experimental y el análisis de datos. Básicamente, el estudio consistió en torcer un manto capilar (recubierto con granos hidrofóbicos) establecido en la brecha entre dos cilindros alineados. El seguimiento de la respuesta mecánica de la superficie a distintas concentraciones de microparticulas, nos ha permitido estudiar los distintos estados de nueva fase bloqueadas y el rol de la “dilatancia” en el sistema.

3.-Se realizaron además estudios de resonancias de “liquid marbles” y se identificó un modulo elástico dinámico de la interface capilar. El resultado mas importante es que la frecuencia de resonancia disminuye cuando la amplitud de la excitación aumenta lo que es bastante sorprendente e indicaría que el modulo elástico disminuye con la excitación. La explicación aportada abre nuevas puertas para la comprensión de estas interfaces puesto que revela que interacciones capilares dipolares entre granos son progresivamente eliminadas por la agitación, disminuyendo la presión de la red de contactos. Un artículo se encuentra en redacción.

En resumen, considero que lo logrado por el Dr. Lagubeau en su período postdoctoral es de alto nivel científico y satisface plenamente nuestros criterios en cuanto a volumen, calidad y pertinencia.

Firma Investigador(a) Patrocinante

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