Bag it!
Intensifying with ultrasound and microfluidics

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June 21st 2016, Newcastle UK
Jeb Berrier, a regular American man, makes a pledge to stop using plastic bags at the grocery store and has his life completely changed.

Initial release: September 1, 2011 (USA)
Director: Suzan Beraza
Screenplay: Michelle Curry Wright
Music composed by: Larry Groupé
Producers: Suzan Beraza, Michelle Hill
Outline

- New PI Principles course in Twente
- Scaling up a microreactor (unaware intensification)
- Bagging valorization benefits
- Future Intensification
PI course in Twente
MSc level

Innovative-entrepreneurial thinking

Process Intensification Principles

Professional behavior
Scales and terminology of this talk

Radicals

Light

Bubbles

Acoustic waves

Microreactors

Milireactors

Ultrasound equipment

Industrial scale

Why is water treatment important?
Among the many examples

Ultrasound applied at high intensity at 20 to 35 kHz generates cavitation.

Creates extreme mechanical shear forces that disintegrates biomass in wastewater.

ovivowater.es
What can one bubble do?

Not these type of bubbles!!

Bubble toothbrush (vimeo.com/104998226)
Difficult Experiments

D. Fernandez Quayside, Newcastle June 2016
D. Fernandez Rivas & S. Wissman, Sattelite bar, EPFL, Dec. 2015
There is more than meets the eye...

- **Free Bubble**
- **Growth by rectified diffusion**
- **Sonoluminescence**
- **Radicals**
- **Shockwave**
- **Collapse**

**Equation**: \( X_{US} = \frac{\Delta H dN_{rad}/dt}{P_{US}} \)

Low efficiencies: \(~10^{-5} – 10^{-6}\)

**Diagram Explanation**:
- **Electricity** to **Oscillating pressure** from **PZT**
- **Collapse against a surface**
- **Bubble collapse close to a surface**
- **Repeated collapse against the surface = Fatigue**
- **Liquid jet** \ (~100 m/s ~ 10 μm)
- **Surface erosion**

**Notes**:
- \(~100 m/s ~ 10 μm\)
- \(~10^{-5} – 10^{-6}\)
Bubbles + Ultrasound + \(\mu\)Fluidics

Fernandez Rivas, et al., Chemical Communications. 48 (89), 10935 - 10947 (2012).
Bubbles + Ultrasound

Our focus

Fernandez Rivas, et al., Chemical Communications. 48 (89), 10935 - 10947 (2012).
Micro-Sono-Reactor

Total volume $\sim 300 \, \mu l$

Fernandez Rivas, et al., Chemical Communications. 48 (89), 10935 - 10947 (2012).
Controlling cavitation as $f(x,y,z,t)$
Physical and chemical effects

Sonochemiluminescence

D. Fernandez Rivas et. al,
Angewandte Chemie Int. Ed.,
(49) 9699 - 9701. (2010).
Physical and chemical effects

Sonochemiluminescence

Sonoluminescence


Power and efficiency

\[ X_{US} = \frac{\Delta H dN_{rad}/dt}{P_{US}} \]

Where is the Energy going?

Bubble rebound, Jet

Jetting, shockwaves and erosion

Can we identify PI in all that?

- Structure – Surface of the reactor
- Time – Ultrasound
- Energy - Alternative
Can we make bubbles work for us?

Make radicals or clean ... ?
Utilization - Valorization

Ready to ship / Webshop
But don’t buy it yet...

Until you see if it works or not with your own eyes!
How to scale-up?

To clean arbitrary shaped objects?

Or for other uses?

- Emulsification
- Graphene
- Nanoparticle synthesis
- Crystallization
- ...
Bags, cavitation and plastic

Poly-propylene  ~10-100 mL

Visible bubbles

Sonochemiluminescence

Does it work better?

Radical generation (OH·)

Does it work better?

Ultrasonic bath 1: 35 kHz (24.2 W/L – 427 kPa, uncertainty of 24%)
Ultrasonic bath 2: 45 kHz (33.3 W/L - 364 kPa).
The 50 µm thick bags allow for 79.4-86.0% of ultrasound transmission.

The microreactor was scaled-up 25 times, with a five-fold increase of its efficiency. The efficiency was increased up to 45.1% compared to bags without pits. Efficiencies are underestimated (ratio of bag to bath volume).

Do you exfoliate?
Nanomaterials Exfoliation

Graphene: a two-dimensional carbon allotrope with excellent optoelectronic properties of use to engineer devices, and functional materials.

Exfoliation of graphene, carbon nanoflower/graphene suspensions and LFP/graphene have caught attention.

Some want to open up the flower.

It is not easy to deal with nanomaterials

Methods
Dispersion and density gradient ultracentrifugation. One gram of MoS\textsubscript{2} powder (American Elements) was dispersed in 70 ml of 2\% w\textsubscript{v} \textsuperscript{-1} Pluronic F68 (BASF) aqueous solution via ultrasonication using a 0.125-inch tip in a steel beaker at 25 W for 2 h. Then, 32 ml of dispersion was carefully added on top of a 6 ml underlayer of 60\% w\textsubscript{v} \textsuperscript{-1} iodixanol and ultracentrifuged at 32 kr.p.m. for 24 h at

2 hours!!!!?
Exfoliating with bubbles

Graphene flakes agglomerate, yet sonication with bubble bags "stretch them";

The suspensions are stable for several months.
Making emulsions

Hexadecane 15% in SDS aqueous solution.

Wrapping it up

• Bubbles can be useful at different scales

• Taming bubbles is possible with crevices

• Scaling-up is “in the bag”

• Many bubbles to come ...

Another use of “bubbles” ...
Thank you for your time ... any questions?

Thanks to the organizers for the invitation to present today!

Many collaborators made possible the results presented
Small bubbles for large scales