#### Bag it!

# Intensifying with ultrasound and microfluidics



**PROCESS INTENSIFICATION NETWORK** 

June 21<sup>st</sup> 2016, Newcastle UK

David Fernandez Rivas Mesoscale Chemical Systems



UNIVERSITY OF TWENTE.



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







WINNER Choise Award ASHILAND





# Outline

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

#### BµBCLEAN <sub>&</sub>





#### Scales and terminology of this talk



D Fernandez Rivas and S Kuhn, Synergy of Microfluidics and Ultrasound, to appear in Current Chemistry, 2016

# 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.

# What can one bubble do?

Not these type of bubbles!!





Bubble toohbrush (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 ...



Collapse against a surface





#### Bubbles + Ultrasound + µFluidics



Fernandez Rivas, et al., Chemical Communications. 48 (89), 10935 - 10947 (2012). D Fernandez Rivas and S Kuhn, Synergy of Microfluidics and Ultrasound, to appear in Current Chemistry, 2016

#### **Bubbles + Ultrasound**



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

# Micro-Sono-Reactor



Total volume  $\sim$  300 µl

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

Controlling cavitation as f(x,y,z,t)



D. Fernandez Rivas et al., American Physical Society, 65th Annual Fall Division of Fluid Dynamics Meeting, 2012. Gallery of Fluid Motion.

#### Physical and chemical effects



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

#### Physical and chemical effects



D. Fernandez Rivas et. al, Angewandte Chemie Int. Ed., (49) 9699 - 9701. (2010). D. Fernandez Rivas et. al, Ultrasonic Sonochemistry., (19) 1252-1259. (2012).

#### **Power and efficiency**

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



D. Fernandez Rivas, et al. Ultrasonic Sonochemistry. (20) 510-524. (2013).

# Where is the Energy going?





D. Fernandez Rivas, et al. Ultrasonic Sonochemistry. (20) 510-524. (2013).

#### Jetting, shockwaves and erosion



Rivas, et al. Ultrasonic Sonochemistry. (20) 510-524. (2013).

# Can we identify PI in all that?



Structure, Energy, Synergy, Time-The Fundamentals of Process Intensification

Tom Van Gerven<sup>†</sup> and Andrzej Stankiewicz\*



- Structure Surface of the reactor
- Time Ultrasound
- Energy Alternative

## Can we make bubbles work for us?



#### Make radicals or clean ... ?

### **Utilization - Valorization**



# But don't buy it yet...

Until you see if it works or not with your own eyes!



# BuBble bags



Patent WO/2015/144918

#### 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 transmision.

#### Does it scales?

	Energy efficiencies after 5 minutes				
$X_{US} = \frac{\Delta H dN_{rad}/dt}{P_{US}}$		Bath 1		Bath 2	
		Non-pitted bag	CIB	Non-pitted bag	CIB
	Efficiency (10 <sup>8</sup> )	1.4	1.9	9.1	13.2
	Efficiency (10 <sup>-5</sup> ), corrected	1.8	2.5	3.3	4.7

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.



Modesto-López, L, et al, (2015) Films of Graphene Nanomaterials Formed by Ultrasonic Spraying of Their Stable Suspensions, Aerosol Science and Technology, 49:1, 45-56, DOI: 10.1080/02786826.2014.991438

# It is not easy to deal with nanomaterials



#### Methods

**Dispersion and density gradient ultracentrifugation.** One gram of  $MoS_2$  powder (American Elements) was dispersed in 70 ml of 2% wv<sup>-1</sup> 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 underlaver of 60% wv<sup>-1</sup> jodixanol and ultracentrifuged at 32 kr p m for 24 h at



# 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.



Rivas, D.F., et al, 2015. A novel ultrasonic cavitation enhancer. Journal of Physics: Conference Series (Vol. 656, No. 1, p. 012112).

#### $PI + US + \mu Fluidics$



D Fernandez Rivas and S Kuhn, Synergy of Microfluidics and Ultrasound, to appear in Current Chemistry, 2016

#### Present and future



D Fernandez Rivas and S Kuhn, Synergy of Microfluidics and Ultrasound, to appear in Current Chemistry, 2016

# 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?



Many collaborators made possible the results presented











# Small bubbles for large scales



Turbine rotor - vóór ultrasone reiniging

Turbine rotor - na ultrasone reiniging