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► **To cite this version:**

M. Ollivier, G. Borda, J. Flouret, S. Charton. Innovative precipitation in emulsion process application to the precipitation of bismuth subnitrate. ECCE10 + ECAB 3 + EPIC 5 - 10th European Congress of Chemical Engineering + 3rd European Congress of Applied Biotechnology + 5th European Process Intensification Conference, Sep 2015, Nice, France. cea-02509187

HAL Id: cea-02509187

<https://cea.hal.science/cea-02509187>

Submitted on 16 Mar 2020

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Innovative precipitation in emulsion process

Application to the precipitation of bismuth subnitrate



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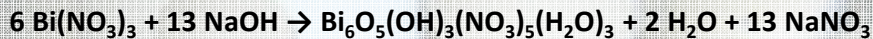
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MOTIVATION AND CONTEXT

A precipitation in emulsion process has been proposed by CEA for the continuous precipitation of radioactive elements [1]. It is based on the **containment of the chemical reagents and solid products into droplets** of aqueous phase dispersed in an inert organic phase, while nucleation is initiated by the drops coalescence.

The division of the reaction volume into droplets overcomes the problems due to injection and micro-mixing [2], usually responsible for reproducibility issues in industrial precipitation. Moreover, prior studies with cerium oxalate [3] have highlighted a **strong correlation between the emulsion properties and those of the particles produced**, thus enabling the size and morphology of the powder to be tuned by varying the droplets properties (e.g. size, velocity and residence time).

Beyond nuclear applications, emulsion precipitation appears as an attractive alternative to conventional processes for the synthesis of high-value precipitates. Bismuth compounds, produced by OCM, have many applications including pigments, paintings, cosmetics, pharmaceuticals, etc. [4]. In this study, the feasibility of bismuth subnitrate (BSN) precipitation, a veterinary product, is investigated.



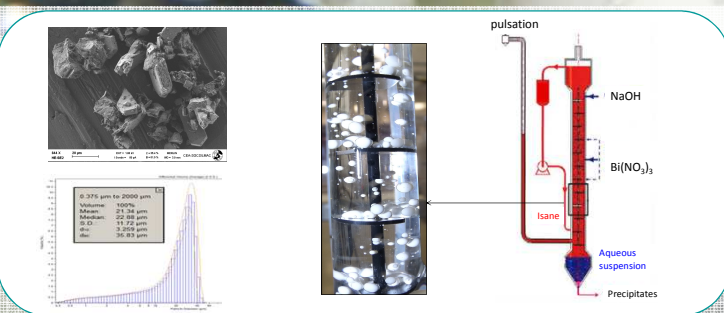
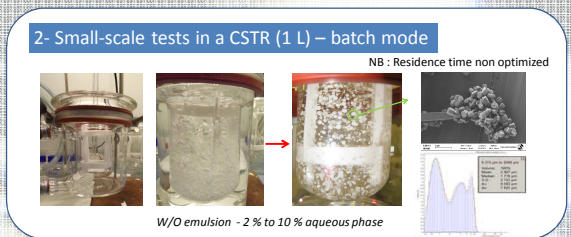
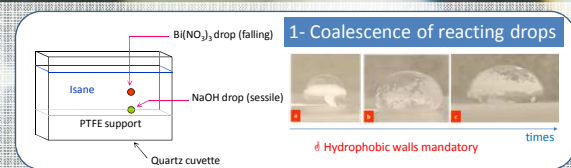
Various BSN grades, including the most preferred $[\text{Bi}_6\text{O}_5(\text{OH})_3]^{5+}$ and $[\text{Bi}_6\text{O}_4(\text{OH})_4]^{6+}$ products can be obtained depending on the pH value [5]. For OCM, possible improvements concern the control of the **product aspect (size, apparent density, color) and purity** at industrial scale. A better **temperature control** of the industrial process is also expected.

EXPERIMENTAL STUDY: METHODOLOGY AND RESULTS

Several aspects have to be considered in order to assess the relevance and feasibility of BSN precipitation in emulsion. A comparative study, based on our feedback regarding oxalate precipitation, was implemented before testing the implementation in a lab-scale pulsed column.

Hence, we gradually verified:

1. The reagents non-solubility and the chemical compatibility of BSN with the (inert) organic phase (e.g. Isane);
2. The efficient triggering of the reaction by the coalescence and the long-term confinement in the daughter drop;
3. The emulsification of NaOH droplets in oil in a CSTR, under similar hydrodynamic conditions as in the column;
4. The efficient droplets circulation during the growth of the (dense) solid particles;
5. The emulsion settling and the effective BSN recovery;
6. The Bi content of the produced particles, their size distribution, structure and shape



Last step: Continuous precipitation in a 15-mm diameter pulsed column and preliminary parametric study :

- Varying pulsation amplitude and frequency (i.e. drop size and volume fraction)
- Varying organic phase flow-rate (i.e. drops residence time and volume fraction)
- Varying reagents feed-rates and ratio (i.e. residence time and supersaturation)

Feasibility and relevance demonstrated. Needs for a more systematic parametric study and modelling !

TOWARDS INDUSTRIAL PILOT ...

[1] Borda, G. et al. Patent WO 2008/025823

[2] Charton, S. et al., Chem. Eng. Res. Des. (2013) 911(4):660-669

[3] Borda, G. et al., Nuc. Eng. Des. (2011) 241:809-81

[4] Flouret, J. et al., STPMF2015 Proceedings, Nancy.

[5] Miersch, L. et al., Inorg. Chem. (2012) 51: 9376-9384

