



**DUTCH-BELGIAN BEAMLINE  
AT ESRF**

**EUROPEAN  
SYNCHROTRON  
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## **Experiment Report Form**

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

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|---|--|---|
|   | Experiment title: <b>Do compost biosurfactants have a macromolecular nature?</b> | <b>Experiment number:</b><br><b>26-02-422</b> |
| <b>Beamline:</b><br>BM-26B 'Dubble'   | <b>Date(s) of experiment:</b><br>From: 28-7-2008<br>To: 30-7-2008                | <b>Date of report:</b><br>8-8-2008            |
| <b>Shifts: 6</b>  | <b>Local contact(s):</b> dr. K. Kvashnina  |   |
| <b>Names and affiliations of applicants</b> (* indicates experimentalists):<br><b>V. Boffa*</b> , <b>D. Perrone*</b> (University Torino); <b>H.L. Castricum*</b> , <b>J.E. ten Elshof*</b> , <b>S.A. Veldhuis*</b> , <b>T. Stawski*</b> , <b>S. Khan*</b> (Inorganic Materials Science, Universiteit Twente); <b>G. Paradis*</b> (ECN). |  |   |

### **Report: (max. 2 pages)**

Urban food wastes, green wastes and their compost products are interesting sources of organic chemicals, because of their high organic matter content and their low cost [1]. Humic acid-like compounds (cHALs) isolated from compost were found to have surfactant properties [2]. These compost biosurfactants are applicable in a large number of industrial processes, i.e. as detergents and as chemical auxiliary for dyeing. As a textile auxiliary, based on sample color intensity and uniformity, this compost biosurfactant performed as good as SDBS. Moreover the compost biosurfactant is completely biodegradable and enables to catalyze the photodegradation of many synthetic dyes.

At the moment, the great potential that these compounds may have as commercial products does not correspond to a proper knowledge of their chemical structure. We have carried out Small-angle X-ray Scattering (SAXS) measurements at the DUBBLE beamline BM-26B on cHALs at different concentration, temperature, and pH. SAXS analysis was performed on a biosurfactant that was isolated from 110 days old compost, prepared from 67% green urban wastes and 33% humid wastes. DLS (dynamic light scattering, from which the hydrodynamic radius of the molecules is determined) measurements had given indications that this biosurfactant consists of macromolecules which have a different conformation depending on their concentration and the pH and temperature of the solution. Analysis at the tensiometer revealed the biosurfactant has a critical micellar concentration (CMC) at about 4 g/L. Thus, any consideration about the morphology of this compound should not set aside the fact that it can form micelles in water at adequate concentrations.

We obtained data at both short and long detector distance and observed clear differences in the conformation of the biosurfactant and its aggregates. Long acquisition times (1 hour) were applied as the electronic contrast with the solvent (water) is very small, requiring the use of synchrotron radiation. The cHALs were introduced in thin-walled glass capillaries. The applied beam energy was 16 keV, and the sample-detector distance 1.6 and 7.6 m. The CCD detector was used for to monitor wide angle scattering during long-distance SAXS. The samples measured at room temperature are reported in table 1.

Table 1 Fractal dimension of the biosurfactant molecule at different concentration of pH

| conc.                 | pH | 5   | 7   | 11  | 13  |
|-----------------------|----|-----|-----|-----|-----|
| 0.3 g·L <sup>-1</sup> |    | -   | -   | -   | -   |
| 1 g·L <sup>-1</sup>   |    | -   | -   | -   | -   |
| 3 g·L <sup>-1</sup>   |    | 2.7 | 2.2 | 2.2 | 1.5 |
| 10 g·L <sup>-1</sup>  |    | 2.3 | 1.9 | 1.7 | 1.7 |
| 30 g·L <sup>-1</sup>  |    | 1.2 | 1.3 | 1.6 | 1.8 |
| 100 g·L <sup>-1</sup> |    | 1.1 | 1.4 | 1.8 | 1.8 |

The biosurfactant was not completely soluble at pH < 5, so no data points were obtained at lower pH. DLS measurements showed that, for concentrations lower than 5 g/L, this biosurfactant has a hydrodynamic diameter of about 90 nm at pH 5 and room temperature. At neutral pH, the size is 110 nm and it increases up to 140 nm at pH 13. The size increment is more appreciable at 60 C. At pH > 7 and at concentrations higher than 5 g/L the measured hydrodynamic diameter is equal to 120 nm and is not changing with the biosurfactant concentration. At pH = 5 the hydrodynamic diameter is increasing with the concentration and at 30 g/L it is equal to 200 nm. Thus, we suppose that at concentrations lower than 5 g/L isolated macromolecules are present in solution. At concentrations higher than 5 g/L the macromolecules form micelles if the pH is = > 7 and different aggregates at lower pH. The data are well reflected by the fractal dimensions given in Table 1. At low concentration, the molecules have a looser structure and a larger hydrodynamic radius for increasing pH. A similar relation is found at pH=5 for increasing concentration.

Data analysis was challenged by a low signal-to-noise ratio. This was mostly evident at low biosurfactant concentration. For instance, the 3g/L samples already needed an acquisition time of 2 hours, while the scattering spectrum of 1 g/L samples was not distinguishable from the background even after 2 hours of data-acquisition.

While we found good resolution at short distance, optimal focusing for long distance or the application of lower beam energies to allow measurement of scattering at very low  $q$  was not well possible. Still, we were able to measure all samples, and found excellent reproducibility of the scattering patterns, even considering the small variations in sample thickness and background intensity associated with the use of capillaries.

## References

- [1] P. Quagliotto et al. Environ. Sci. Technol. 40 (2006) 1686.
- [2] M. Baalousha et al. Colloids and Surfaces A: 272 (2006) 48.