Pressure Cell for XPD, XAS and SAXS Synchrotron Experiments

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***Abstract*—CuBe hydrostatic cell pressure using B4C anvil have been developed to XPD, XAS and SAXS beam line experiments since 2004. An optimized version of this cell was used to measure the effect of pressure on the structure and electrical conductivity of cardanol-furfural-polyaniline blends in 2010. Recently, a new version was used to investigate orange juice under pressure in SAXS beam line at - LNLS. The results showed the good performance of this new version of hydrostatic pressure cell up to 1.8 GPa. In special, it was possible to visualize the morphology change of structures in the orange juice, when the hydrostatic pressure is higher than 0.5 GPa.**

***Index Terms*— hydrostatic pressure; cell pressure; SAXS**

I. Introduction

Products from fruit have a high concentration of soluble solids (Brix 65-80 ºBrix). The presence of low pH and organic acids inhibit growth of many organisms such as *Salmonella Typhi* and other [1]. Using heat treatments and different concentrations of the pH industry tries to destroy these organisms in fruit juices. However, the bacterium *Alicyclobacillus acidoterrestris* is an organism acidophilic and thermophile, which forming spore able to survive when the temperature increases up to 95° C for 2 minutes inside a pH liquid range of 2.5 up to 6.0 [2].

Controlling the growth of these organisms using only heat treatment or change in pH is not always efficient. In this context the use of high hydrostatic pressure (HHP) was introduced as a non-thermal technology capable of maintaining high capacity preservation of foods without the use of chemical additives [3].

An interesting (HHP) example relates to the case of *Alicyclobacillus acidoterrestris*. Control of microorganisms using thermal cycles is inefficient because they form highly resistant spores. However, a pre-treatment using high pressures up to 900MPa active spore germination. Once activated, a small thermal load imposed upon activation produces efficient results for the destruction of microorganism, which preserve the sensorial product quality [5].

In this work we show a new hydrostatic pressure cell, which developed was based on our previous models [5] and optimized for SAXS measurements under applied magnetic field simultaneously.

II. Experimental

The CuBe with B_4C anvil pressure cell described here was designed and built for use in SAXS and angle-dispersive X-ray scattering experiments under hydrostatic pressures up to 1.2 GPa.

The first advantage of the B_4C anvil pressure cell is that anvils of sintered B_4C yield an X-ray absorption spectrum free of Bragg peaks, in contrast to spectra taken through diamond anvils. The second advantage over conventional diamond anvil cells (DAC) is the large transmission coefficient of incoming X-rays (>60% at 10 keV) and the applicability of the B_4C cell in experiments that require lower pressure (up to 1.0 GPa) compared to the conventional DAC pressure range. The third advantage is the relatively low costs (approximately half the price of a DAC) for development and manufacture. The fourth and main advantage of this home-built B_4C anvil pressure cell is that the body of the cell is made of CuBe alloy to allow magnetic measurements without interference. Using this concept, it was possible to measure ac magnetic susceptibility, X-ray absorption and diffraction using synchrotron radiation with the same pressure cell. Details of B_4C anvils building was described before [6].

Hydrostatic pressure conditions were obtained by filling the CuBe gasket hole (2×10^{-3} m in diameter and 0.45×10^{-3} m thick) with orange juice. The inner pressure was calibrated by a superconducting $Hg_{0.8}Re_{0.2}Ba_2Ca_2Cu_3O_{8.8}$ (Hg,Re-1223) manometer biological insolating placed in the gasket cavity and mounted in the inner gasket border, forming a ring outside the X-ray beam spot [6]. For X-ray diffraction experiments, the sample was placed in the center of this superconductor ring. The inner hydrostatic pressure is linearly dependent on the critical superconducting temperature in the optimal oxygen-doped Hg,Re-1223 superconductor, with dT_c/dP=1.9 GPa K^{-1},

Figures 01, 02, and 03 shows several details of the new SAXS cell pressure made with CuBe body.

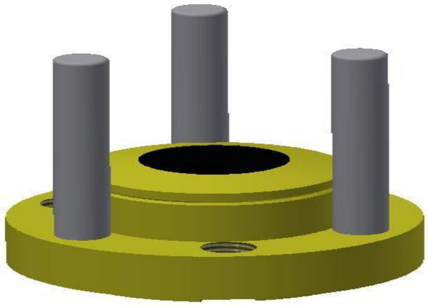

Figure 01. The bottom part of the SAXS pressure cell. The diameter of this cell is 1 inch and the total thickness 0.9 inch.

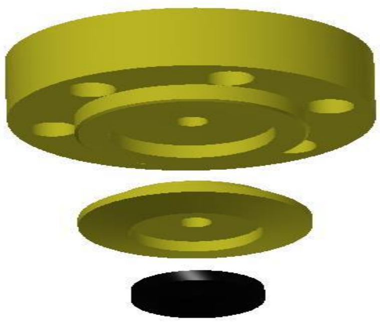

Figure 02 - Schematic view of top CuBe body. The black plate is B_4C anvil. The B_4C is incrusted in CuBe cell top body .

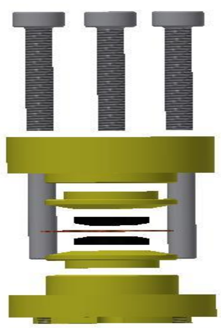

Figure 03 - Exploded schematic view of pressure cell setup. The black part is the B_4C anvil. The crews are used to clamp the pressure, after the pressure cell is charged.

The measurements are performed in National Synchrotron Light Laboratory (LNLS) - Campinas - Brazil SAXS beamline. It was selected the Q range from 0.08 up to 1.3 nm^{-1} to investigate the effect of pressure on the spore present in the orange juice.

The result of pressure effects in SAXS experiments on orange juice are shown in the Figure 04

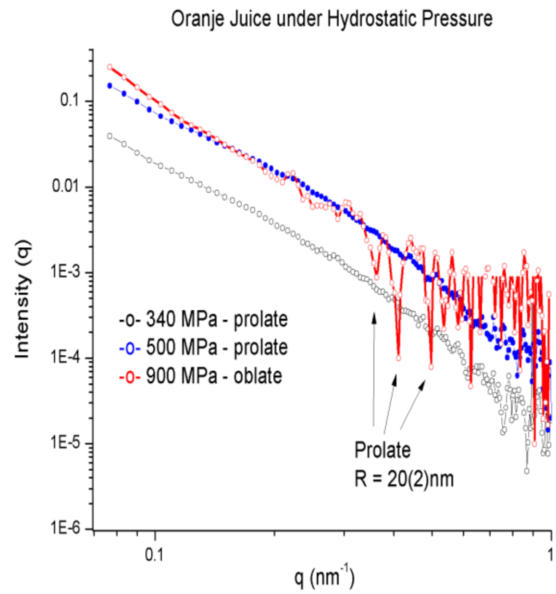

Figure 04 - The behavior of morphology of spore inside the orange juice as a pressure function. The morphology changes for pressure higher than 500MPa.

III. Conclusion

The Figure 4 reveals that the spore presented on the orange juice change its morphology from ellipsoid probate to oblate when the pressure is higher than 500MPa. These results are in agreement with others researches [4]. The pressure cell did not present any leakage and the pressure could be increased without open the cell as a continuum procedure. This new cell will be used new experiment at LNLS Synchrotron Laboratory to extend the Q range up to 3.0.

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