Experimental Studies of solubility of Elemental Sulphur in Supercritical Nitrogen

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1 Introduction

The chemical engineering department of LaTEP has been working for many years on the problem of sulphur deposition especially in natural gas network. The solid sulphur appears immediately downstream of a pressure reduction facility. One of the hypotheses proposed to explain the solid formation and based on a thermodynamic approach is the desublimation of sulphur. During gas expansion, the pressure and the temperature both decrease. Consequently, the gas may become over saturated in sulphur. Because we are below the temperature of the triple point of sulphur, part of the gaseous sulphur can be transformed into solid particles. Thus, it is important to obtain the solubility data of sulphur in natural gases. CO_2 and N_2 are major components of natural gas. In this paper, solubility of elemental sulphur was measured in CO_2 and N_2, with temperatures and pressures respectively up to 363.15 K and 30 MPa.

2 Experimental apparatus

The principle of the experimental pilot can be resumed following three steps: saturation of the gas with sulphur, trap of all dissolved gaseous sulphur and finally quantification. Although the principle is simple, experimental difficulties occur at the three steps. Moreover their interdependency has to be taken into consideration.

Classically an equilibrium cell is utilised to provide gas saturation. Nevertheless, since sulphur solubility value is weak and decrease with decrease of pressure, the volume cell is necessarily important. Moreover, to keep constant the cell pressure during the trap step, the volume must be variable. To ensure that, the cell is equipped with a piston and a nitrogen counter-pressure.

To trap the sulphur in the saturated gas, we developed an original gaseous sulphur trapping method. Its principle is based on reactive absorption with solvent. The gas passes through a pressurised separarator and bubbles into a liquid solution, which traps gaseous sulphur. After that, the gas pressure is reduced to 1 atmosphere and it bubbles in 2 trapping solutions to capture residual sulphur.

Finally the trapping solutions are analysed by gas chromatography to determine sulphur solubility in the gas for the chosen temperature and pressure conditions.

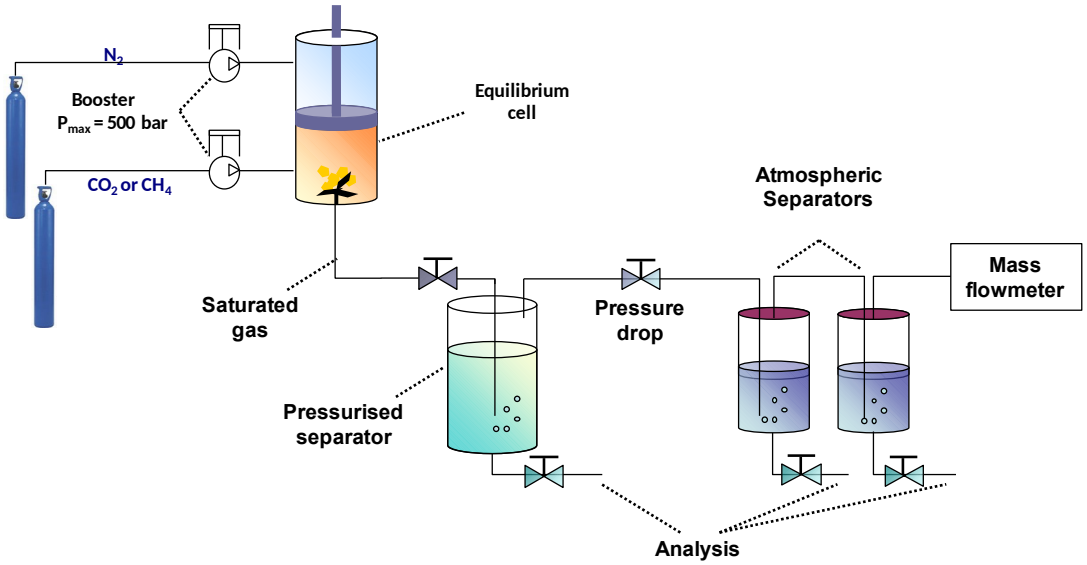

Figure 1: Apparatus for measurement of solubility of sulphur.

3 First results

We have measured sulphur solubility in supercritical carbon dioxide in the pressure range of 10–30MPa, for 363.15K. Our experimental data are close to those reported by [1,2,5].
We observed the influence of pressure on solubility. Indeed, at fixed temperature, solubility of sulphur in CO_2 increases with pressure.
Preliminary results for N_2 indicate a similar comportment of sulphur solubility with pressure.

4 Conclusion

We developed an original experimental apparatus based on a review of the experimental works available of sulphur solubility[3,4].
This new experimental device presents numerous advantages compared to the previously existent ones. The main advantage is that trapping the sulphur before the pressure drop prevents solid sulphur formation in device lines.
Future works will be dedicated to study sulphur solubility in methane, the main component of natural gas.

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