

# Photopyroelectric technique applied to sodium alginate hydrogel characterization

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The search for new friendly and biodegradable products on scientific and industrial demand has been increasing in the last years. Hydrogels based on polymeric and biopolymeric materials forming a three-dimensional network, are considered promising options in medical and pharmaceutical fields, as well as in food industry, being used as emulsifiers, stabilizers and encapsulating agent. Among them is sodium alginate (SA), a water-soluble polysaccharide, isolated from brown algae species. The chemical structure, consisted of high molecular weight linear copolymers of  $\beta$ -D-mannuronic and  $\alpha$ -L-guluronic acids, presents selective binding capacity for polyvalent cations (e.g.,  $\text{Ca}^{2+}$ ). The gel is formed by ionic exchange between  $\text{Na}^+$  and  $\text{Ca}^{2+}$  ions, crosslinking the polymer chain in egg-box structure, which is achieved in mild conditions.

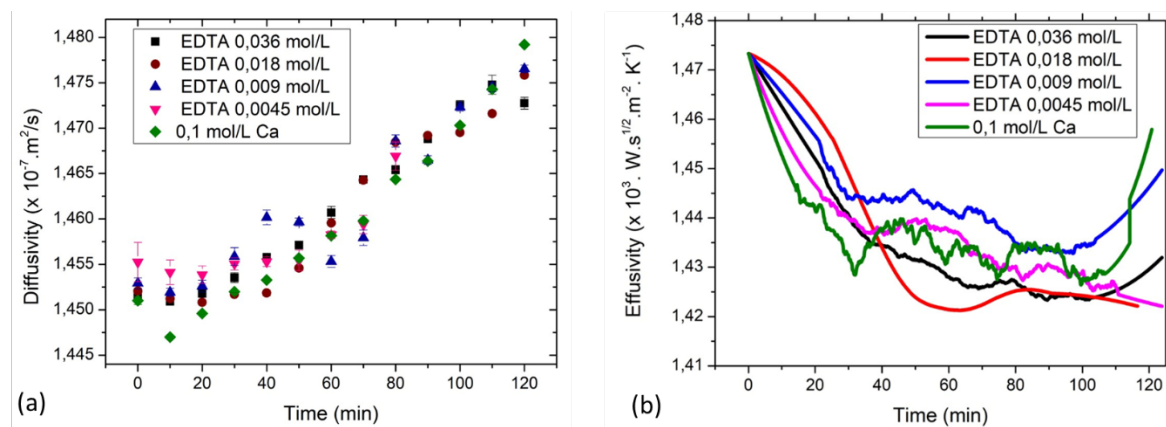
Among the photothermal techniques, the photopyroelectric (PPE) methodology is the most appropriate for a complete thermal characterization of liquid and pasty samples. The temperature oscillations are measured by a pyroelectric transducer, in contact with the sample, by induced changes in the surface polarization charge over the sensor electrodes, which is detected as ac electric signal by a lock-in analyser. The PPE technique enables the investigation of the dynamic thermal properties such as thermal conductivity, diffusivity and effusivity, in addition to the specific heat [1].

In this work, we used the back (BPPE) and front (FPPE) photopyroelectric configuration, to measure thermal diffusivity and effusivity, respectively, of sodium alginate. Aqueous solutions of Sodium Alginate in different concentrations (1, 2 and 3%) were measured, as well as standard samples used as reference. Besides, for the 3% sample, we measured thermal diffusivity and effusivity during the gelation process, which happens simultaneously with drying.

In the experimental setup, we used a 305  $\mu\text{m}$  thick PZT (lead–titanium–zirconate) sensor, for both BPPE and FPPE. The radiation source was a 60mW diode laser (630 nm) electronically modulated. For the BPPE configuration, the sample thickness is controlled by an attached micrometer. For the FPPE configuration, a frequency scan is performed. In both configurations the sample and sensor were considered thermally thick, and the pyroelectric signal was measured by a SR830 lock-in analyser, using current mode. The hydrogels were produced using sodium alginate (Sigma-Aldrich) and Ca-EDTA (Ethylenediamine tetraacetic acid) (Sigma-Aldrich), with glacial acetic acid (Vetec, P.A) being used to release  $\text{Ca}^{2+}$  and give rise to the ionic exchange/gelation.

Measurements of thermal diffusivity and effusivity for reference samples, as ethylene glycol, water and ethanol, are in agreement with literature (accuracy of 1-3%). SA samples were characterized in three

concentrations (aqueous solutions of 1%, 2% and 3%) and the values of thermal diffusivity and effusivity do not present significant differences, with values in the ranges  $(1.45 - 1.46) \times 10^{-7} \text{ m}^2/\text{s}$  and  $(1460 - 1510) \text{ W}\cdot\text{s}^{1/2}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ , respectively.



**Fig. 1. (a)** Thermal diffusivity and **(b)** thermal effusivity of the 3% alginate sample as function of time.

Figure 1(a) shows the thermal diffusivity of the 3% alginate sample, for several EDTA concentrations. The values seem to be constant until 30 minutes and then present a linear increase. The same behaviour is observed for all EDTA concentrations. For the same alginate concentration (3%), figure 1(b) presents the thermal effusivity as function of time, for several EDTA concentrations. We observe a slightly decreasing during the first 30 minutes followed by a noisy constant-like behaviour.

The PPE methodology showed to be suitable for hydrogels thermal characterization and sensitive to changes promoted by the gelation process. It is worth to mention that we also have the drying of the hydrogels, which happens simultaneously to the gelation (data not shown). It is well known that the gelation process occurs mainly during the 20-30 minutes [2], so that thermal effusivity seem to be more sensitive than thermal diffusivity, which probably undergo the changes in Fig. 1(a) more due to drying than the gelation.

## References

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- [2] P.O. Vargas et al., Shrinkage and deformation during convective drying of calcium alginate, *LWT - Food Science and Technology*. 97 (2018) 213-222. <https://doi.org/10.1016/j.lwt.2018.06.056>