

Cite this: DOI: 00.0000/xxxxxxxxxx

Hydrogen and graphite production from polymer degradation in molten hydroxides salts[†]

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Received Date

Accepted Date

DOI: 00.0000/xxxxxxxxxx

Supplementary information for the publication



Fig. 1 picture of cage, front and back view

1 Pictures of the system

Here are presented the pictures of gold cage used to immerse the sample into the molten salt. This device prevents efficiently the samples from being destroyed by heat at the surface of the molten salt and allow the reaction to be mediated by the salt and the different species that are solubilized inside. The volume of the cage is approximately $1.5 \times 1 \times 1 \text{ cm}^3$ and about 1 g of sample is introduced for each treatment.

2 Mass spectra of gas emitted

The mass spectra emitted during the treatment of the hexanoic acid are shown in fig. 2 and of the decanoic acid in fig. 3. The carboxylic acids are successively immersed in the same bath. Each introduction of acid is performed when the previous reaction is

completed. The acids are treated in the following order: butanoic acid 4 C, hexanoic 6 C and decanoic 10 C.

3 Residues, filter and filtrate

After the reaction, the block of cold salt is recovered and dissolved in distilled water, then filtered to recover the solid fraction of graphite carbon. However, carbon losses are observed during this step, in particular in the form of nanoparticles lost in the filter fig. 4a and in the filtrate fig. 4b. The latter contains nanographite particles in suspension, visible by the Tyndall effect when a laser passes through the solution.

Due to these losses, the mass of carbon formed can only be estimated. Moreover, in the neoprene case, the CHONS elemental analysis table 1 indicates a carbon content of 45.8 wt%.

Experimentally, after reaction we collect a residual carbon mass of about 32% \pm 3.5% of the initial sample mass, which corresponds to a difference of approximately 13% with the CHONS results. This difference can be related to carbon lost in the filter and filtrate. Even if a perfect quantitative analysis cannot be carried out, these two values of the same order of magnitude clearly show that the majority of the carbon in the sample is found in the form of graphite and not released in the form of carbonate or CO_2 .

Table 1 CHONS elemental analysis of neoprene before treatment

Sample	C [wt%]	H [wt%]	N [wt%]	S [wt%]
Neoprene	45.8	4.825	0.48	1.650

Conflicts of interest

There are no conflicts to declare.

Notes and references

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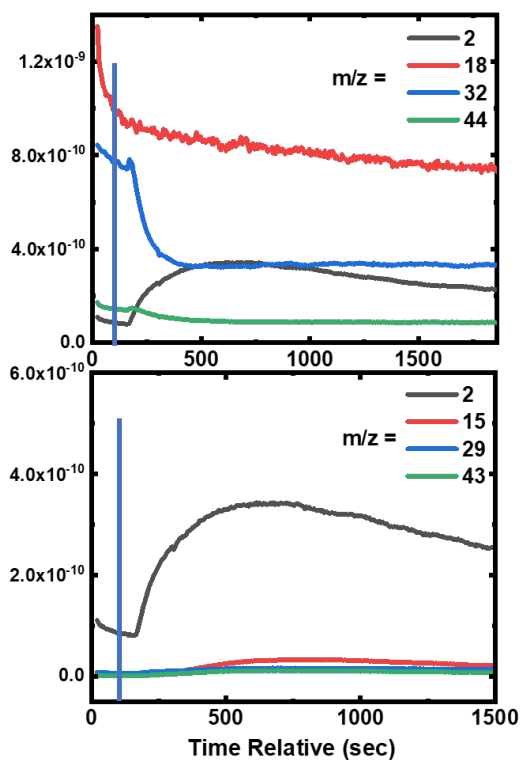


Fig. 3 Mass spectra of gas emitted during the treatment of decanoic acid, the vertical line corresponds to the insertion of sample after a stabilization time of the atmosphere. Spectrum a) with $m/z=2$ for H_2 , 18 for H_2O , 32 for O_2 and 44 for CO_2 , Spectrum b) Relative comparison in alkanes/ H_2 intensity with $m/z=2$ for H_2 , 15 for CH_3 , 29 for C_2H_5 and 43 for C_3H_7

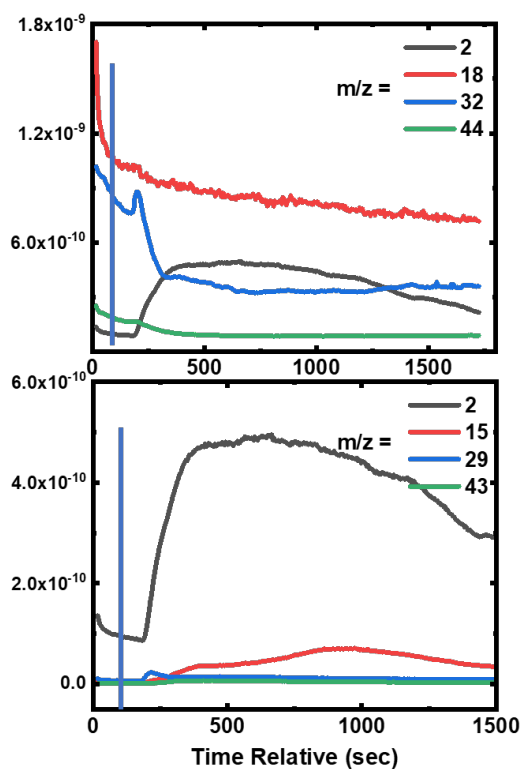


Fig. 2 Mass spectra of gas emitted during the treatment of hexanoic acid, the vertical line corresponds to the insertion of sample after a stabilization time of the atmosphere. Spectrum a) with $m/z=2$ for H_2 , 18 for H_2O , 32 for O_2 and 44 for CO_2 , Spectrum b) Relative comparison in alkanes/ H_2 intensity with $m/z=2$ for H_2 , 15 for CH_3 , 29 for C_2H_5 and 43 for C_3H_7

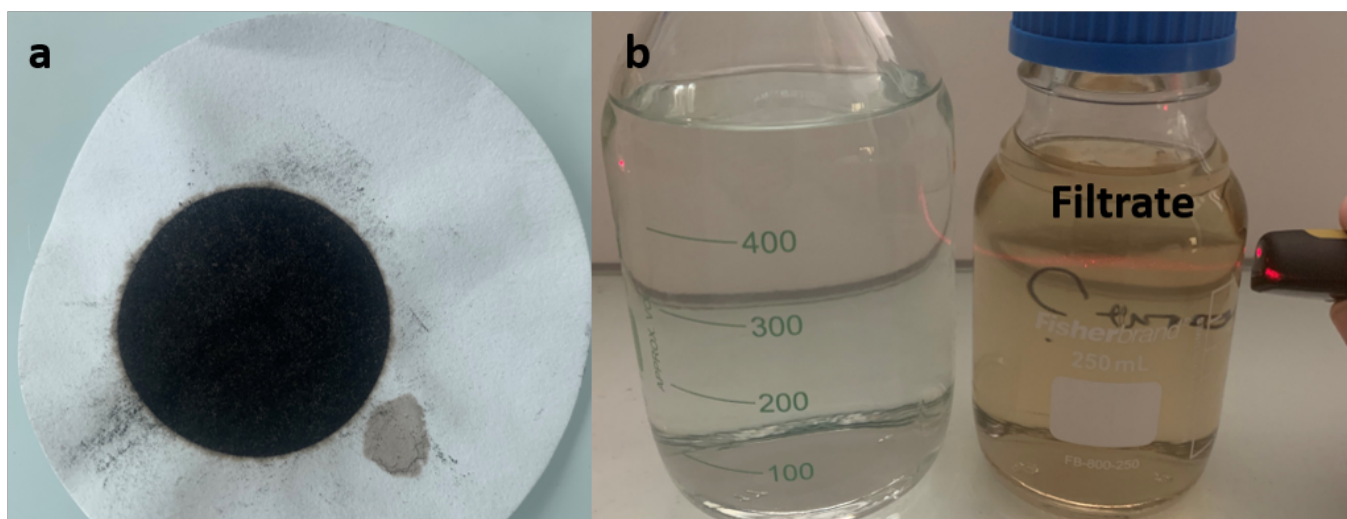


Fig. 4 Photographs of filter (a) and filtrate with Tyndall effect(b)