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## Ultrasound contribution to cation substitution in LTA zeolite

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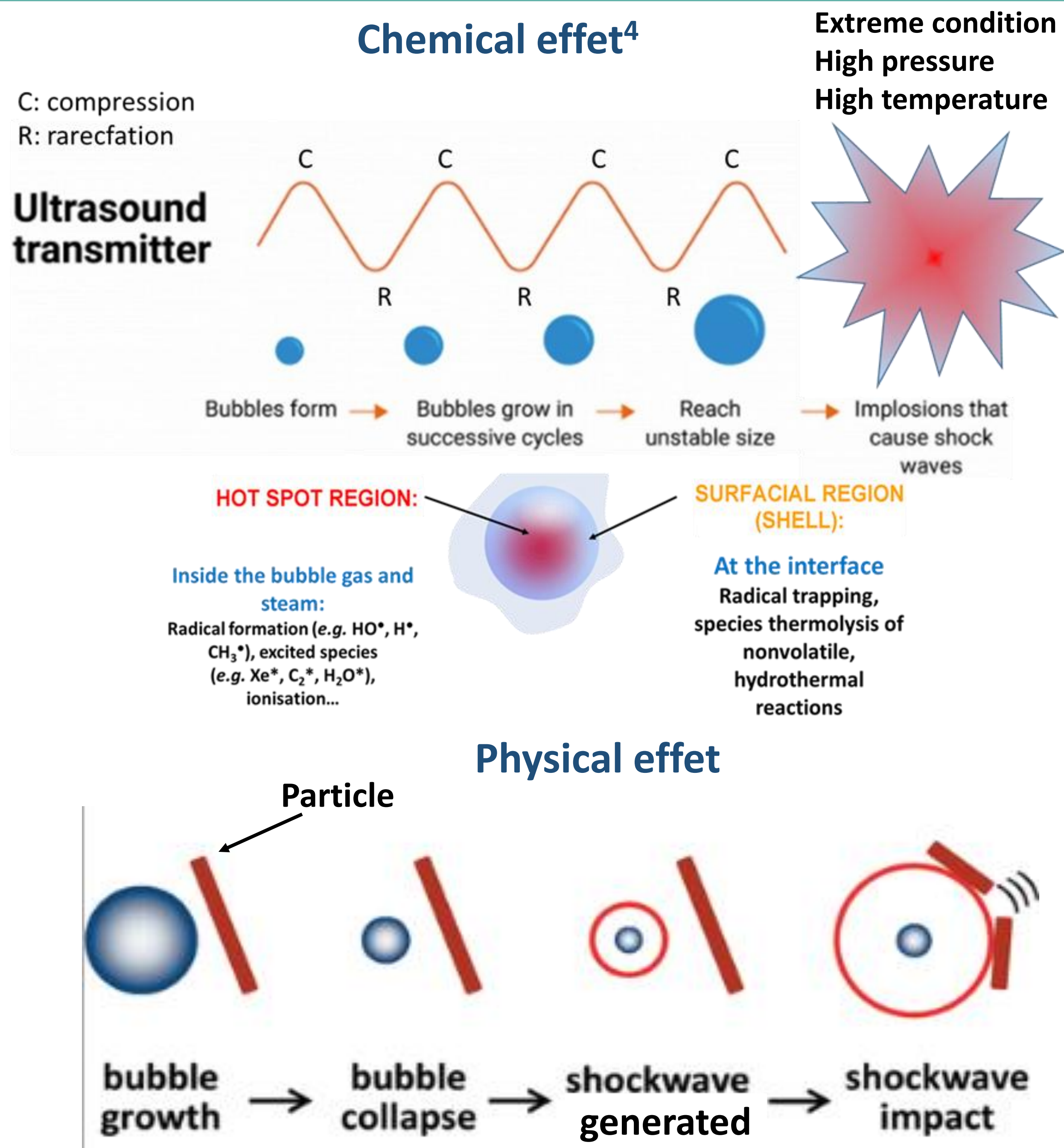
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## 01 Introduction

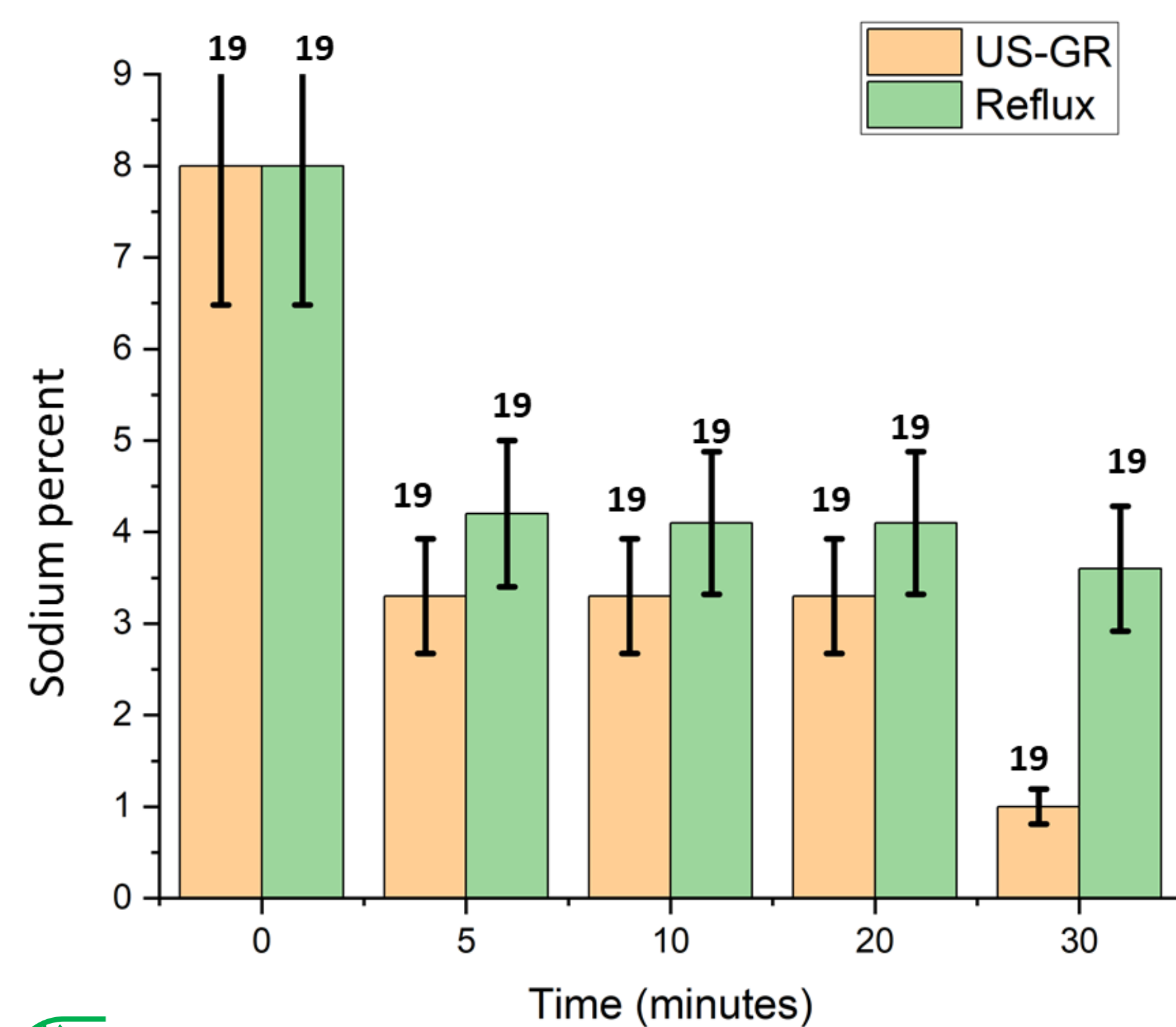
The physical and chemical effects of power ultrasound derive from acoustic cavitation. Ultrasound-assisted synthesis and treatment under hydrothermal conditions using a sonohydrothermal (SHT) reactor has been developed in our group during the last decade<sup>1</sup>. Recently, ultrasonic treatment has received much attention for the preparation of zeolites due to a significant acceleration of their growth/crystallization, which considerably reduces the overall synthesis time<sup>2-3</sup>. Herein we propose the use of power ultrasound as an alternative to zeolite A synthesis and conventional reflux cation substitution methods. The cation exchange of Na-A zeolite to the protonic form (H-A zeolite) was then investigated for the first time under ultrasound in a glass reactor (US-GR) with a plunging sonotrode. We have also studied the ultrasonic-assisted cation exchange of Na-A zeolite against another extra-framework cation ( $\text{Ca}^{2+}$ ) at 20 kHz. The reflux cation substitution procedure was used as a benchmark method.  $\text{Na}^+ \rightarrow \text{Ca}^{2+}$  exchanged zeolite A is indeed extensively used as an adsorbent in petroleum and gas purification applications.

## 02 Sonochemistry: acoustic cavitation



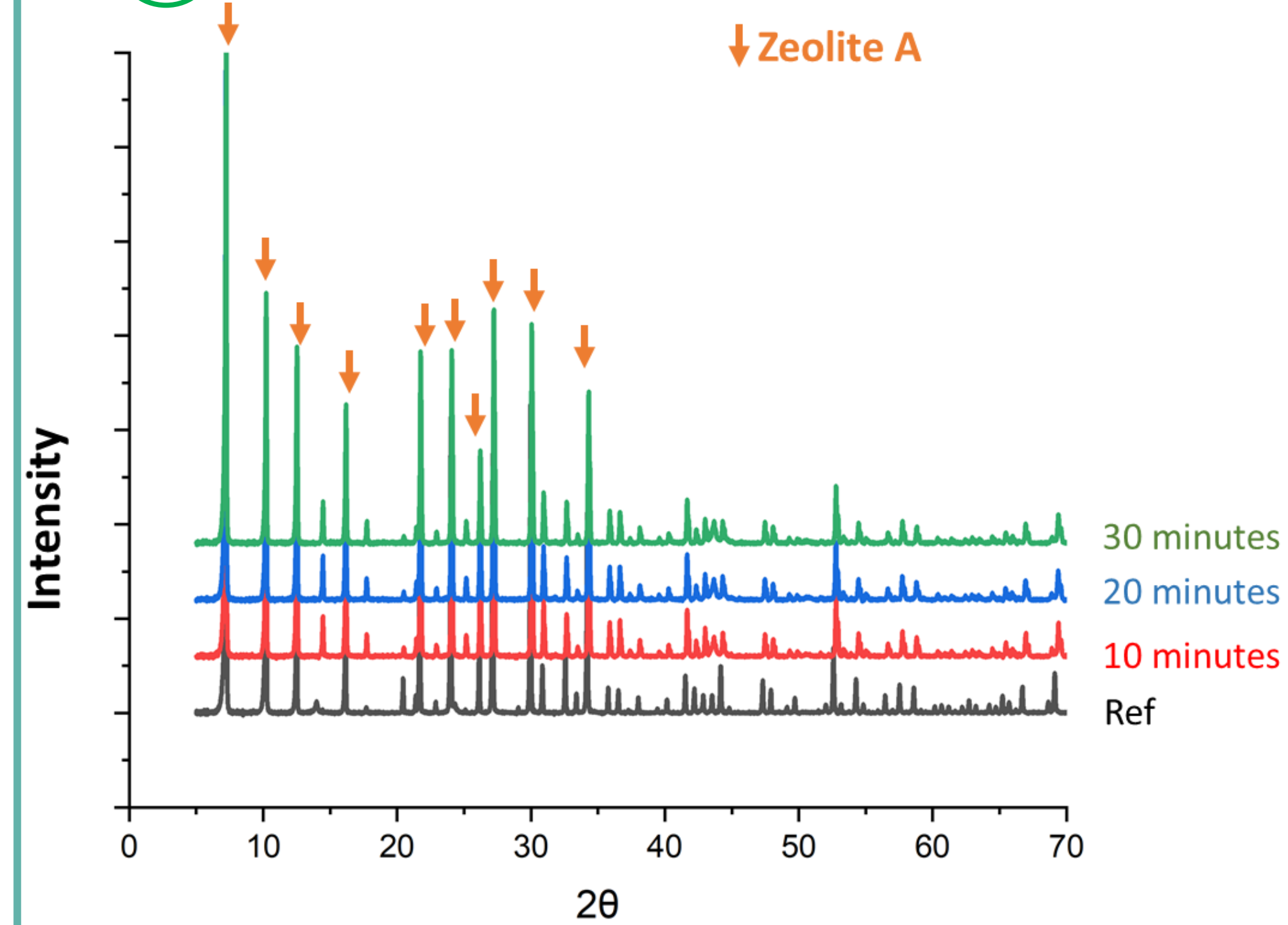
## 05 Results and discussion

**EDS analysis: evolution of sodium content in % at in calcium-substituted zeolite. Comparison between US-GR/ reflux method.**



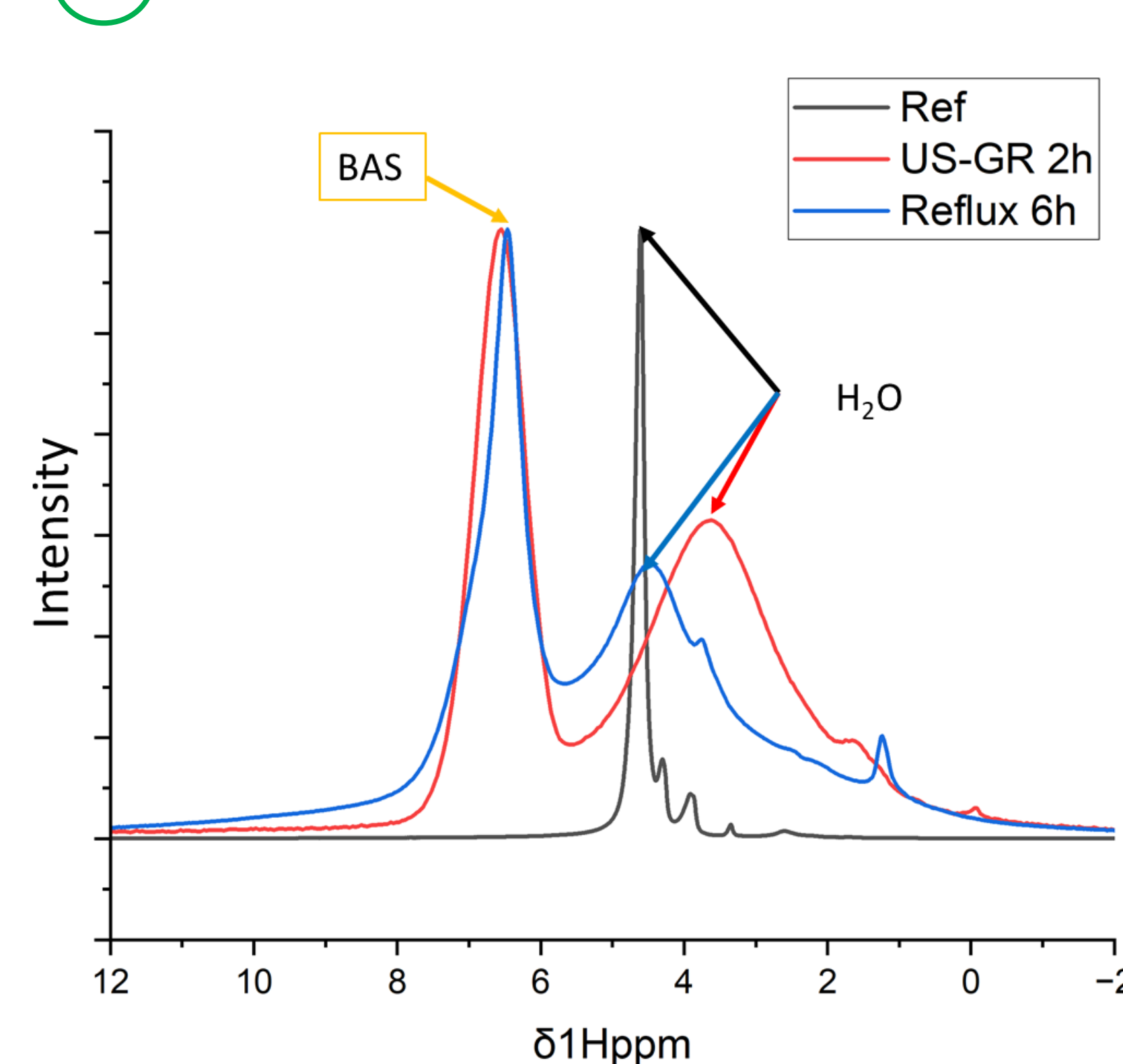
The degree of cation exchange is **higher** for the materials treated **under ultrasound** than under reflux.

**XDR pattern: Ca exchange US-GR**



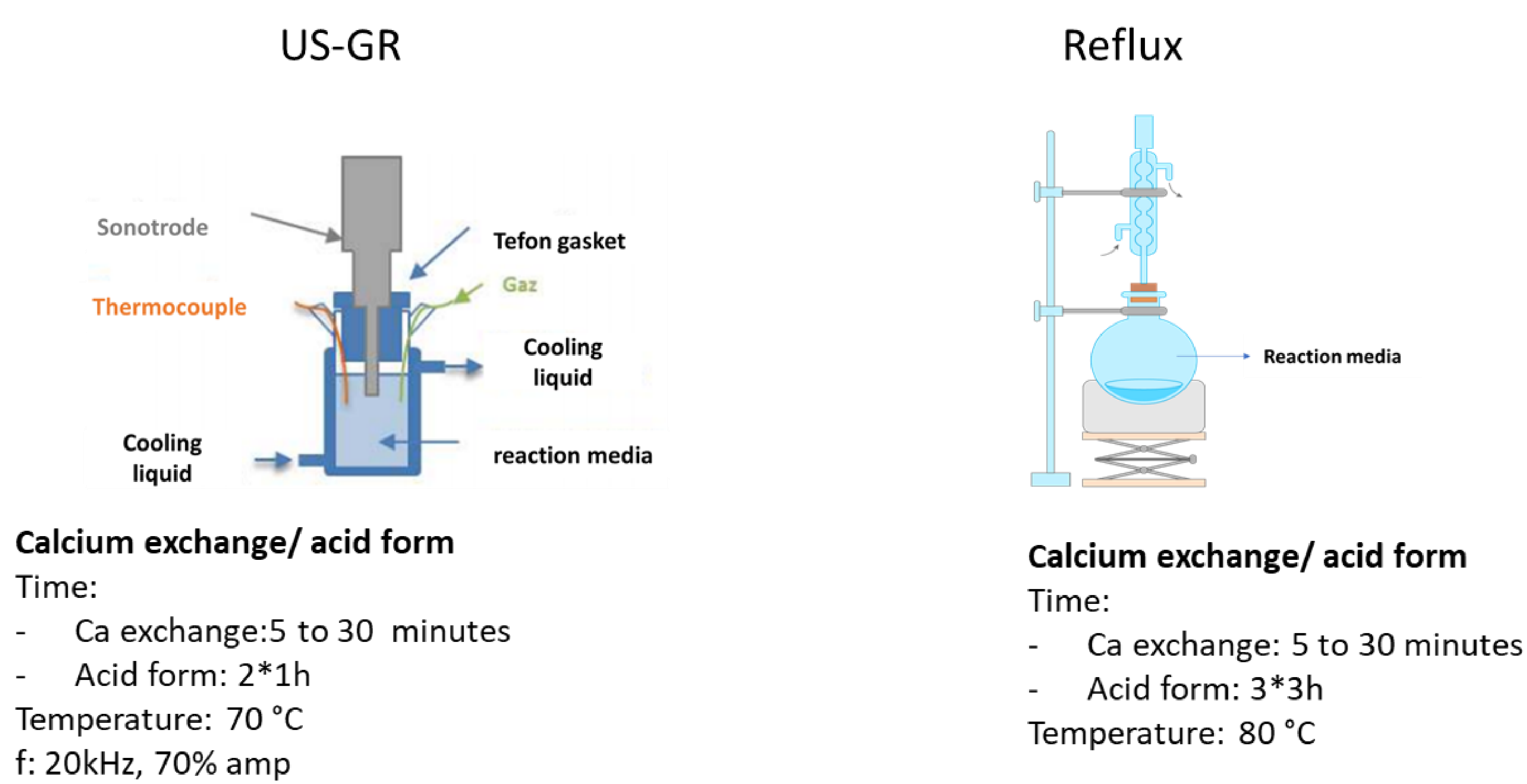
The use of the **ultrasound-assisted** procedure for cation exchange has **no impact** on the zeolite structure after treatment as confirmed by XRD analysis.

**<sup>1</sup>H NMR spectrum of treated compound (acid form)**

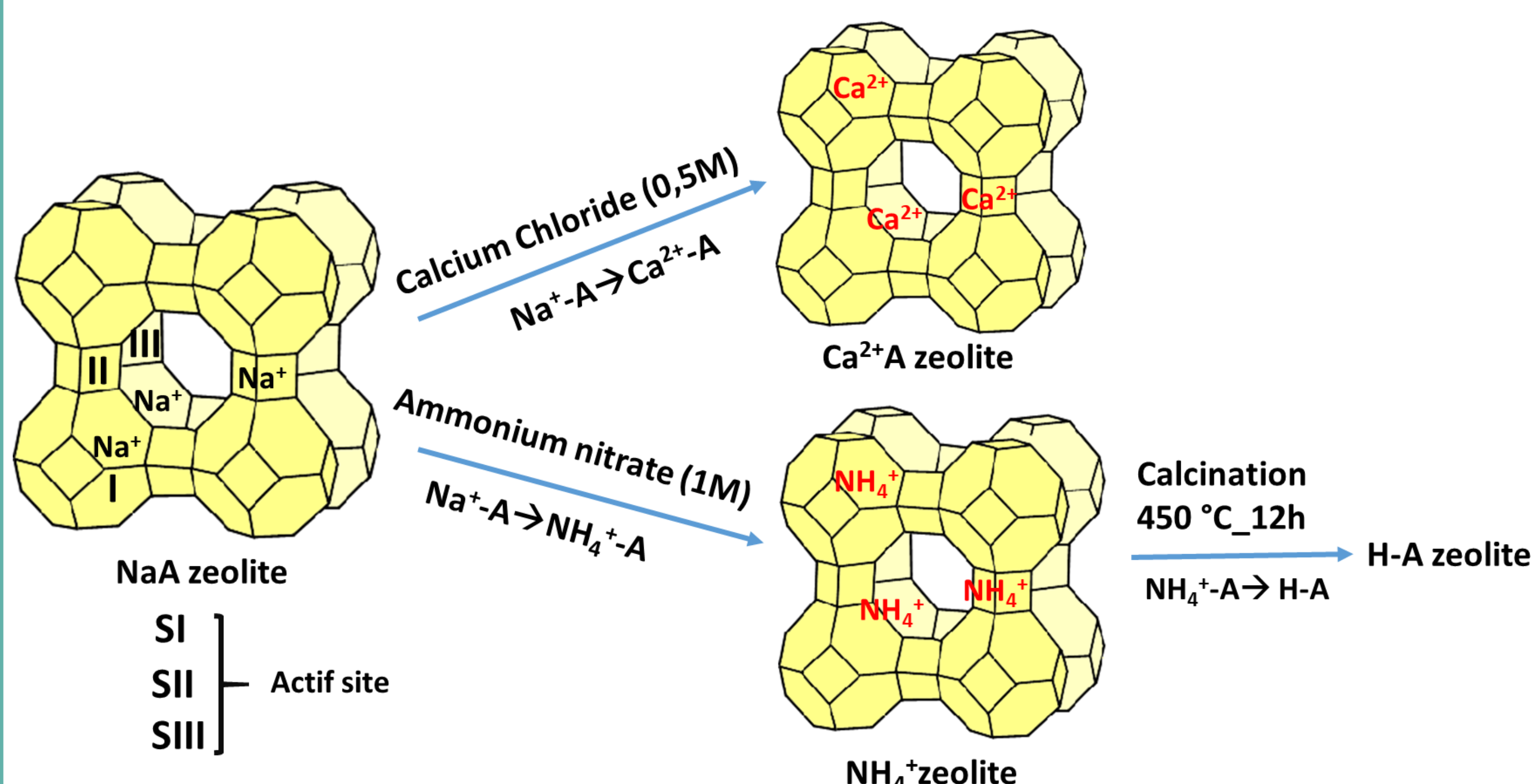


The NMR spectrum shows that **using ultrasound** to obtain the acid form is **more efficient** than reflux. Indeed, the intensity of the acid proton (BAS: Base Acid Site) is at its **maximum after 2h in US-GR** compared with 6h in reflux.

## 03 Experimental condition



## 04 Ion exchange, mechanism



## Conclusion

Substitution of sodium by calcium is **3 times faster under ultrasound**. Similarly, the transition to the acid form is **3 times faster under ultrasound** than under reflux. The positive effect of ultrasound on the extent of cation exchange can be attributed to an acceleration of mass transfer thanks to cavitation bubbles and the inherent physical effects observed under low-frequency ultrasound.

## 06 Reference

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- [4] Nalesso & al, US 57, 125–138 (2019)