Transformation of construction and demolition waste into high value zeolitic materials

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Abstract: Zeolites, microporous crystalline materials composed mainly of silicon and aluminum, possess physicochemical properties that make them valuable in water treatment and catalysis applications. Using construction and demolition waste (CDW) as raw material for producing zeolites represents a sustainable strategy to reduce its environmental impact. Thus, the present work addresses the synthesis of zeolites from CDW within the framework of the circular economy. Crushed brick waste was used and subjected to a hydrothermal process in a stainless-steel reactor with 2 M KOH, at 160 °C, for 6, 8, 10, and 12 hours. After treatment, the products were characterized by powder X-ray diffraction to evaluate their crystallinity. The results showed a progressive evolution in the formation of crystalline phases: at 6 hours an amorphous material was obtained; at 8 hours the first signs of nucleation of ordered phases were identified; at 10 hours zeolitic structures were consolidated, including aluminosilicates typical of zeolites, such as phillipsite; and finally, at 12 hours secondary phases were observed. It is concluded that it is feasible to transform CDW into functional zeolites by hydrothermal synthesis, with an optimum reaction time of about 10 hours. This approach allows the valorization of solid waste and contributes to the production of high-value materials, promoting sustainable practices in the materials science field.

Keywords: circular economy, phillipsite, bricks, hydrothermal synthesis, zeolites

Introduction

Zeolites are microporous materials composed of crystalline structures consisting of three-dimensional tetrahedral arrangements of silicon (Si^{4+}) and aluminum (Al^{3+}) cations surrounded by oxygen anions (O^{2-}), which form channels and cavities of molecular size. According to their origin, zeolites can be classified into three main categories: natural, synthetic, and modified (Gallo-González & Vázquez-Rodríguez, 2021). Naturally occurring zeolites are generated when there is an abrupt drop in temperature in basaltic magmas with high silica content, or when these magmas interact with saline or alkaline solutions. As a result of these specific geological conditions, natural zeolite deposits are commonly found near active or inactive volcanoes (Schifter & Bosch, 1988). As for synthetic zeolites, these are obtained from non-zeolitic precursors, commonly through a process known as hydrothermal synthesis. This method uses materials with a high silica content as starting materials, such as aluminosilicates, volcanic glass, diatomite, ashes, glass or ceramic waste, among others. Finally, modified zeolites are natural zeolites whose surface or internal structure has been altered or modified to improve their properties and applications (Yuna, 2016).

Physicochemical properties of zeolites, such as their high specific surface area, pore size, thermal stability, and ion exchange capacity, in combination with their structural properties, explain their numerous applications in water treatment processes and the capture of atmospheric pollutants, as well as in catalysis in the petrochemical industry and for biodiesel production, to name a few (Rhodes, 2010).

An important aspect that has emerged in recent years is the possibility of using waste as raw material for new products, which represents a key strategy to reduce the environmental impact of waste and promote the circular economy (Malladi *et al*., 2024). As mentioned above, several studies have demonstrated that it is possible to synthesize zeolites from solid waste. A particular case is construction and demolition waste (CDW), which includes all waste generated by construction, renovation, and demolition activities of buildings, works, and structures; CDW includes concrete, ceramics, bricks, gypsum, and other mineral-based materials (Hasibuan *et al*., 2025). This waste, which has traditionally been considered an environmental problem due to its high volume of generation and limited reuse, has gained relevance as a potential source of aluminosilicates, essential compounds for zeolite synthesis.

For example, Wang and Chen (2017) developed a method to produce analcime-type zeolites from CDW using a combined sintering and alkaline hydrolysis process, obtaining a granular material with a multiscale porous structure and high heavy metal adsorption capacity. Similarly, Wang and Zhang (2013) developed a zeolite-loaded ceramsite from CDW, whose porous structure showed favorable properties for water treatment. More recently, Hernández-Palomares and Espejel-Ayala (2022) successfully synthesized precipitated silica, alkali silicates, and zeolites from thermally and chemically treated CDW, confirming their potential as precursors of high value-materials.

These precedents support the technical feasibility of recycling CDW as a raw material for zeolite production. However, challenges remain regarding the optimization of synthesis conditions, the characterization of the resulting phases, and their functional application in real-world environments. Therefore, the main objective of this work is the synthesis of zeolites using a hydrothermal process and CDW as the starting material.

Materials and Methods

CDW used in this study was provided by the Engineering and Architecture Department of the university. Representative brick samples were selected and subjected to a multi-stage pretreatment to obtain the starting material. First, the bricks were processed using a jaw crusher (Allis Mineral Systems, Oak Creek, USA), yielding fragments approximately 2–3 cm in diameter. Subsequently, a roller mill (Quinn Co., Whittier, USA) was used to reduce the size to particles smaller than 1 cm. The resulting material was homogenized using a sample divider (HGG-I, CGOLDENWALL, Germany) to ensure the representativeness of the final subsample. This subsample was then pulverized using a Bico E mill (Burbank, USA), and finally, an agate mill (Pulverisette 2, Fritsch, Germany) produced a fine powder. This powder was sieved with a No. 200 mesh to obtain particles smaller than 0.074 mm.

With the prepared starting material, hydrothermal synthesis was carried out. A laboratory-scale, high-pressure stainless-steel reactor (TGYF-C, 50 mL) was used. The reactor was loaded with 2.5 g of the starting material and 30 mL of a KOH 2 M solution. The reactor was maintained at 160°C for the following synthesis periods: 6, 8, 10, and 12 hours.

Once the process was complete, the reactor was allowed to cool to room temperature. The resulting material was washed with distilled water, centrifuged to separate the product, and then dried in an oven at 100°C for 16 hours. Finally, the dried product was crushed and sieved again with a No. 200 mesh. Product characterization was performed by powder X-ray diffraction (PXRD) on an Inel instrument (Equinox 2000, USA) with a cobalt source and an analysis time of 15 minutes per sample.

Results and Discussion

PXRD characterization was performed on the four samples corresponding to the products obtained after hydrothermal treatments of 6, 8, 10, and 12 hours. Figure 1 shows the characterization results, which reveal a progressive development of crystallinity as a function of the reaction time.

In the sample resulting from the 6-hour treatment, a diffraction pattern characterized by low counts in most peaks (< 250) was observed, indicating a predominantly amorphous phase. Despite the partial dissolution of the precursor (powdered brick), crystalline remnants were identified with signals located at 25.60°, 32.45°, 32.80°, and 35.45° (2θ), associated with silicon dioxide, as can be seen in Figure 2.

These remnant peaks indicate that, although the dissolution of the original brick structure has begun, the reorganization process toward new phases is not yet significant. This behavior is characteristic of the initial stage of hydrothermal synthesis, in which the precursor components begin to deconstruct, generating an amorphous matrix rich in silicates but lacking long-range order.

After 8 hours of treatment, a moderate improvement in the diffraction pattern was observed. New peaks appeared at 32.29°, 32.78°, 35.30°, 38.30°, and a distinctive peak at 41.30° (2θ). Although the intensities of these peaks remain relatively low (less than 500 counts), an increase in the structural organization of the material is evident, as shown in Figure 3.

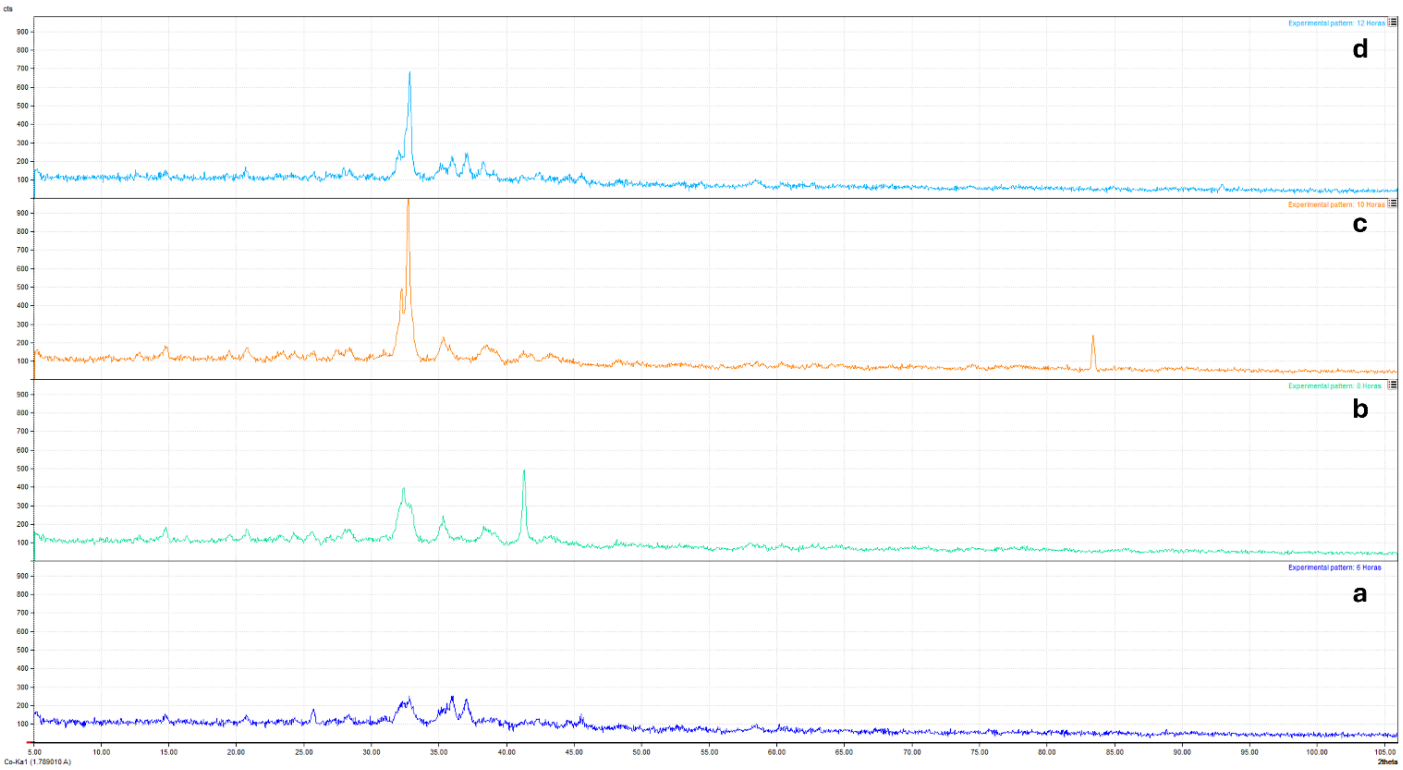

Figure 1. Diffraction patterns of the samples synthesized after (a) 6; (b) 8; (c) 10; and (d) 12 hours.

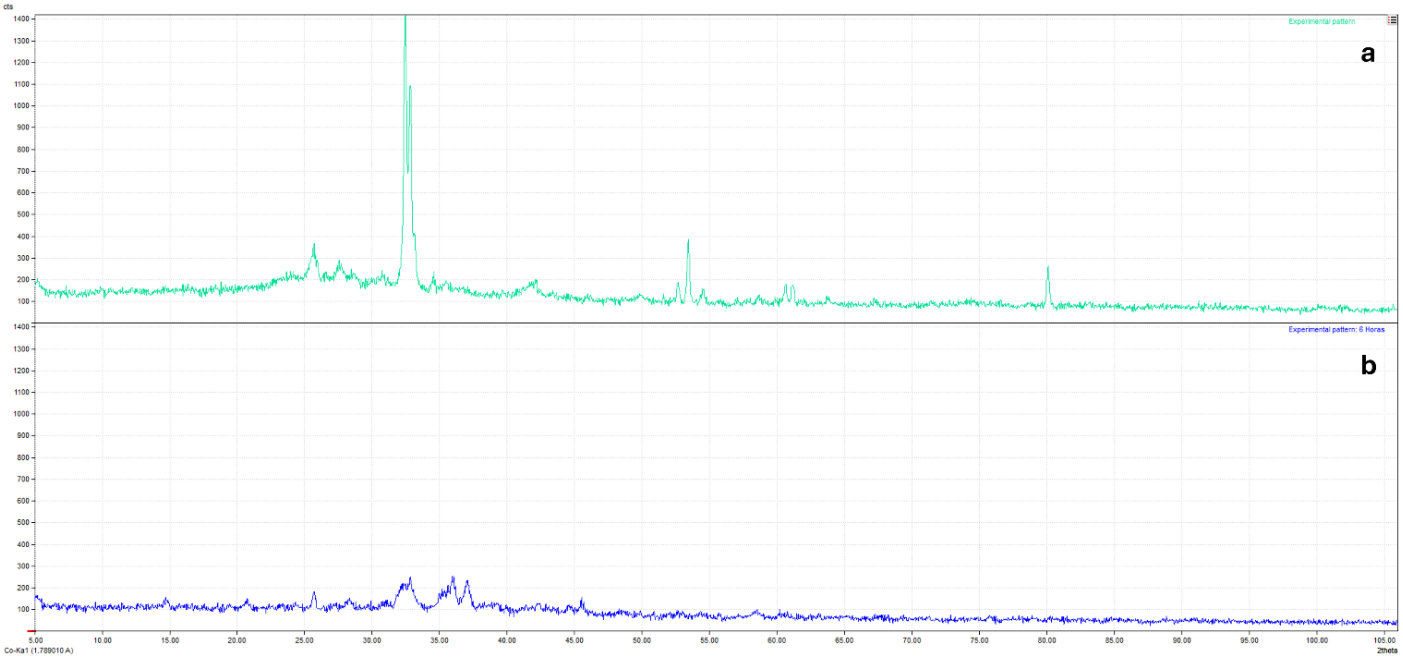

Figure 2. Diffractograms of (a) silicon dioxide and (b) the sample synthesized after 6 hours.

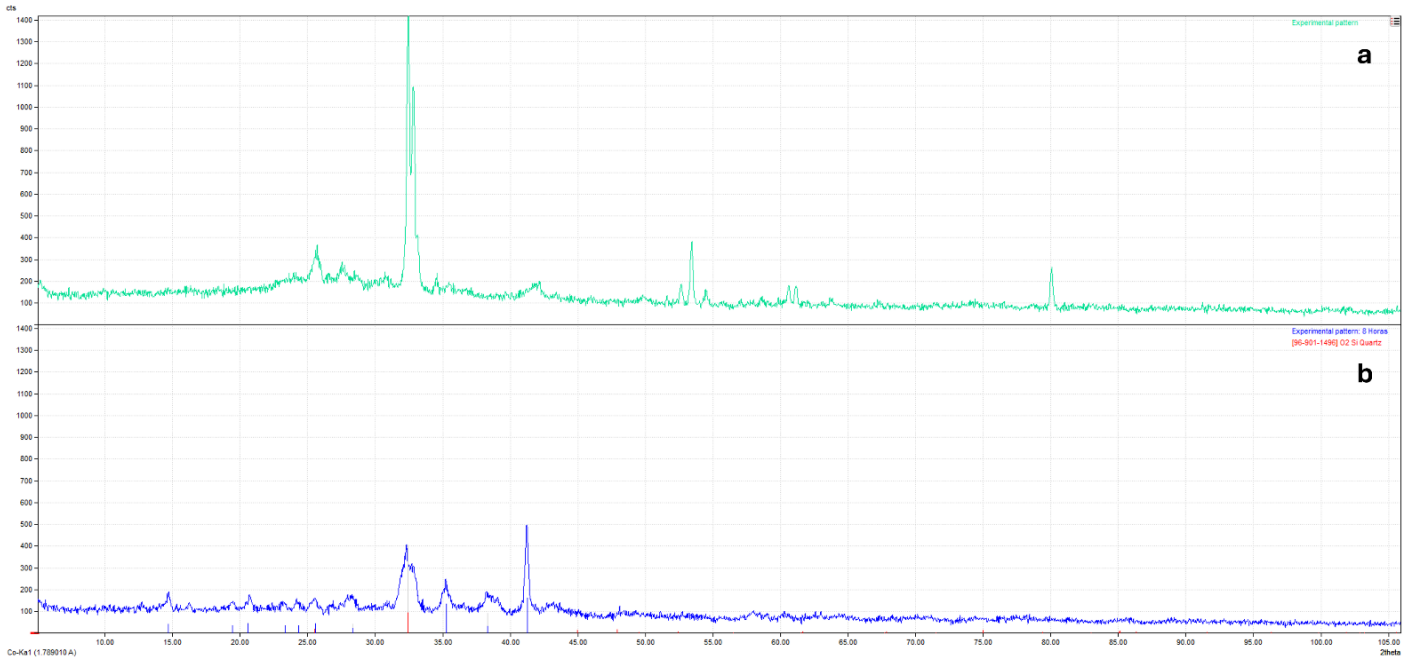

Figure 3. Diffractograms of (a) silicon dioxide and (b) the sample synthesized after 8 hours.

These new peaks suggest the onset of crystalline growth, likely associated with early stage zeolitic phases. Despite the difficulty in precisely identifying all signals due to their low intensity, the presence of silicon dioxide was again confirmed, along with indications of emerging phases that are still not well defined. This stage represents a transition between the initial disorganization and the beginning of the reconstruction of ordered structures typical of zeolites.

After 10 hours of treatment, the XRD pattern of the sample showed a notable change compared to the samples obtained at shorter synthesis times (Figure 4). The peaks became more defined, with higher intensity and a lower baseline, which indicates a considerable increase in the crystallinity of the product. Several phases were identified with greater confidence:

- Silicon dioxide (SiO_2) as the main phase
- A potassium-rich phase
- Mixed potassium–sodium aluminosilicate, compatible with zeolitic structures

The development of these phases reveals a significant transformation of the precursor into crystalline structures with characteristics of zeolitic materials. The identification of the potassium–sodium aluminosilicate is particularly relevant, since these compounds are typical in the synthesis of phillipsite-type zeolites, which are characterized by well-defined intracrystalline channels and high cation-exchange capacity (Pansini *et al.*, 1996). Furthermore, the increase in the intensity of the peak located at 32.78° (2θ) stands out as a predominant signal of the growth of a specific crystalline phase, possibly corresponding to the zeolitic phase of interest.

The sample treated for 12 hours showed an XRD pattern similar to that of the sample synthesized in 10 hours, although with slight modifications in the relative intensity of some peaks and the appearance of new signals at 35.98°, 37.08°, and 38.33° (2θ) (Figure 5). These signals may indicate the formation of secondary phases, possibly due to structural reorganization reactions or the depletion of key reactants in the hydrothermal medium.

These new phases were tentatively associated with compounds containing aluminum, potassium, and silicon. However, due to the complexity of the pattern and the possible overlap of peaks, complementary analysis using other techniques (for example, infrared spectroscopy or scanning electron microscopy) is required to confirm the composition and morphology of these phases.

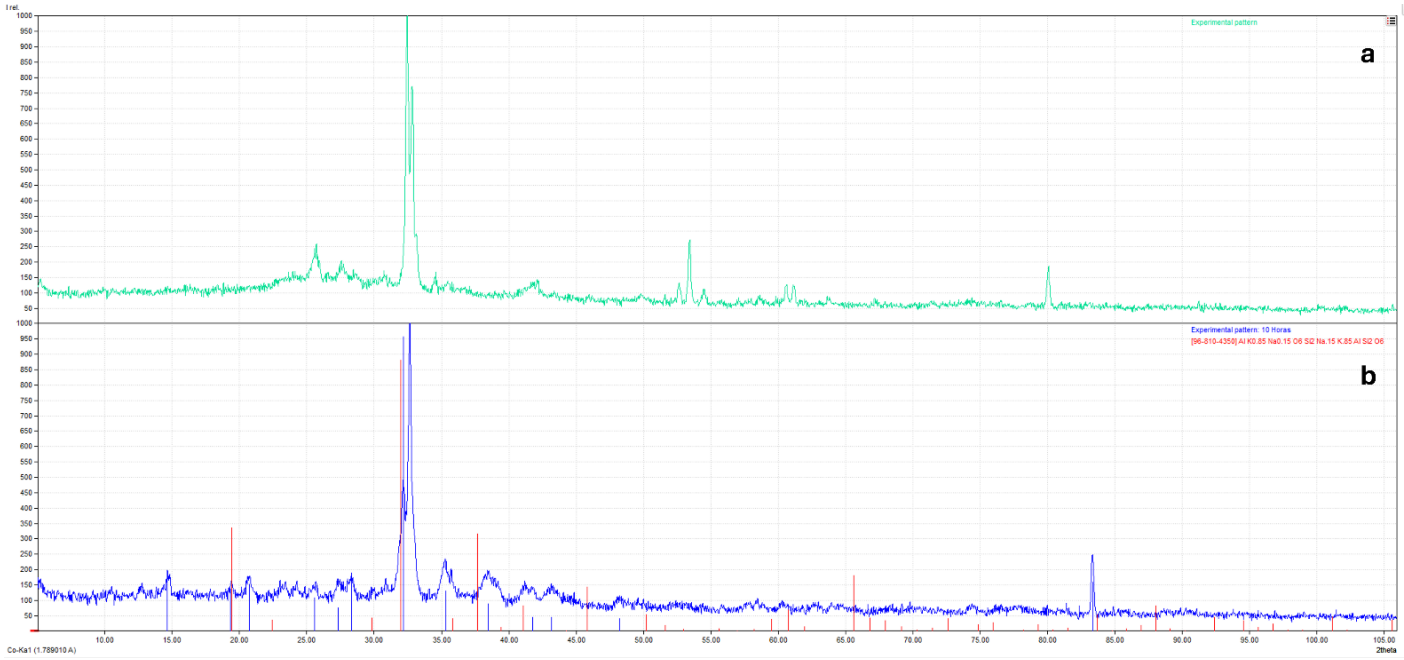

Figure 4. Diffractograms of (a) silicon dioxide and (b) the sample synthesized after 10 hours

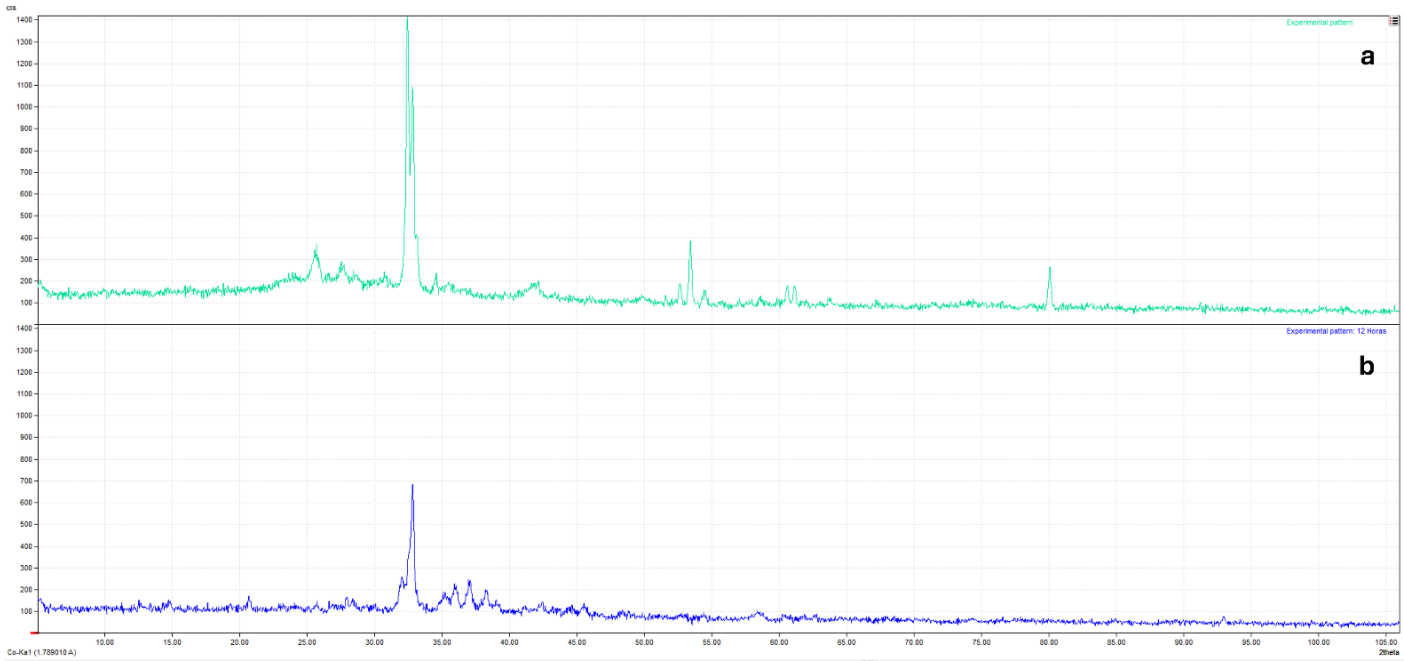

Figure 5. Diffractograms of (a) silicon dioxide and (b) the sample synthesized after 12 hours

It is worth noting that, although a good level of crystallinity is maintained in this sample, the slight decrease in the intensity of the peak at 32.78° suggests that undesired or less-ordered phases may be forming, because of recrystallization-related processes or the development of competing structures.

The mineralogical evolution observed throughout the synthesis process indicates a progressive transformation of the precursor material toward more ordered structures. The XRD analysis shows that reaction time is a critical factor in the development of zeolitic phases, as summarized in Figure 6.

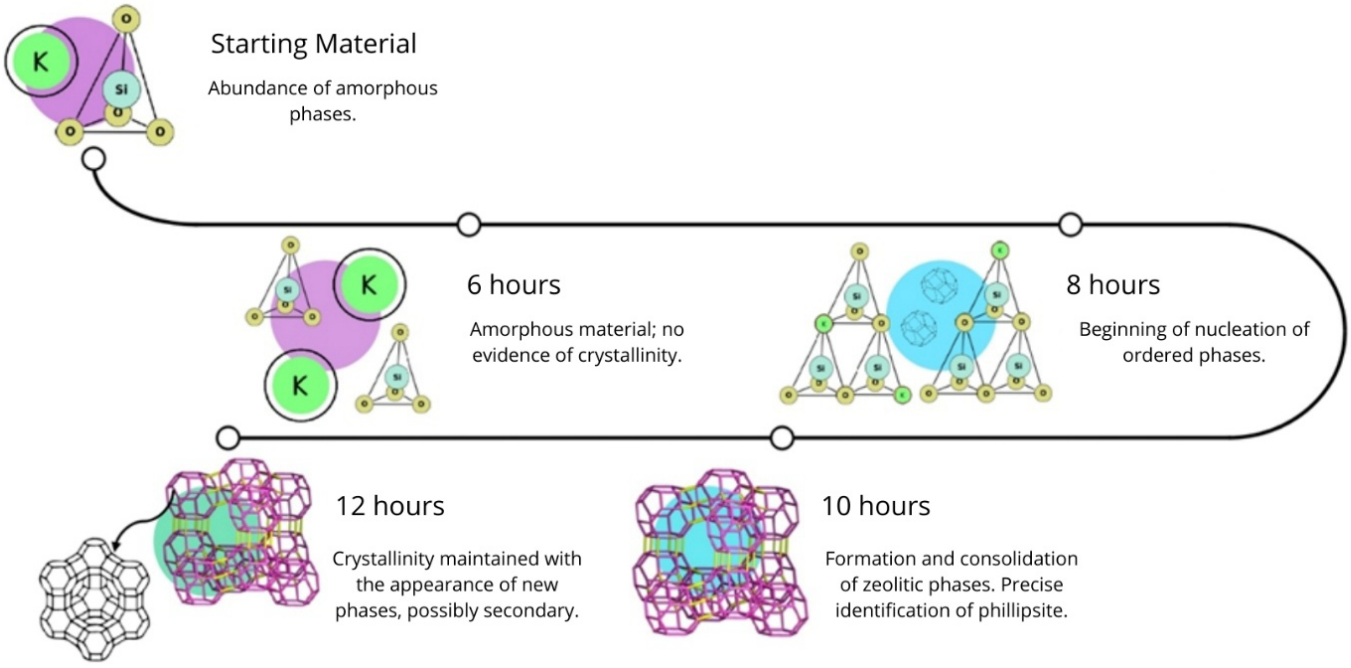

Figure 6. Synthesis of phillipsite by the hydrothermal method

These results support the feasibility of using construction and demolition waste (CDW) as a raw material for zeolite synthesis via hydrothermal processes, contributing to the valorization of this waste within the circular economy framework. However, they also highlight the need to optimize synthesis parameters to prevent the formation of secondary undesired phases over extended periods.

Conclusions

The present study demonstrates the technical feasibility of transforming construction and demolition waste, specifically bricks, into zeolites through a hydrothermal synthesis process. Through different treatment times (6, 8, 10, and 12 hours), a progressive evolution in the material's crystallinity was observed, and optimal formation of zeolitic phases was found after approximately 10 hours. This reaction period led to the identification of compounds characteristic of phillipsite-type structures in the product. These findings not only validate the use of construction and demolition waste as viable precursors for obtaining high value zeolitic materials but also align with the principles of the circular economy by converting an abundant solid waste into a functional resource. However, it was observed that excessive reaction times may induce the formation of undesired secondary phases, highlighting the need for careful optimization of process parameters. Overall, this work offers a sustainable strategy for waste management and the development of materials with applications in water treatment and catalysis, among other fields.

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