CONVERSION OF SOLID WASTE FROM ELECTRICITY COGENERATION INTO SLOW-RELEASE FERTILIZER

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ABSTRACT

Sugarcane bagasse from the sugar and ethanol industry is generated in large quantities in Brazil and is typically burned in plant boilers to produce electricity in the so-called cogeneration process. After combustion, the remaining solid waste, known as sugarcane bagasse ash (SCA), does not have many applications and is usually discarded around the industries. SCA can be converted into zeolites, which can be applied as support for the slow-release fertilizers, treatment of industrial effluents, carbon sequestration and capture, among others. Therefore, the present study aimed to obtain high-purity type A zeolite from SCA with NH_4^+ incorporated into its structure and evaluate its application in agriculture. An SCA sample generated in a power cogeneration plant located in São Paulo, Brazil, was used to synthesize zeolite type A (ZASC), which was characterized in terms of chemical composition (XRF), mineralogical composition (XRD), and morphology (SEM). Ammonium ion was incorporated into ZASC by ion exchange. The presence of NH_4^+ in the zeolite structure was confirmed by using the Fourier Transform Infrared Spectroscopy (FTIR) technique. The NH_4^+ modified zeolite was labelled as 'ZASCN1'. The amount of NH_4^+ released from ammonium-modified zeolite (ZASCN1) over time was evaluated through contact with water and saline solution in a static system and the concentration was measured by ion chromatography. Results showed that ZASCN1 can release a greater amount of nutrients in saline solutions when compared to water. In addition, both curves showed that the release of NH_4^+ occurred slowly and steadily for more than 1 month. Therefore, it can be concluded that the sugarcane industry has many possibilities for research, as well as the development and generation of products with market competitiveness, as the slow-release fertilizers, following the principles of the circular economy.

Keywords: circular economy, sugarcane industry, slow-release fertilizer.

1 INTRODUCTION

Partnerships between sugarcane processing industries and universities or research institutes can foster the development of technological research aimed at discovering new products and exploring new business opportunities based on the circular economy, with a focus on sustainability. In this context, in recent years, numerous studies have been focused on the reuse of by-products from sugar and ethanol production, such as the production of biodegradable plastic from polyhydroxy butyrate (PHB), extracted from sugarcane bagasse [1], [2], and the production of second-generation ethanol from sugarcane bagasse [3], among others.

Sugarcane bagasse is generated in large quantities in Brazil and is usually burned in plant boilers to produce electric power in the so-called cogeneration process. This energy source contributes significantly to the Brazilian electricity matrix and has been responsible for most of the country's biomass-burning energy generation in recent years. In 2021, biomass burning accounted for 7.9% of Brazil's electricity generation [4], [5].

According to the Sugarcane and Bioenergy Industry Union from Brazil [4], bioelectricity generated from sugarcane is the fourth most important energy source in the Brazilian electricity matrix. Fig. 1 shows an illustrative diagram of the production of ethanol and electricity by industries in this sector.

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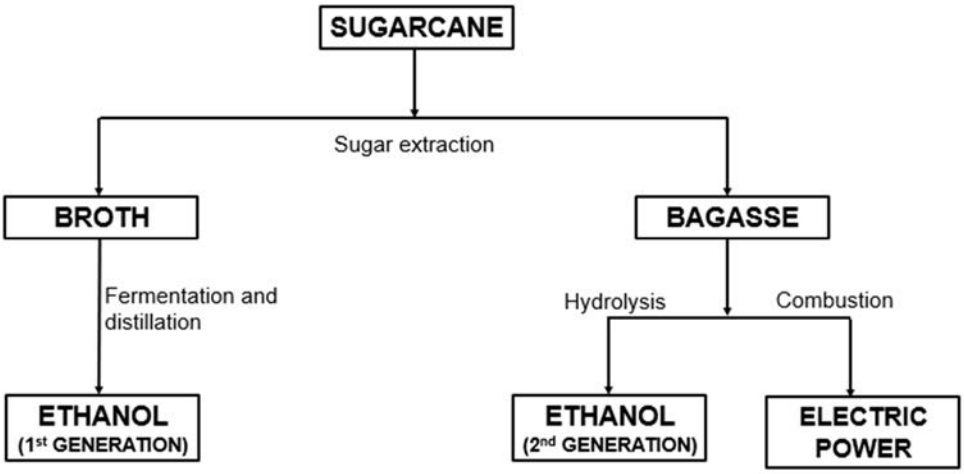


Figure 1: Illustrative diagram of ethanol and electricity production by Brazilian sugarcane processing industries. *(Source: Adapted from: https://encurtador.com.br/9c6oR.)*

The remaining solid waste from the bagasse combustion, known as sugarcane bagasse ash (SCA), does not have many applications and is usually discarded around the industries. In other cases, it is returned to the sugarcane seedlings as fertilizer. However, its excess can cause soil desertification in the long term. Therefore, new research aimed at converting sugarcane bagasse ash into higher value-added products with diverse applications in agriculture and industry is urgently needed.

The high silica content in SCA can allow it to be a substitute for sand in the manufacture of cement and concrete [6]. Fungaro and Izidoro [7] also proved that sugarcane ash can be converted into high-purity zeolites by applying a simple and low-cost methodology. Zeolites are materials with high porosity that have diverse applications such as support for the low release of fertilizers [8], [9], treatment of industrial effluents [10], carbon sequestration and capture [11], among others.

Zeolites have three main characteristics that make them useful in agricultural activities, such as cation exchange capacity, water retention capacity and ion retention capacity. Thus, they can be used as soil conditioners, in the decontamination of soils impacted by metals and radioactive isotopes, in the formulation of herbicides, insecticides and fungicides, in the preservation of fruits, grains, and seeds, in the production of organomineral fertilizers, and in the production of slow-release fertilizers [12].

The nutrient release can occur from the ions conditioned into the zeolite structure or by zeolite and commercial fertilizer blends. The zeolite’s high moisture retention capacity is also a positive factor for its application in water-deficient climates [13].

Slow-release fertilizers (SRF) are defined as those that release nutrients to plants more slowly than commercial fertilizers. The SRF can increase nutrient efficiency used by the plant by reducing losses through leaching, volatilization, denitrification, and fixation, and thus increase plant absorption through gradual supply, according to crop demand [14].

The demand for potassium fertilizers in Brazil is very high, coming almost entirely from sources outside the country, and is still expected to grow. Studies have shown that potassium zeolites obtained from waste have great potential for use in agriculture [15]–[19].

Recycling solid waste generated in large quantities by agro-industrial production sectors, such as sugar and alcohol plants, enables its return to agriculture and reduces environmental impact, becoming a component of the circular economy. In this approach, agricultural waste management will contribute significantly to achieving sustainable development goal (SDG) 12, specifically item 12.5: ‘By 2030, substantially reduce waste generation through prevention, reduction, recycling and reuse’, and SDG 2, related to sustainable agriculture [20].

Thus, the present study aimed to obtain high-purity type A zeolite with nutrient incorporated into its structure, from the residue remaining from the burning of sugarcane bagasse, to be applied in agriculture.

2 MATERIALS AND METHODS

2.1 Sugarcane ash sample

This study used the sugarcane ash sample generated in a power cogeneration plant located in the city of Franca (São Paulo, Brazil). The ash was initially dried in the sun (in the industrial area) and then went through the steps of aeration (to remove the lighter particles, mainly containing unburned carbon), washing with water, drying in the sun, and subsequent removal of iron (by dry magnetic separators) (Fig. 2).

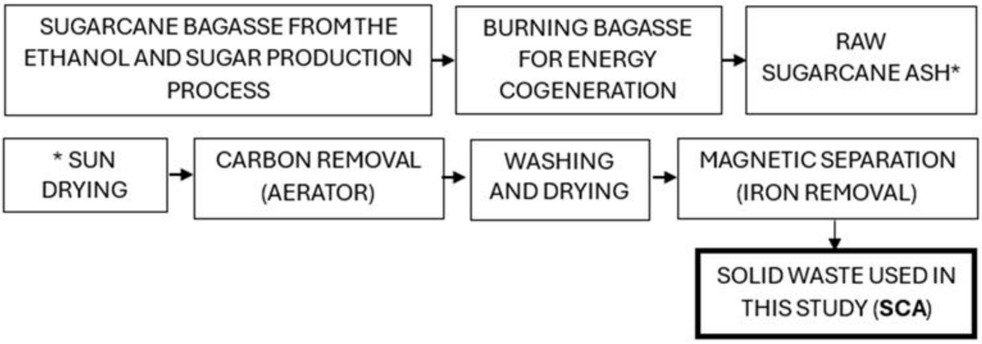


Figure 2: Diagram of the preparation of solid waste from the burning of sugarcane bagasse as raw material in this study.

Approximately 5 kg of dry product underwent a magnetic separation step to remove iron. Low-field separator with 1,500 Gauss magnetic field, medium-field separator with 7,500 Gauss magnetic field and high-field separator with 21,000 Gauss magnetic field were used, respectively. The high-field separation was performed in three stages, each occurring at speeds of 200, 150, and 100 rpm, respectively. The sugarcane ash sample without iron was labelled as ‘SCA’.

2.2 Synthesis of type A zeolite

The synthesis methodology of zeolite ‘A’ from sugarcane ash was based on Izidoro et al. [21]. However, new synthesis parameters were adjusted to optimize the product, since the precursor residue used differed. Several synthesis methodologies were tested over months, varying the fusion time, the residue/NaOH ratio, time and stirring mode (with magnetic and/or mechanical stirrer), and different crystallization times, in parallel with the observation and interpretation of X-ray diffractograms of the products, to determine the optimal synthesis conditions suitable for the synthesis of zeolite type A from sugarcane ash.

The prepared sugarcane ash without iron was mixed with grounded NaOH and subjected to a fusion step. After cooling to room temperature, water and aluminium were added to the mixture to adjust the SiO_2/Al_2O_3 molar ratio, which was suitable for synthesizing type ‘A’ zeolite. The suspension was then stirred, and hydrothermal treatment was performed for 3 h. After cooling at room temperature, the suspension was filtered, and the solid was washed until the pH of the wash water reached 9.0. The solid material was then dried in an oven at 105°C overnight and sent for characterization. Zeolite A from sugarcane ash was labelled as ‘ZASC’.

2.3 Nitrogen incorporation into zeolite

The dried type A zeolite ZASC was submitted to an ion exchange process with NH_4Cl salt at a concentration of 1.5 mol L^{-1}. In this nutrient incorporation step, ammonium salt and zeolite were placed in a beaker under stirring for 40 min. Then, the solid was washed with distilled water to remove excess reagent and dried at 30°C for 24 h. Confirmation of the presence of the macronutrient (NH_4^+) in the zeolite structure was performed using the Fourier Transform Infrared Spectroscopy (FTIR) technique, described later. The product was stored in the refrigerator and labelled as ‘ZASCN1’.

2.4 Characterization of starting material and products

All samples (SCA, ZASC and ZASCN1) were characterized in terms of chemical composition and mineralogical composition, determined by X-ray fluorescence (XRF) (Malvern Panalytical Spectrometer – Zetium) and by X-ray diffraction (XRD) Rigaku – Miniflex II, with a monochromator and Cu Kα radiation at 30 kV and 15 mA), respectively. The scan rate of XRD used to verify the crystalline phases was 0.05°/s and ranged from 5–90° (2θ). To verify the morphology, each sample was examined using a high-resolution scanning electron microscope (SEM) JEOL, model JSM-6701F.

The presence of ammonium ions in the zeolite structure was confirmed by FTIR (Thermo Nicolet, model Nexus 870FTIR). Samples were prepared in KBr (1% m/m sample) and previously dried at 105 ºC. The evaluation range on the equipment was from 400 to 4000 cm^{-1}.

2.5 Study of ammonium ion release by zeolite

The amount of available ammonium ions was evaluated by the chemical extractor method, where a liquid extractor, such as water or a saline solution, was placed in contact with the fertilizer for a specified period. Afterward, the amount of NH_4^+ present in the extract solution was determined. The extractor resembles the ability to plant roots to absorb the nutrients available in the soil.

Samples of 0.1 g of the ZASCN1 were placed in contact with 50 mL of deionized water or 50 mL of a simulated soil solution (prepared from a mixture of 5.0 mmol L^{-1} of $CaCl_2$, 1.0 mmol L^{-1} of $MgCl_2$, and 0.25 mmol L^{-1} of KCl) [22]. In the first test, samples were left to rest for 3, 6, 12, 24, 36, 180, 360, 540, 720, and 900 h. After each rest period, the samples were filtered, and the NH_4^+ ion concentration in the solutions was determined in triplicate by ion chromatography (ICS-2100, Thermo Fischer Scientific, USA). The experimental conditions employed a 4 × 250 mm IONPAC CS12A column with a flow rate of 1 mL min^{-1}

of 22 mM H_2SO_4 eluent. The determination of pH and conductivity of each sample were measured using a pH meter (Gehaka, model PG2000).

3 RESULTS AND DISCUSSION

3.1 Comparison of the visual aspect of the samples

The visual aspect of the sugarcane ash, the zeolite A synthesized from the residue, and the sample with ammonium incorporated is shown in Fig. 3. Results show that the ash sample (Fig. 3(a)) has a dark grey coloration, while the zeolite synthesized from it (ZASC-b) is white. This change is due to the purification of the ash, where contaminants in smaller quantities, such as iron, were removed in the washing stage of the synthesis. In addition, white-coloured reagents, such as NaOH and $NaAlO_2$, were also used in the synthesis of the product, in addition to the occurrence of a change in the crystalline structure (shown later by XRD and SEM techniques). The sample with incorporated ammonium ion (ZASCN1-c) has a slightly yellowish colour compared to the ZASC sample, possibly due to the higher moisture content in this sample, since it was dried at 30°C (the ZASC sample had been dried at 105°C).

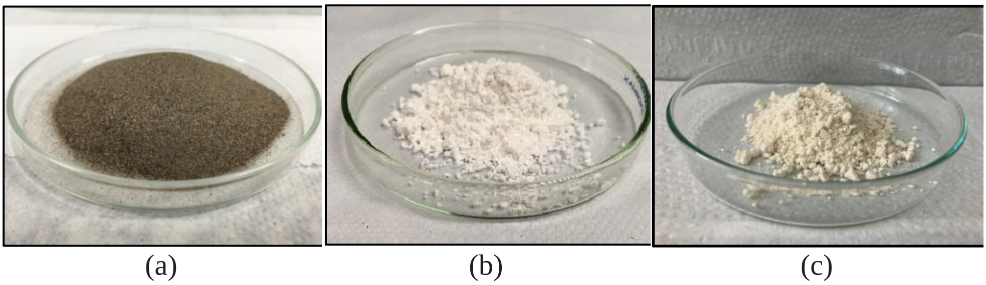


Figure 3: (a) Sugarcane ash sample from ethanol industry (SCA); (b) Sample of zeolite A synthesized from the sugarcane ash (ZASC); and (c) Sample of zeolite A with incorporated nitrogen (ZASCN1).

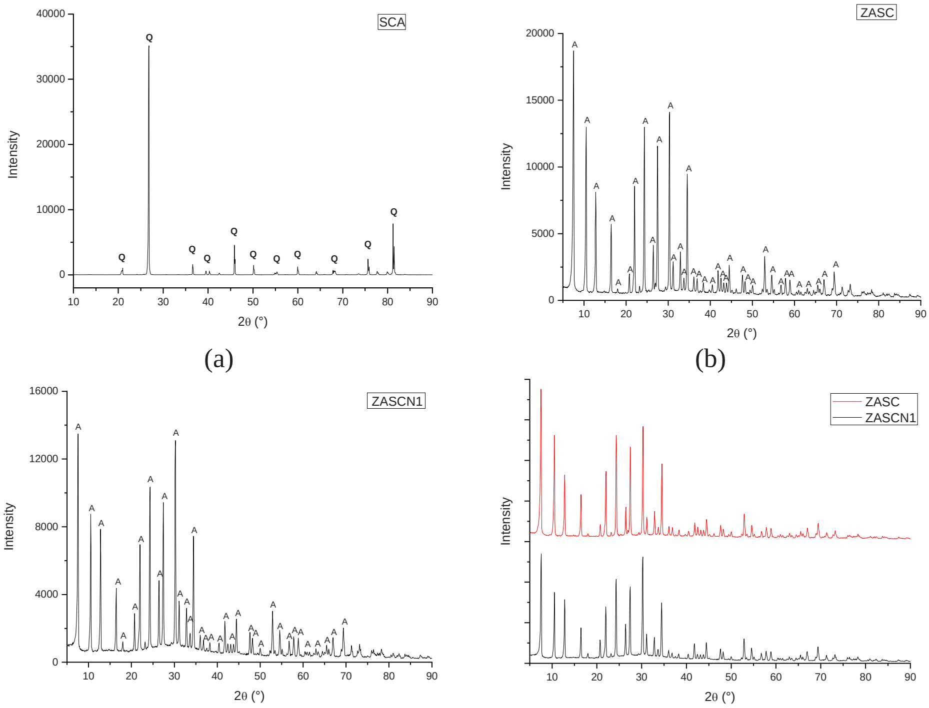
3.2 Chemical composition and crystalline phases

The semiquantitative chemical composition of sugarcane ash (SCA), zeolite type A from sugarcane (ZASC), and nitrogen-incorporated zeolite A (ZASCN1), determined by XRF, is shown in Table 1 (by % weight). According to the results, the SCA sample presented a high silicon content (98.3%), which enables it to be used for the synthesis of zeolite, through the addition of aluminium from an external source [7].

Raw material common impurities such as titanium, chromium, manganese, copper, and zinc were included in ‘others’ and accounted for 0.23%. A part of them was removed during synthesis, along with some iron. For the ZASC and ZASCN1 products, the content of Si and Al, which are the structural elements for zeolites, represented together at least 50% for both samples, while sodium content, which is the exchangeable cation of the zeolitic structures, was incorporated. These results are similar to the other study that synthesized zeolite A, but using another solid waste [11]. Synthesized zeolites also presented a loss of ignition (LOI) of 17.8% and 20.8% for ZASC and ZASCN1 samples, respectively. LOI values can indicate the difference in the dry temperature for both samples. The ZASCN1 sample was dried at

Table 1: Chemical composition (wt%) of sugarcane ash (SCA), zeolite type A synthesized from sugarcane (ZASC), and zeolite A modified with nitrogen (ZASCN1) determined by X-ray fluorescence technique (XRF).

Component	SCA	ZASC	ZASCN1
SiO_2	98.3	34.0	36.3
Al_2O_3	0.41	29.9	31.1
Na_2O	–	18.1	11.4
Fe_2O_3	0.86	0.10	0.13
CaO	0.06	0.03	0.05
K_2O	0.03	0.02	0.02
SO_3	–	0.01	0.01
Cl	<0.01	<0.01	0.10
Others	0.23	0.03	0.09
LOI	<0.10	17.8	20.8



(a) (b)

(c) (d)

Figure 4: XRD patterns of (a) Sugarcane ash from sugar and ethanol industry (SCA); (b) Zeolite type A synthesized from sugarcane ash (ZASC); (c) zeolite type A synthesized modified with nitrogen (ZASCN1); and (d) Comparison of XRD Patterns of ZASC and ZASCN1 samples. Q = Quartz – SiO_2; A = Zeolite A – $Na_2Al_2Si_{1.85}.O_{7.75}.1H_2O$).

30°C while the ZASC was dried at 105°C. Sample with incorporated ammonium ion needs to be dried at a low temperature because ammonium can be converted into ammonia gas; therefore, the type of material is usually dried in air [23].

The X-ray diffractograms of the SCA, ZASC, ZASCN1 samples, as well as a comparison between ZASC and ZASCN1 are shown in Fig. 4. Results show that sugarcane bagasse ash mainly consists of quartz (SiO_2). The presence of this crystalline structure in the ash is usually related to the temperature reached by the reactor during bagasse combustion. The higher the combustion temperature, the less amorphous silica remains in the residue. Therefore, the crystalline profile of quartz suggests that the bagasse was likely burned at high temperatures (around 1000°C). Also, the results demonstrate that the ZASC and ZASCN1 samples are very similar and primarily composed of the zeolite A phase, with specific peaks corresponding to zeolite A standard (JCPDS No. 38-241). The zeolites' X-ray diffraction patterns are consistent with previous studies that synthesized high-purity A zeolites using different sources [21], [24], [25], and sugarcane bagasse ash [26]. The comparison shown in Fig. 4(d) indicates that the NH_4^+ ion incorporation method did not affect the crystalline phases formed. The SEM results presented later confirm these XRD findings.

3.3 Morphology of raw material and zeolites

The micrographs of SCA, ZASC, ZASCN1 samples, are shown in Fig. 5. Scanning electron microscopy (SEM) was used as a complementary technique to verify differences between raw materials and products, as well as to confirm the presence of the crystalline phases identified by X-ray diffractometry. The SCA sample (Fig. 5(a)) showed inconsistent particle shapes. In contrast, ZASC and ZASCN1 samples consist of cubic-shaped structures that align with the form described by other researchers (Fig. 5(b) and 5(c)) [11], [21], [24], [25].

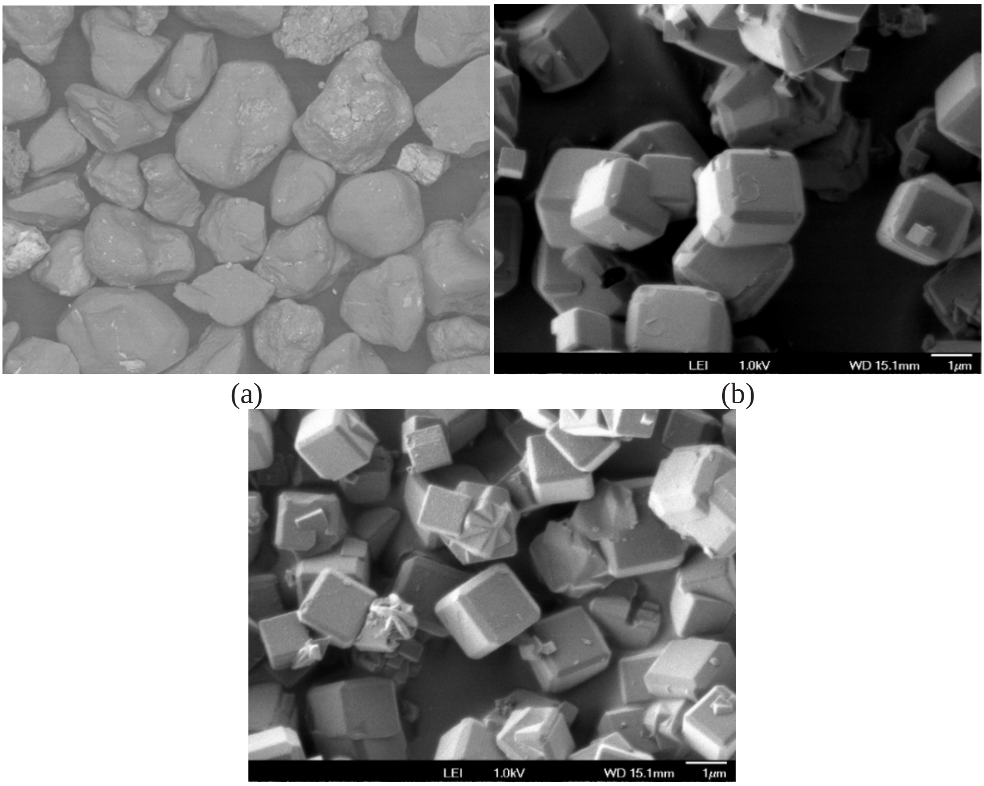
Fig. 6 shows the micrographs of ZASC and ZASCN1 samples with the same magnification to show the similarity between them. Results indicated that the NH_4^+ ion incorporation methodology did not influence the cubic-shaped structure, with the edges of the cubes measuring 1.751 µm and 1.700 µm, for samples ZASC and ZASCN1, respectively.

3.4 Confirmation of the presence of NH_4^+ in the zeolite structure by FTIR

The presence of the ammonium ion in the zeolite structure was confirmed using the FTIR technique in the range of 400 to 4000 cm^{-1} (Fig. 7). The result shows that FTIR spectra of the synthesized zeolites presented the typical absorption peaks of the commercial zeolite 4A [27]. The band around 1400 cm^{-1}, highlighted in the figure, indicates the presence of NH_4^+ in the structure of the modified sample (ZASCN1), while it is absent in the unmodified sample (ZASC). These results are following other studies with have incorporated ammonium ions in zeolite structure [17], [23].

3.5 Evaluation of ammonium ion release by synthetic zeolite

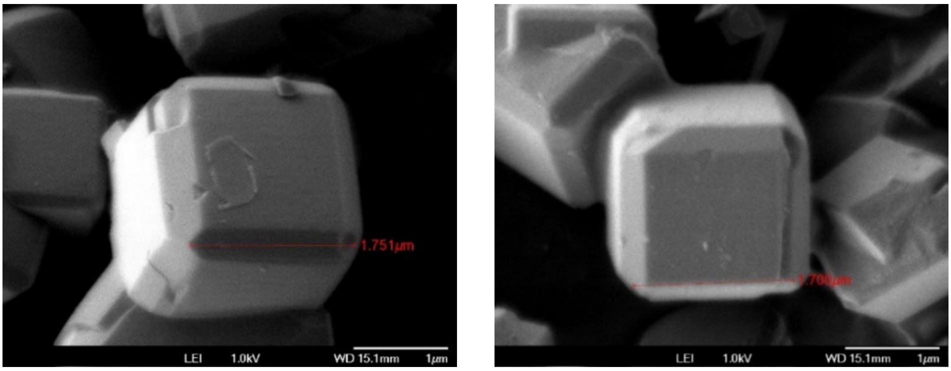
The monitoring of the release of ammonium ions from the ZASCN1 zeolite, after contact with water and saline solution, in a static system is shown in Fig. 8(a). Ammonium capture occurred spontaneously through pore diffusion and ion exchange processes. The release of NH_4^+ and Cl^- ions into water occurs only by dissolution (hydration together with pore diffusion). The release of NH_4^+ in saline solution occurs by dissolution and ion exchange with the ions contained in the saline solution [28]. Results showed that the slow-release fertilizer can release a greater amount of nutrient in saline solution when compared to water,



(a) (b)

(c)

Figure 5: SEM micrographs of (a) Sugarcane ash (SCA) – 200× magnification; (b) High-purity zeolite A (ZASC) – 10000× magnification; and (c) Zeolite A with nitrogen incorporated into the structure (ZASCN1) – 10000× magnification.



(a) (b)

Figure 6: SEM micrographs of (a) Zeolite A – ZASC; and (b) Zeolite A modified with NH_4^+ – ZASCN1, with 25000× magnification to show the similar size of both cubic structures.

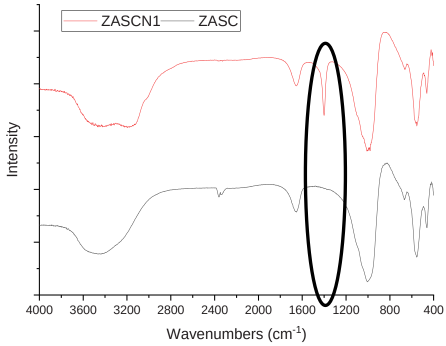
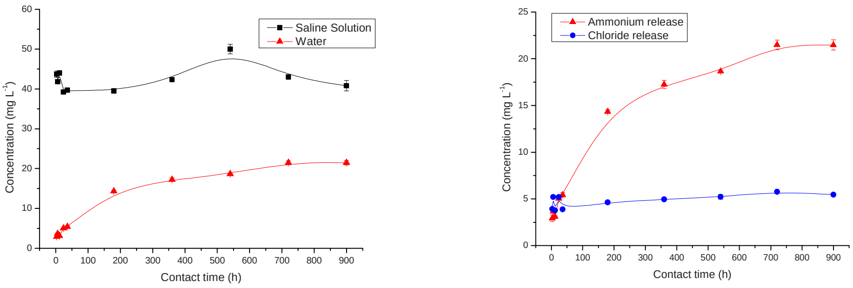


Figure 7: Comparative FTIR spectra of unmodified (ZASC) and modified (ZASCN1) zeolite A.



(a)

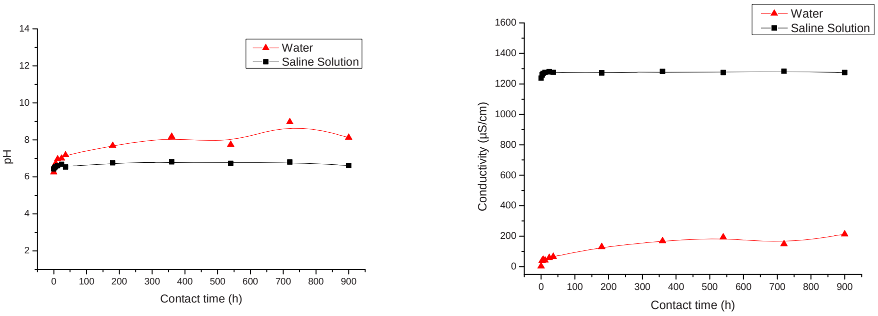
(b)

Figure 8: (a) Time-dependent release of NH_4^+ from ZASCN1 after contact with water and saline solution, in a static system; and (b) Release of NH_4^+ and Cl^- ions by ZASCN1 in water as a function of time.

with values ranging from 39.2 mg L^{-1} to 50.0 mg L^{-1} after 24 h and 540 h of contact, respectively, while the maximum amount of ammonium released by the water occurred after the maximum contact time (900 h), reaching a value of 21.5 mg L^{-1}. In addition, both curves showed that the release of NH_4^+ occurred slowly and steadily for more than a month. A similar ammonium release behaviour using zeolite A synthesized from coal fly ash, was observed by Inácio [17]. A comparison of the release of ammonium and chloride ions by ZASCN1 in water is shown in Fig. 8(b). This comparative study was not performed with saline solution because it is a mixture of chloride salts, which would interfere with the result.

As can be seen in Fig. 8(b), the amount of NH_4^+ released by ZASCN1 is greater than the amount of Cl^- and reached a maximum value of 21.5 mg L^{-1}, while the release of Cl^- varied from 3.80 mg L^{-1} to 5.80 mg L^{-1}, with an average release value of 4.72 mg L^{-1} over time. The difference in behaviour is associated with the residual NaOH found in the final zeolite product. In the release of NH_4^+, dissolution and ion exchange with Na^+ occur, while in relation to the release of Cl^-, hydration is inhibited. It is believed that the complete removal of the chloride ion from zeolite A can be done through a greater number of washes before the material is dried, which would not prevent its application as a slow-release fertilizer later.

The pH and conductivity for each sample after contact with water and saline solution over time, in a static system, are shown in Fig. 9(a) and 9(b), respectively. Results showed that the pH of the water samples ranged from 6.25 to 8.97, while for the saline solution, it ranged from 6.42 to 6.81, although the pHs remained constant over time. It is believed that the pH of the zeolite sample in water was slightly higher than that of the saline solution due to the remnants of OH$^-$ in the zeolite after synthesis with NaOH, which was not completely removed in the washing step. This effect was smaller for the saline solution, since the pH of the deionized water used in the preparation of this solution was around 5.0 and may have neutralized the supernatant.



(a) (b)

Figure 9: (a) pH measurements of the supernatant after contact of ZASCN1 with water and saline solution over time; and (b) Conductivity measurements of the supernatant after contact of ZASCN1 sample with water and saline solution over time.

Fig. 9(b) shows that the conductivities of the supernatants after contact of ZASCN1 with saline solution were higher than water. In both cases, the values increased slightly over time. The average value for the water samples was 100.64 µs/cm, while for the saline solution, it was 1270.73 µs/cm. This result was expected since the saline solution is composed of the salts $CaCl_2$, $MgCl_2$, and KCl, which provide a greater quantity of ions in solution, and consequently, give greater conductivity to the samples.

4 CONCLUSION

Sugarcane ash, produced in large amounts by the sugar and ethanol Brazilian industry, was applied as raw material for the synthesis of zeolite. High-purity Na-A zeolites (ZASC) structures were confirmed by XRD and SEM techniques. ZASC was subsequently modified with NH_4Cl and results indicated that the NH_4^+ ion incorporation did not affect the crystalline phase formed and morphology. The release of ammonium from zeolite was observed in water and compared to a saline solution. The ammonium release curves exhibit a pattern characteristic of slow-release fertilizers. Therefore, modified zeolite A from sugarcane waste ash has broad potential application as a nitrogen fertilizer for soil amendment. However, complete chloride removal during ammonium release is essential to prevent environmental damage.

It is worth highlighting that Brazil is a large territorial area country, which means that transportation costs, both for raw materials and products, are usually high because most of the industrial products are transported by roads (not by rail). Therefore, finding a way to use sugarcane industry solid waste at the same place where the ash is generated can reduce the costs of products, which must be competitive in the market, and can be applied on a large scale. Given the above, it can be concluded that the sugarcane industry has many possibilities for research, as well as the development and generation of products with market competitiveness, as slow-release fertilizers.

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