



Article

The influence of binders for the pelletization of fly ash zeolites on sulfur dioxide sorption properties

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Abstract

Fly ash zeolites are economically and ecologically attractive alternatives to synthetic and natural zeolites. Their use as sulfur dioxide sorbents is one of the possible applications of these materials. During the process of fly ash zeolite synthesis, a light powder is formed, which is not acceptable in practical applications due to technical problems, such as a marked drop in pressure, diffusion limits, hydraulic resistance, clogging in the packed beds and the possibility of losing a bed. It is therefore necessary to perform a pelletization process. Thickening of the material during pelletization influences sorption capacity negatively due to diffusing limitations, while the lack of an additional binder may result in a material of low mechanical durability. In this study, pressure pelletization experiments with fly ash zeolite were performed. Binders were selected on the basis of economic considerations as well as their potential to exert a positive influence on the sorption properties of the produced pellets. Cyclic sorption experiments were conducted (on sulfur dioxide) in which one zeolite powder sample was subjected to pelletization without a binder and another sample was subjected to the process with selected binders added. The results of the experiments were then analysed to ascertain the influence of the pelletization process on sulfur dioxide sorption capacity.

Keywords: fly ash, pelletization, sulfur dioxide, zeolite

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Fly ash zeolites can be used as effective sorbents in multiple processes (Bandura *et al.*, 2015; Ściubidło & Majchrzak-Kucęba, 2019; Zhao *et al.*, 2019). Their use on a larger scale, however, remains limited, as there are no regulations that would encourage industry to invest in solutions to the problem of dealing with this kind of waste. The disadvantages associated with the transformation of fly ash into zeolites include the energy penalty, the creation of alkaline aqueous waste and the relatively low proportion of fly ash that is converted into zeolites (Längauer & Cablík, 2018).

The early study of Breck (1973) shed light on the ability of zeolites to capture sulfur dioxide (SO₂). Later work highlighted the economic potential of fly ash zeolites' capacity for SO₂ sorption (Srinivasan & Grutzeck, 1999). Zeolites synthesized from fly ash can be used as sorbents for SO₂ (Srinivasan & Grutzeck, 1999; Suchecki *et al.*, 2004; Czuma *et al.*, 2016a, 2019; Strossi Pedrolo *et al.*, 2017; Gorai, 2018).

On completion of the synthesis process, fly ash zeolite is in the form of a light powder (Czuma *et al.*, 2016b; Król & Mikula, 2017). On a scale larger than that of a laboratory, the use of material in this form may present significant difficulties. Application of powders causes a marked drop in pressure, diffusion limits, hydraulic resistance, clogging in the packed beds and the possibility of losing a bed due to powder particles being

readily transported by gases. It is therefore necessary to test the zeolite material's pelletization capability (Franus *et al.*, 2015). The industrial-scale process of particle enlargement using an agglomeration technique is one of the most significant operations in the transformation of fine powders into free-flowing, dust-free pellets. This is generally classified as a wet or dry pelletization process (*e.g.* steam pelletization, freeze pelletization, foam pelletization or pneumatic dry pelletization) (Shanmugam, 2015).

Application of a binder in the pelletization process not only affects the mechanical durability of the pellet, but also the sorption capacity of the pelletized material. In the case of an improperly chosen material, the adsorbent pores may be blocked, which will negatively affect its sorption capacity. Inclusion of a binder in the sorption process will have a positive effect on the performance of the pelletized material.

There is relatively little information in the literature on zeolite pelletization methods (dry or wet, pressure or pressure-free and with or without a binder). The most popular materials used for pelletization are bentonite (Li *et al.*, 2001; Rongsayamanont & Sopajaree, 2007), kaolinite (Li *et al.*, 2001), other clays (Brandt *et al.*, 2007; Müller *et al.*, 2015a, 2015b), water glass (Brandt *et al.*, 2007) and organic binders (Kulprathipanja, 2010). However, the literature does not provide a sufficient comparison of individual additives to the pelletization process and their effects on the adsorption capacity of the individual adsorbate (Wdowin *et al.*, 2015). It would therefore be useful to conduct a comparative study of the effect on absorption capacity (relative to the selected sorbate) of adding zeolite fly ash binder to the pelletization process.

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In the present study, a simple and low-cost method of pelletizing the fly ash zeolite material was carried out using elevated pressure, with and without the addition of a binder to improve mechanical durability. The binder's influence on the sorption capacity of the pellets, the nature of the sorption process and the pellets' susceptibility to regeneration in relation to SO₂ were then studied. The data presented could be valuable as the basis for further research, as such a comparison has not been presented previously.

Experimental

Materials

The fly ash used to synthesize zeolite was obtained from a Polish heat power plant (hard-coal combustion, pulverized bed). The chemical composition of the fly ash and zeolite was investigated with X-ray fluorescence (XRF) using an Epsilon 3 PANalytical XRF spectrometer and is presented in Table 1.

Zeolite was synthesized from fly ash using a hydrothermal method with 3 mol dm⁻³ NaOH. The synthesis was performed at ~90°C for 24 h (Macuda & Klima, 2017). The mineral composition of fly ash and fly ash zeolite was determined by X-ray powder diffraction (XRD) using a Philips X'pert avalanche photodiode diffractometer with a PW 3020 goniometer, a Cu tube and a graphite monochromator. Analysis was performed within the range 5–65°2θ.

The XRD study showed the presence of Na-P1 zeolite (Fig. 1) in the synthesized material, which can be used as a sorbent for SO₂ (Srinivasan & Grutzeck, 1999), and a reduction in the intensity of the quartz and mullite peaks, as well as a slight decrease of the amorphous phase.

Chemical composition data on the fly ash and synthesized fly ash zeolite showed a decrease in the levels of most components (due to their transfer to the solution during synthesis). The concentration of Na₂O increased significantly as the synthesis was carried out in the presence of sodium hydroxide.

The morphology of the synthesized zeolite was studied by scanning electron microscopy (SEM) using an FEI Quanta 250 field-emission gun (Fig. 2a).

Visual evidence of the presence of Na-P1-type zeolites is indicated by the characteristic needle-like shapes (Fig. 2a); the spherical particles that can be observed belong to unreacted fly ash remains. The rough surface is due to surface dissolution in alkaline media during zeolite synthesis. The composition of point 1 in Fig. 2a (based on energy-dispersive spectroscopy analysis) (Fig. 2b) is consistent with the possible presence of Na-P1 zeolite of the form Al₂O₃:xSiO₂:14Na₂O:840H₂O (Sharma *et al.*, 2016).

Pelletization

Due to the powdery form of the material obtained after synthesis, the investigated material should undergo a pelletization process. In this work, pressure pelletization was performed using a hydraulic press due to the economic advantages of the method. The choice of the appropriate loading and pressing time for the powders was based on preliminary tests designed to determine the optimum pressure and time, which were finally set at 10 tons and 2 min, respectively. Initially, the zeolite powder was subjected to a pelletization process without adding a binder (sample G). However, the resulting pellets were delaminated (Fig. 3a).

A binder additive was proposed to enhance the stability and durability of the pellets obtained (Fig. 3b). Given the need to

Table 1. Oxide composition (wt.%) of fly ash and fly ash zeolite.

Component (wt.%)	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	TiO ₂	K ₂ O	ZnO	Na ₂ O
Fly ash	54.8	3.9	23.5	7.7	1.9	1.4	4.0	1.4	0.7
Fly ash zeolite	42.2	4.0	28.6	8.7	2.6	1.5	0.9	1.5	9.4

investigate the effect of the binder additive type on sorption capacity, various binders were tested (selected on the basis of their potential to improve the viscosity of the pelletized material). The most desirable outcome would be to obtain non-delaminated pellets that would have a positive or neutral influence on sorption capacity. The selected binders are presented in Table 2.

SO₂ sorption

The SO₂ sorption experiments were performed at 25°C using an installation equipped with a Sartorius sorption microbalance with a sensitivity of 0.00005 g and a precision of 0.0002 g (Fig. 4).

The experiment was focused on measuring the increase in sample mass as the SO₂ pressure increased. The pressure was increased in time intervals, allowing for saturation of the sample at each stage. The apparatus typically allows water vapour to be introduced, but this was not done in the present study. An air inlet was used to fill the apparatus with gas once a vacuum had been established. Only sorption curves were recorded.

The sorption was performed in three cycles, including a sorption and desorption process on the same pellet. Figures 5 and 6 show sorption curves only. Desorption was performed by reducing the pressure to 10⁻² Pa. The cyclic adsorption-desorption experiments aimed to investigate the regeneration possibilities of the material analysed. The SO₂ sorption experiment was performed on a powder fly ash zeolite sample. Zeolite pellets were prepared in various ways as follows: without the use of a binder (G); with the addition of polyethyleneimine (GPEI); with the addition of starch (GS); and with the addition of montmorillonite (GM). For the sake of clarity and to enable analysis, sorption tests were also performed with pure binders.

To determine the nature of SO₂ binding, Fourier-transform infrared (FTIR) spectra were obtained for pellets before and after SO₂ sorption with a Thermo Nicolet 380 FTIR spectrometer. Sixty scans were obtained for each spectrum in the range of 4000 to 400 cm⁻¹. The samples being investigated were mixed with KBr and pressed into discs.

The chemical composition of the pellets before and after SO₂ sorption was determined by XRF.

Results

Pelletization

Pelletization of the zeolites with and without the addition of binders was conducted with a hydraulic press. The pellets produced without the addition of a binder were split into two parts (delaminated), indicating that an additional binder would be necessary. The pellets formed with the addition of a binder showed improved mechanical durability. However, after ageing and drying of the pellets, delamination was again observed. Nevertheless, the pellets did not disintegrate and only slight delamination was observed. It is possible that the production of a pellet with a smaller surface would eliminate delamination. In addition, a drop of excess liquid was observed during the powder-pressing process

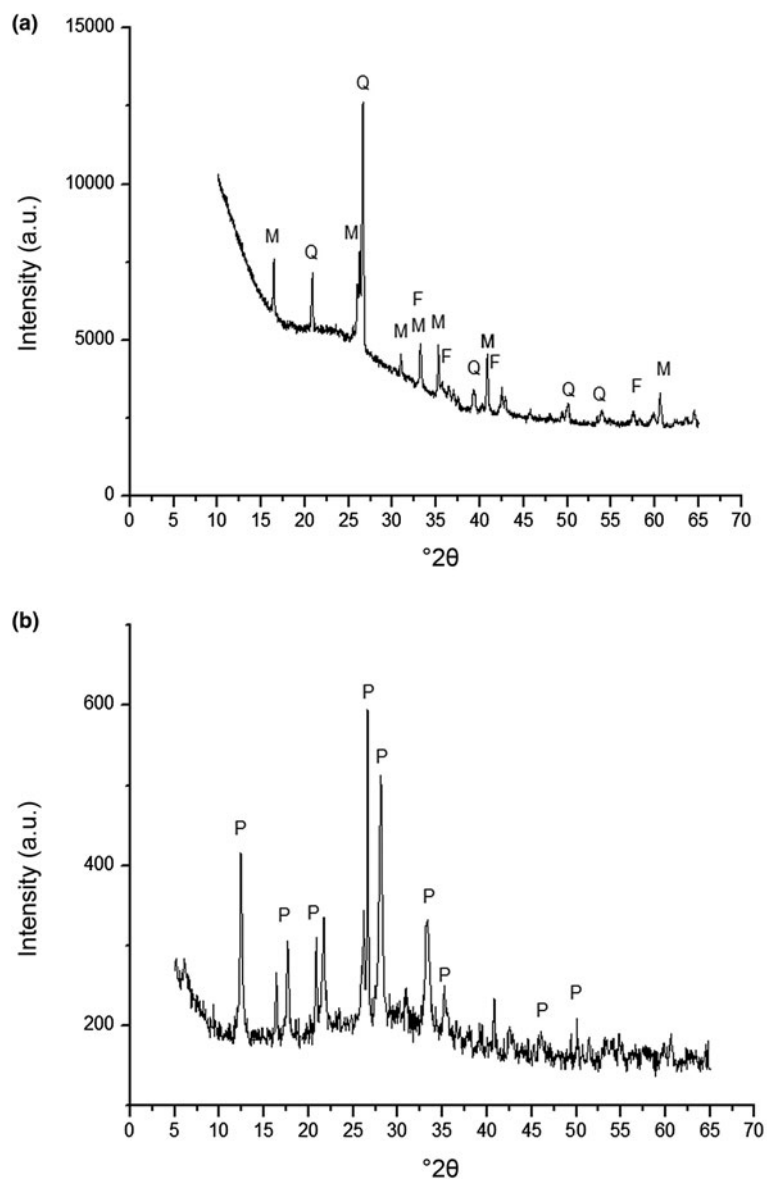


Fig. 1. XRD traces of (a) the fly ash and (b) the fly ash zeolite. M = mullite; P = zeolite P1; Q = quartz; F = Fe₂O₃.

with aqueous polyethyleneimine solution. This may suggest that excess binder had been added and that the observed binder remains did not enter the pores or interpellet spaces of the fly ash zeolite.

SO₂ sorption

The adsorption isotherms of SO₂ on the zeolite pellets are presented in Fig. 5.

The adsorption isotherms of SO₂ on pure binders are presented in Fig. 6.

The chemical composition of the pellets before and after SO₂ sorption is listed in Table 3.

The FTIR data of the samples before and after the SO₂ adsorption are presented in Fig. 7.

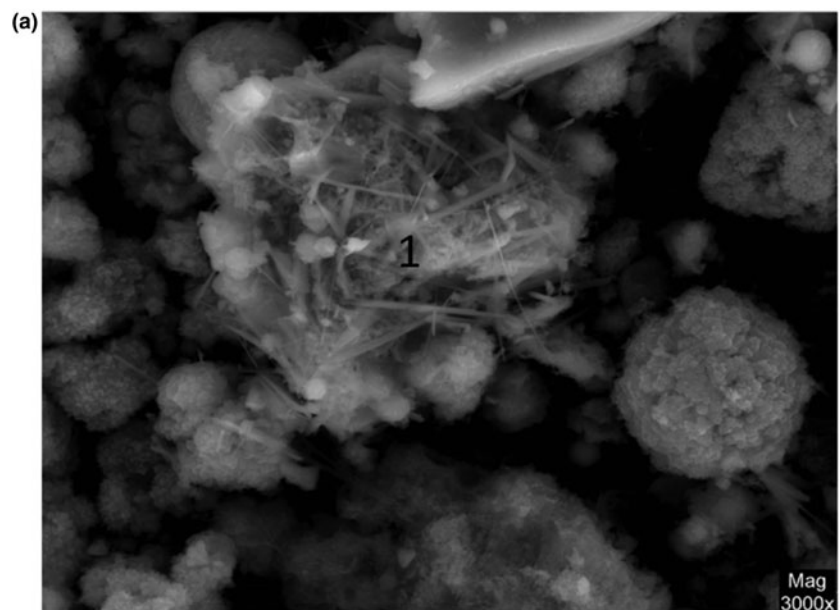
Discussion

The sorption experiments showed that synthesized fly ash zeolite had the ability to adsorb SO₂. However, the pelletization process

with binders did not lead to a permanent increase in pellet durability.

The sorption capacity of the material dust was tested in cycles. The sorption capacity was 0.8 mmol g⁻¹ in the first cycle. In the second and third cycles, a decrease in sorption capacity of up to ~0.25 mmol g⁻¹ was observed. The decrease in sorption capacity is most likely related to the chemisorption reaction of SO₂ with unreacted fly ash or sodium hydroxide residues present in the material, as it was not possible to completely remove these from the material after the synthesis process, thereby creating sulfites. This can be confirmed by the relatively constant sorption capacity values in the second and third cycles.

The pellets produced without a binder showed a decrease in sorption capacity in comparison to the powder material with respect to the first and subsequent cycles. The decreased sorption capacity values were relatively constant for the second and third cycles. A reduction in the sorption capacity value of the material subjected to pelletization is associated with a reduced likelihood of SO₂ diffusion into the interior of the pellets due to the sample compacting (Juan *et al.*, 2009).



1- point for EDS analysis

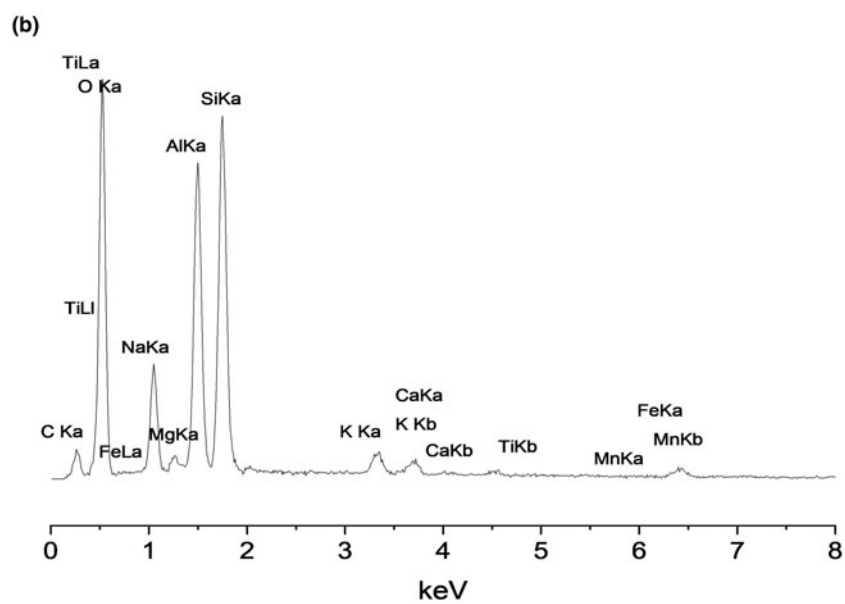


Fig. 2. (a) SEM image and (b) EDS spectrum of the fly ash zeolite.

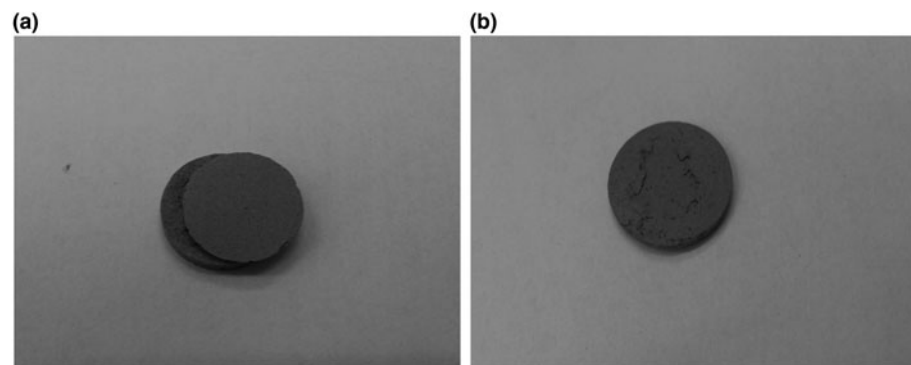
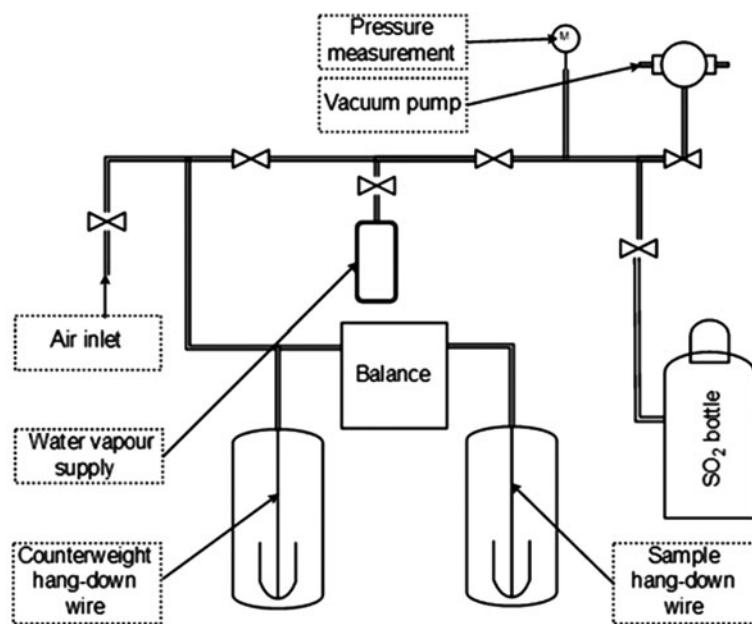


Fig. 3. Photographs of (a) a fly ash zeolite pellet without binder and (b) with added starch binder.

Table 2. Binders used to improve the mechanical durability of fly ash zeolite subjected to the pelletization process.

Binder	Amount	Comments	Sample designation
Polyethyleneimine, 50% water solution	10% addition by mass	The zeolite material was mixed thoroughly and subjected to pelletization	GPEI
Food starch	10% addition by mass	Reduce the width of this column so that the highlighted text here reaches the bottom of the table, as this text relates to both the 'Food starch' and the 'Montmorillonite' rows	GS
Montmorillonite	10% addition by mass		GM

**Fig. 4.** Experimental setup for the SO₂ sorption experiments.

The binder additives produced interesting results in terms of their influence on sorption capacity. Both the polyethyleneimine and starch additives led to a greater absorption capacity than pellets formed without an added binder, suggesting that the binders are also active in the sorption process. This was confirmed by analysis of the sorption capacity of the pure binder (Fig. 6). A decrease of the sorption capacity after the first cycle was observed in all of the experiments. The sorption capacity values in the second and third cycles were similar to those observed for non-pelletized powder for most of the binders. In practice, this means that the pelletization process decreases only slightly the sorption capacity of material produced with the addition of a binder in the second and third sorption cycles. The use of montmorillonite had the least influence in terms of the effect on sorption properties because it yielded the lowest values. This is an unexpected result because previous work has shown that the use of bentonite as a binder is beneficial due to its elastic properties, which determine performance properties such as the pellets' resistance to deformation (Li *et al.*, 2001). The main constituent of bentonite is montmorillonite, which has ion-exchange properties, a large specific surface area and absorption and adsorption properties of ionic and polar substances (Uddin, 2018), making it a sorbent itself. Impurities typically present in bentonite include quartz, illite, muscovite, plagioclase and calcite, which do not have sorption properties (Karnland, 2010). Therefore, montmorillonite rather of bentonite was used in this study. Pure montmorillonite has a greater SO₂ sorption capacity than the fly ash zeolite. This was not observed in the sorption capacity of the pellets as the sample with added montmorillonite had the smallest sorption

capacity. This might be associated with the diffusion limits due to thickening of the material, as was observed with the remaining pellets. Nevertheless, the sorption experiments on the GM sample and pure montmorillonite (and comparison of these with the GPEI and GS samples) did not provide an explanation for this result.

From an economic point of view, the most promising material used as a binder was starch due to its low price. Starch was selected for this work due to its adhesion properties (Agusta *et al.*, 2017). In pellets with added starch, similar sorption capacity values were recorded to those of samples pelletized with polyethyleneimine. However, despite its positive effect on sorption (Tailor *et al.*, 2014), the high price of polyethyleneimine means that its use is not economically feasible. In addition, stable sulfate salts are formed when polyethyleneimine is used due to the high affinity of SO₂ for amino groups (Nelson *et al.*, 2014), which may explain the lower sorption capacity observed after the first sorption cycle.

Previous work on the use of fly ash zeolites for SO₂ adsorption with a feed-gas mixture of SO₂, O₂, CO₂ and N₂ has reported complete removal of SO₂ after 7 min of gas flow (Srinivasan & Grutzeck, 1999). Hence, it may be assumed that the sorption capacity of the pellets in this study is very close to the value obtained with a feed-gas mixture.

Analysis of the chemical composition of the materials before and after SO₂ sorption shed light on the nature of the sorption process. A large increase in sulfur content was observed in all of the samples after SO₂ sorption, suggesting that there was an initial chemical reaction between SO₂ and the remains of NaOH (present in the samples after synthesis, activated by the basic solution), the fly ash constituents or the binders used.

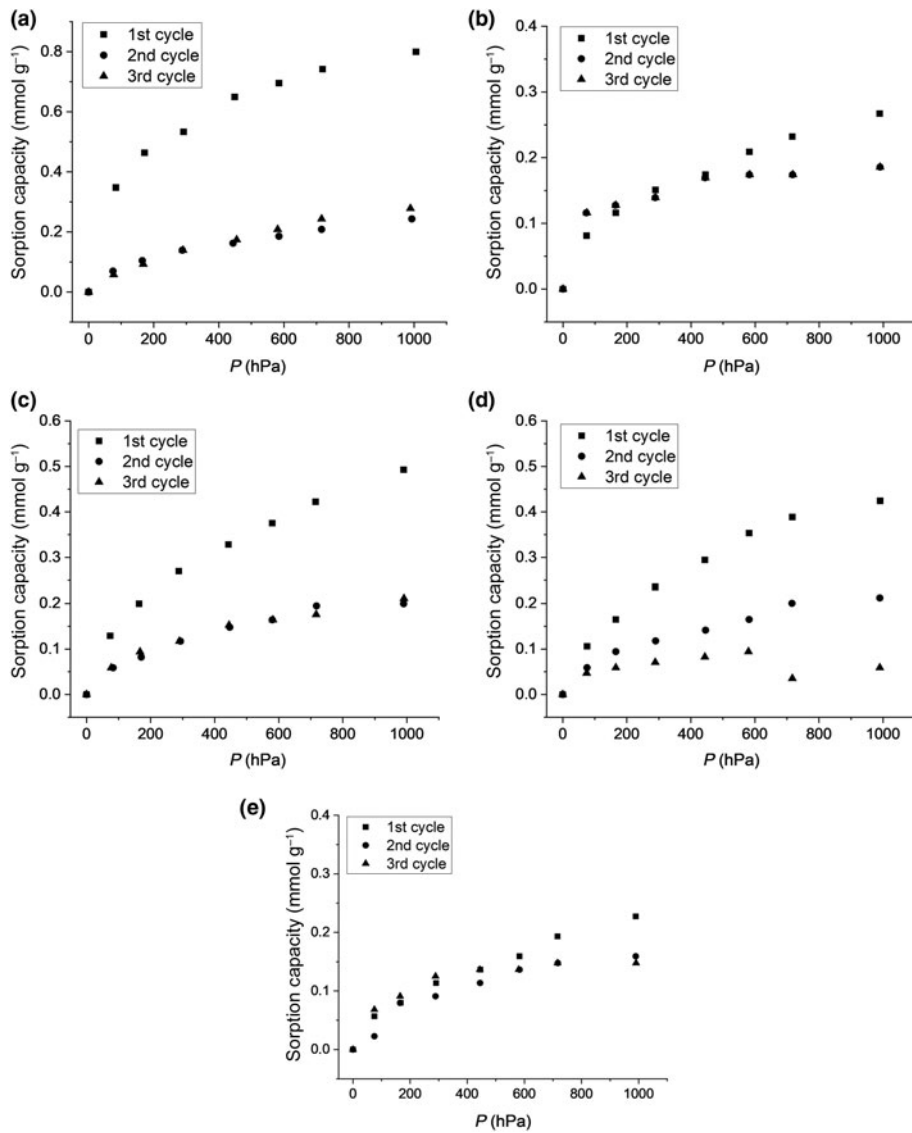


Fig. 5. Results of the SO₂ sorption experiments: (a) non-pelletized fly ash zeolite; (b) sample G; (c) sample GPEI; (d) sample GS; (e) sample GM.

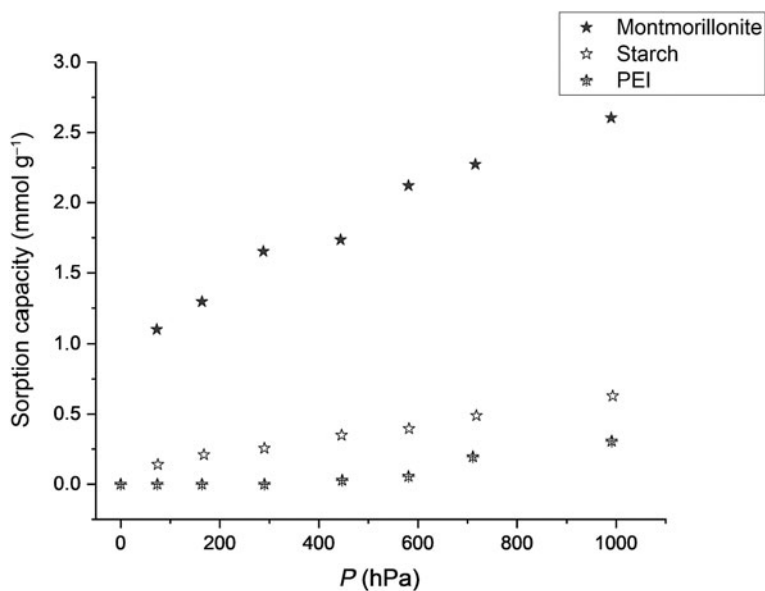


Fig. 6. Results of the SO₂ sorption experiments with pure binders.

Table 3. Chemical composition (wt.%) of fly ash and the pellets before and after SO₂ sorption.

Component (wt.%)	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	TiO ₂	K ₂ O	ZnO	Na ₂ O	SO ₃
Sample G	42.20	4.00	28.60	8.70	2.60	1.50	0.90	1.50	9.40	bld
Sample G post-SO ₂ sorption	40.99	3.17	22.65	6.45	1.03	1.15	2.64	0.04	7.78	13.62
Sample GPEI	47.40	3.99	25.39	8.60	1.42	1.45	3.22	0.05	7.31	bld
Sample GPEI post-SO ₂ sorption	39.65	3.19	22.65	6.45	1.03	1.15	2.64	0.04	7.28	13.62
Sample GS	48.23	3.67	26.54	7.65	1.38	1.34	3.00	0.04	8.13	bld
Sample GS post-SO ₂ sorption	41.34	3.34	23.26	7.07	1.17	1.18	2.66	0.04	7.44	11.42
Sample GM	52.02	3.11	24.80	6.54	1.40	1.15	2.78	0.03	7.10	bld
Sample GM post-SO ₂ sorption	46.30	2.98	22.12	6.24	1.19	1.10	2.64	0.03	6.04	10.46

bld = below limit of detection.

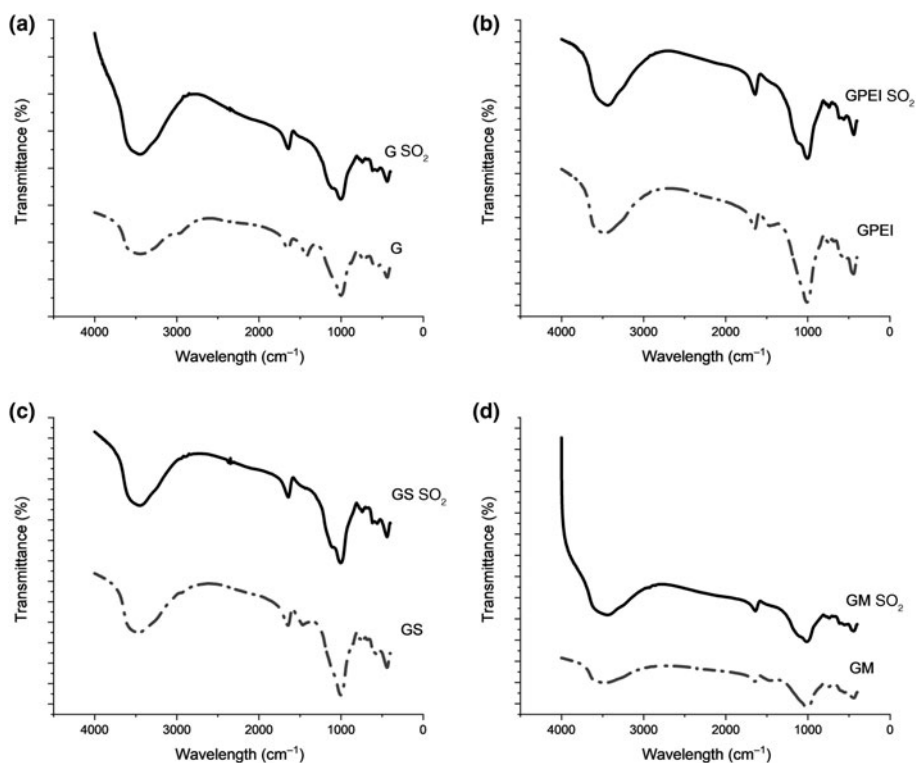


Fig. 7. FTIR spectra of samples before and after SO₂ sorption: (a) sample G; (b) sample GPEI; (c) sample GS; (d) sample GM.

Additional confirmation of the chemical reaction that is most likely to have taken place can be found from the analysis of the FTIR data. Indeed, in all of these cases, the FTIR spectra of the post-SO₂ sorption samples show the presence of a new band in the 1110–1170 cm⁻¹ region, which is attributed to S = O stretching (www.instruction.greenriver.edu).

Summary and conclusions

Fly ash is a suitable raw material for the synthesis of zeolites. However, pelletization of the zeolite powder should be performed prior to testing for use in various applications. The pelletization process performed in this study led to thickening of the material and prevented disintegration of the pellets. The addition of a binder did not enhance permanently the durability of the pellets in comparison to pellets formed without the addition of a binder.

The fly ash zeolite investigated has sorption capacity for SO₂. Non-pelletized fly ash zeolite showed the greatest sorption capacity, which decreased after the first sorption–desorption cycle, probably due to the reaction of SO₂ with the sodium hydroxide present in the sample after the synthesis process. The pelletization process led to a decrease in sorption capacity due to thickening of the material and

occurring as a result of this diffusion limit. After the first sorption–desorption cycle, a decrease in sorption capacity was observed, as in the case of non-pelletized zeolite. The pure binders also had sorption capacity for SO₂. The greatest sorption capacity was observed for montmorillonite. Regarding the remaining pellets formed with the addition of a binder, the slight increase in sorption capacity (in comparison to pellets formed without a binder) is attributed to the amount of binder taking part in the sorption process.

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