



## The effectiveness of modified sodium bicarbonate in the purification of exhaust gases from HCl and HF

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### ABSTRACT

**Purpose:** The effectiveness of gas purification depends on the sorption properties of sorbents. The aim of the research was to determine the ability of sodium sorbents to remove gaseous pollutants such as HCl and HF.

**Design/methodology/approach:** The research used baking soda subjected to mechanical and thermal activation, which was introduced into the hot flue gas circuit, similar to the dry flue gas cleaning method used in all kinds of boilers.

**Findings:** In almost all cases, mechanical and thermal treatment of baking soda allowed for a significant improvement in the reduction of hydrogen chloride and hydrogen fluoride concentrations in the exhaust gases produced during the combustion of polyethylene terephthalate (PET) bottles.

**Research limitations/implications:** The preparation of sorbents must consider the optimum development of the material's specific surface area, especially during thermal activation, to prevent pores from sintering.

**Practical implications:** In wet and dry flue gas cleaning processes, sodium compounds are used in the power industry. Appropriate preparation of the sorbent allows for improving the effectiveness of reducing the concentration of harmful substances and reducing investment and operating costs.

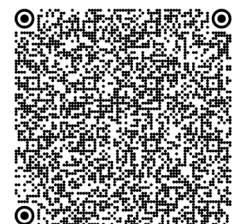
**Originality/value:** From the point of view of process optimisation, the results obtained will contribute to the identification of optimal operating conditions in dry sorbent injection systems to prevent the pores on the surface of the sorbent from clogging when injected into hot flue gases.

**Keywords:** Sodium bicarbonate, Removal of HCl and HF, Mechanical and thermal activation, Dry sorbent injection

**Reference to this paper should be given in the following way:**

D. Wysocki, A. Szymanek, The effectiveness of modified sodium bicarbonate in the purification of exhaust gases from HCl and HF, Journal of Achievements in Materials and Manufacturing Engineering 114/2 (2022) 57-66. DOI: <https://doi.org/10.5604/01.3001.0016.2156>

### PROPERTIES



## 1. Introduction

Bearing in mind the increasingly stringent limits of pollutant emissions, more reactive products are needed to improve flue gas sorption efficiency and meet legal requirements without the need to change the technology. To meet these requirements, this work focuses on developing a methodology for removing HCl and HF compounds from flue gases.

The justification for such a decision is confirmed by the preparation by the European Parliament and the European Council of documents which address the issue of, among others, the level of HCl and HF emitted during the combustion of hard coal, lignite or solid waste. It indicates permissible hydrogen chloride emissions for power plants with a thermal capacity below 100 MW of 1-6 mg/nm<sup>3</sup> for new plants and 2-10 mg/nm<sup>3</sup> for existing plants, and plants above 100 MW of 1-3 mg/nm<sup>3</sup> and 1-5 mg/nm<sup>3</sup>, respectively. Furthermore, for all fluidised bed combustion boilers, plants operating less than 1500 hours/year, and plants burning fuels where the average chlorine content is at or above 1000 mg/kg dry weight, the upper limit of the HCl emission range is 20 mg/nm<sup>3</sup>. Additionally, the permissible HCl emission for existing sources with a capacity above 100 MW equipped with wet flue gas desulphurisation systems and a flue gas heater located at the outlet behind the flue gas desulphurisation system shall be 7 mg/nm<sup>3</sup>.

Permissible emission of hydrogen fluoride for sources with a heat capacity below 100 MW for new plants are below 1-3 mg/m<sup>3</sup>n and below 1-6 mg/m<sup>3</sup>n for existing plants, while for sources with a heat capacity above. 100MW, respectively below 1-2 mg/nm<sup>3</sup> and below 1-3 mg/nm<sup>3</sup>. As in the case of HCl emissions, the upper limit of HF emissions in all existing plants equipped with wet flue gas desulphurisation systems and a flue gas heater located at the outlet behind the flue gas desulphurisation system, in fluidised bed combustion boilers and plants which are operated for less than 1500 hours/year, shall be 7 mg/nm<sup>3</sup>. Emission limits for HCl and HF are also in place for waste incineration plants where the average daily air emission limit value for HCl is 10 mg/nm<sup>3</sup> and for HF. 1 mg/nm<sup>3</sup> [1-3].

Due to the introduced emission limits, it is necessary to have more effective purification of waste gases through optimised technologies. Sodium sorbents are an example of a commonly used compound in flue gas cleaning processes, showing very good sorption properties in the reduction of SO<sub>2</sub>, HCl, HF and NO<sub>x</sub>. One of the most common application methods for sodium sorbents is the dry sorbent injection process, in which the absorbent material is injected

into the hot flue gas stream and can remove up to 95% of SO<sub>2</sub> and almost all SO<sub>3</sub>, HCl and HF.

Because of the low investment cost and ease of operation, dry injection of sodium sorbents is used in many boilers as an alternative to wet flue gas cleaning processes. High capital and operating costs make them uneconomical for smaller utility boilers. (<250 MW), industrial coal-fired boilers and waste-to-energy boilers [4-6].

## 2. Material and methods

### 2.1. Properties and application of sodium bicarbonate

The material used in the study of the reduction of HCl and HF concentrations from flue gas was sodium bicarbonate offered by the baking soda manufacturer and sodium carbonate Soda Polska CIECH Sp. z o.o. Baking soda is a high-purity commercial product of standard class, which in its composition contains over 99.8% of sodium bicarbonate NaHCO<sub>3</sub>. The product was characterised by the average grain size of 126 μm and, as a crystalline product, a compact structure with a weakly developed structure without pores and cracks [7]. The full chemical composition of the material used is shown in Table 1.

Table 1.  
Physical and chemical properties of baking soda [7]

Component	Unit	Value
pH	-	8.1
NaHCO <sub>3</sub> on the basis of dry weight	%	99.81
Na <sub>2</sub> CO <sub>3</sub>	%	0.22
Chlorides	%	0.005
Sulphates	%	<0.02
Insoluble Substances	%	0.0005
Mass loss (50°C)	%	0.02
Ignition loss (280°C)	%	36.8
Cadmium (Cd)	mg/kg	<0.025
Cobalt (Co)	mg/kg	<0.025
Copper (Cu)	mg/kg	0.05
Lead (Pb)	mg/kg	<0.025
Nickel (Ni)	mg/kg	<0.025
Iron (Fe)	mg/kg	1.8
Zinc (Zn)	mg/kg	<5.0
Mercury (Hg)	mg/kg	0.004
Fluorine (F)	mg/kg	0.000

Many factors influence the performance of sodium sorbents used in flue gas cleaning. Apart from reactivity, grain size and porosity are decisive. To reduce their size, they are mechanically modified by comminution. Figure 1 shows the compact surface of sodium bicarbonate. In

contrast, Figure 2 shows the effect of grinding the material, which caused disintegration into smaller particles and the formation of cracks and scratches on the surface of the formed grains.

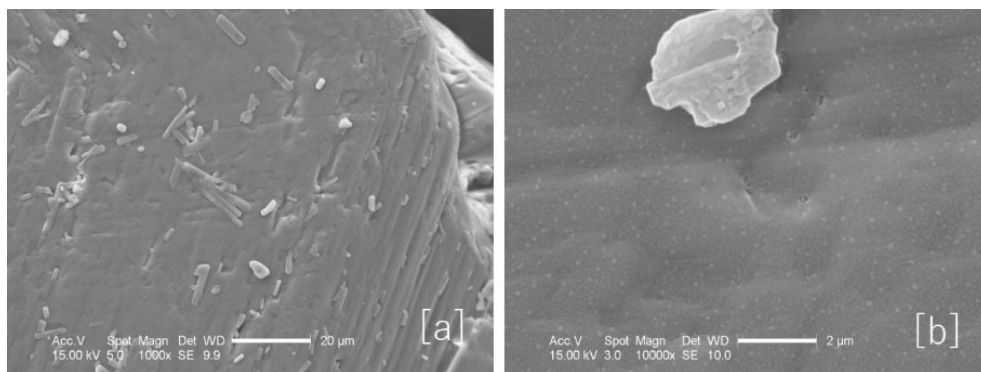


Fig. 1. Surface of unmodified sodium bicarbonate enlarged: a) 1000x, b) 10000x [8]

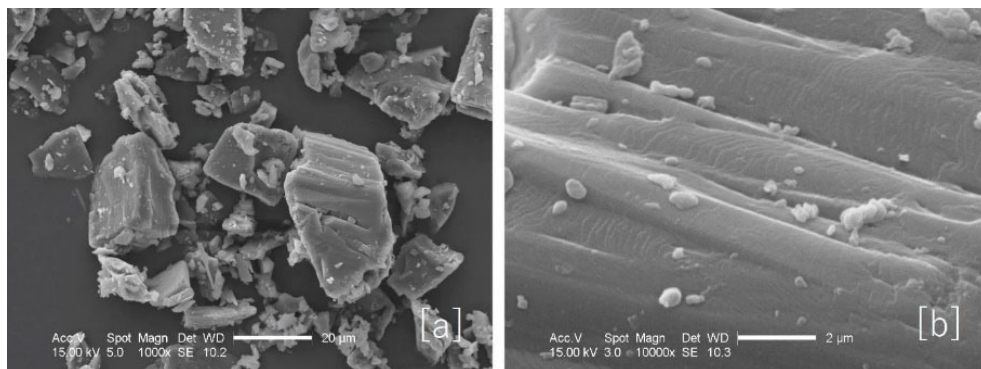


Fig. 2. Surface of comminuted sodium bicarbonate enlarged: a) 1000x, b) 10000x [8]

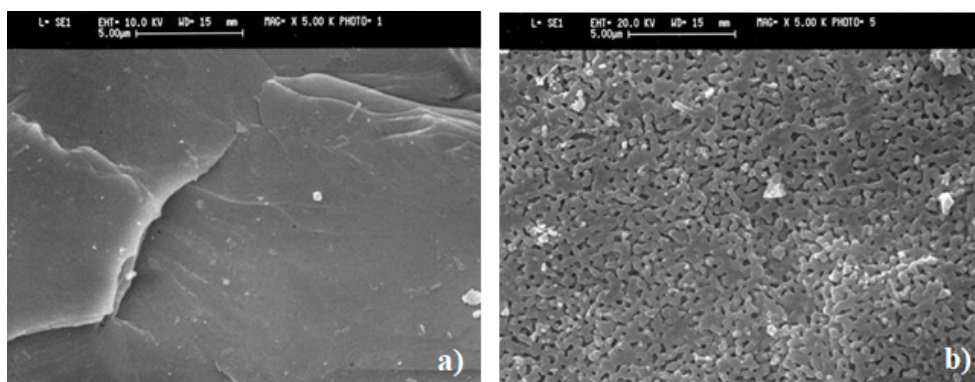


Fig. 3. a) Unmodified sodium bicarbonate under the microscope, b) Calcined sodium bicarbonate under a microscope [5]

In contrast to larger ones, smaller particles have a larger active surface area reacts faster and more efficiently with contaminants. In addition, sodium sorbents are thermally activated to increase their porosity. In the dry sorbent injection system, sodium bicarbonate  $\text{NaHCO}_3$  is injected directly into the hot flue gas, which calcines into porous sodium carbonate  $\text{Na}_2\text{CO}_3$ . Porous sodium carbonate enables rapid gas-solid reactions with the acid gases in the flue gases ( $\text{SO}_2$ ,  $\text{SO}_3$ ,  $\text{HCl}$ ,  $\text{HF}$ ,  $\text{NO}_x$ ). The chemical reactions between porous activated sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) and acid gases are:

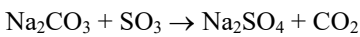
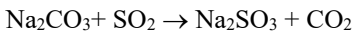
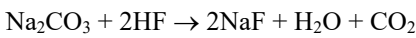
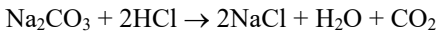


Figure 3a shows crude sodium bicarbonate, characterised by a uniform crystalline structure. Figure 3b shows the effect of bicarbonate calcination, the formation of pores, and the expansion of the specific surface [5].

Figure 4 shows a typical sorbent dry injection system based on sodium bicarbonate. The sorbent can be injected almost anywhere on the flue gas outlet as long as the flue gas temperature enables its conversion into sodium carbonate. In addition, sodium-based sorbents do not require make-up water injection compared to calcium-based sorbents. The effectiveness of dry sodium injection depends on several factors, such as the mixing between the sorbent and the flue gas, residence time, flue gas temperature, the size of the sorbent grains and the rate of its administration.

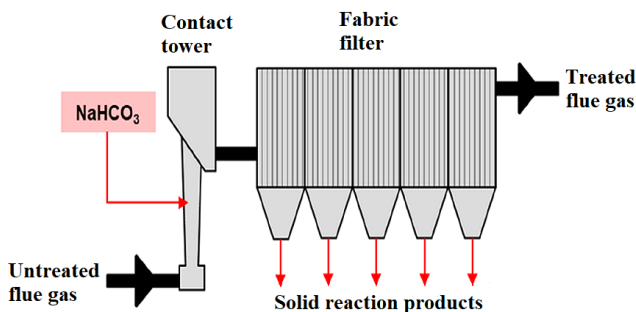


Fig. 4. Typical dry sorbent injection system based on sodium bicarbonate [10]

The efficiency of dry sodium injection depends on several factors, such as mixing between sorbent and flue gas, residence time, flue gas temperature, sorbent grain size and feed rate. Therefore, it is important to properly adjust the sorbent to the flue gas cleaning system used. In industry, dry sorbent injection is performed before a particulate collection device, which is usually a fabric filter [8-10].

## 2.2. Mechanical activation of sodium sorbents

The mechanical activation process involves micronising material grains using various comminution techniques. Equipment for the mechanical processing of mineral raw materials used in the industry uses the following grinding or crushing mills: hammer, vibrating, ring, disc, ball, jet, mixing, colloidal, and fluidised. Each of the listed grinding methods has its specific characteristics. Nevertheless, regardless of the grinding method used, there is a search for such operating conditions for industrial equipment, which on the one hand, guarantees the cleanliness of shredded products through a selection of appropriate parameters of the working medium and, on the other hand, enables reduction of energy consumption during the grinding process and guarantees improvement of environmental protection requirements. Figure 5 shows the diagrams of the comminution process [11,12].

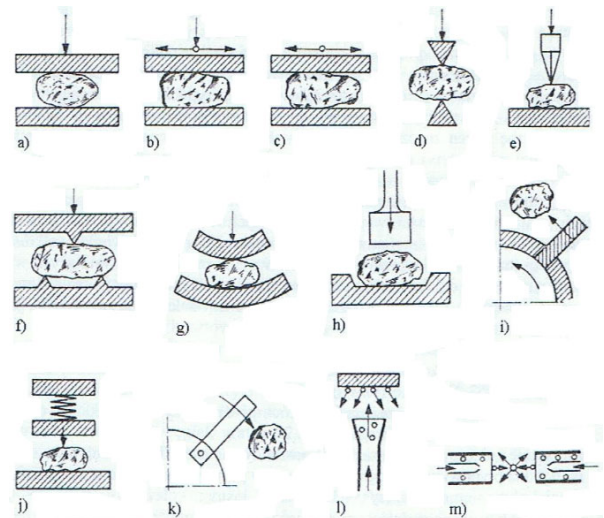


Fig. 5. Schematic of comminution process: a) bending, b) abrasion pressure, c) abrasion, d) shearing, e) splitting, f,g) breaking, h, i) impact, j,k) elastic impact, l) jet crushing, grains hitting against the crushing plate, m) jet crushing without mechanical working elements, mutual collision of grains [11]

During the research, impact and electromagnetic field crushing was carried out in two grinding devices: an impact mill and an electromagnetic mill. The 160 UPZ impact mill uses the milling technique, in which the raw material is crushed when struck by steel fingers arranged on a disc and a spinning rotor. The material to be tested is fed into a hopper, where a vibrating chute feed it into the mill chamber. The rotations of the spinning disc (rotor) are regulated, and the ground material, resulting from the vacuum generated by the fan, settles on the walls of the bag filter, from which it is then collected [13,14]. The Hosokawa-Alpine impact mill, shown in Figure 6, was used for activation.

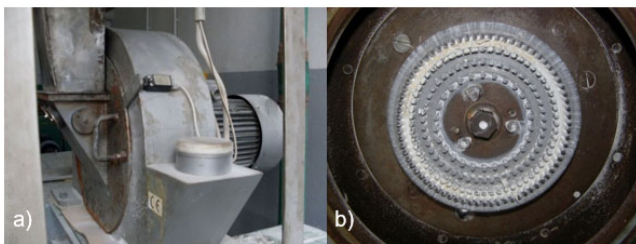


Fig. 6. Impact mill 160 UPZ: a) general view, b) working chamber

The second device used for mechanical activation of the sorbent was an electromagnetic mill, shown in Figure 7, in which the shredded material is subjected to a rotating magnetic field by means of ferromagnetic grinding media with dimensions ranging from a few to several millimetres.

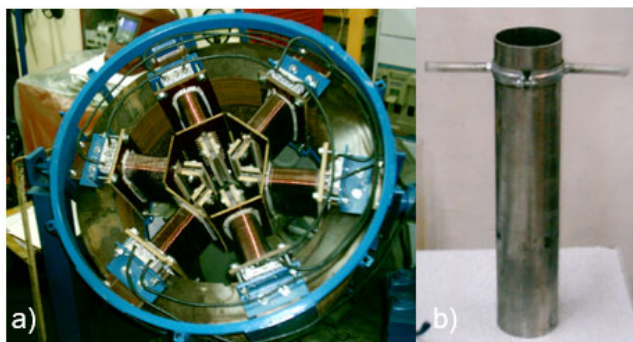


Fig. 7. Electromagnetic mill: a) general view, b) working chamber

The large number and high speed of the grinding media ensure that the ultra-fine particle structures of the shredded material are repeatedly struck, resulting in a very rapid and

turbulent micronisation process in the working chamber. The energy generated in the active zone of the mill is concentrated mainly in the collision areas of the rotating grinding media and reaches significant values, causing local pressures of up to 1000 Pa, and the power supplied to the grinding zone reaches a value of 1.5-2.0 MW/m<sup>3</sup> [15,16].

### 2.3. Thermal activation of sodium sorbents

After mechanical activation, the obtained sorbents were subjected to thermal activation consisting of annealing sodium bicarbonate at elevated temperatures for a specific period. Sorbents were annealed at 100°C and 250°C for 30 minutes, which, as mentioned earlier, caused the disintegration of its crystalline structure and, consequently, an improvement in its structural parameters, increasing the sorption potential of chemical compounds.

Figure 8 shows the study results [10] of the effect of thermal activation at 150°C, 180°C and 300°C for 90 minutes on the morphology of a soda-based sorbent.

As shown in Figure 8a, sodium bicarbonate is a non-porous solid. During the activation of NaHCO<sub>3</sub>, H<sub>2</sub>O and CO<sub>2</sub> are released, causing the formation of a porous structure in the resulting Na<sub>2</sub>CO<sub>3</sub> can be observed in Figures 8b 150°C and 8c 180°C. Figure 8d shows the surface of a sample annealed at 300°C. According to the author, the pores formed during the calcination of sodium bicarbonate undergo sealing. The sintering of porous sodium carbonate grains takes place, which leads to a decrease in specific surface area and, consequently, to a deterioration of the sorption capacity of the sorbent.

### 2.4. Reduction of HCl and HF concentrations in the flue gas

The analysis of the exhaust gas cleaning was carried out as follows. First, the concentration of gaseous substances was measured during the combustion of the alternative fuel in the form of PET bottles (background), which was done before the purification process at 850°C. The background time was measured until the concentrations of the gaseous substances HCl were fully stabilised at 20 mg/nm<sup>3</sup> and the concentration of HF at 45 mg/nm<sup>3</sup>. Subsequently, the prepared sodium sorbents in the form of baking soda, both ungrounded and mechanically and thermally modified, similarly to the dry flue gas cleaning method, were fed continuously into the second boiler pass at the flue gas outlet before the electrostatic precipitator into the flue gas stream at a temperature of about 300°C. A diagram of the workstation is presented in Figure 9.

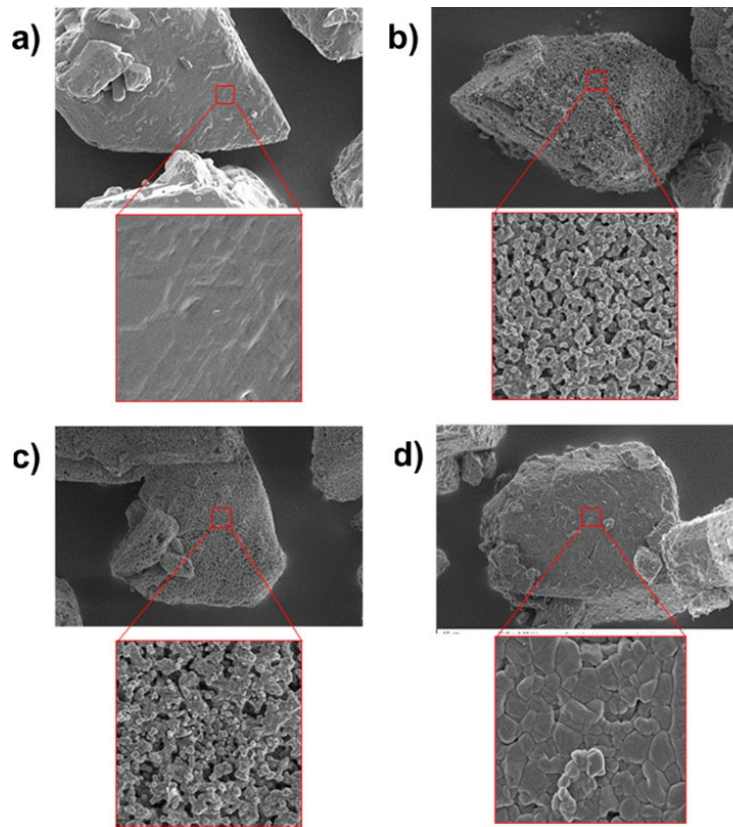


Fig. 8. SEM photography of the sodium bicarbonate a) before activation, b) activation at 150°C, c) activation at 180°C, d) activation at 300°C [10]

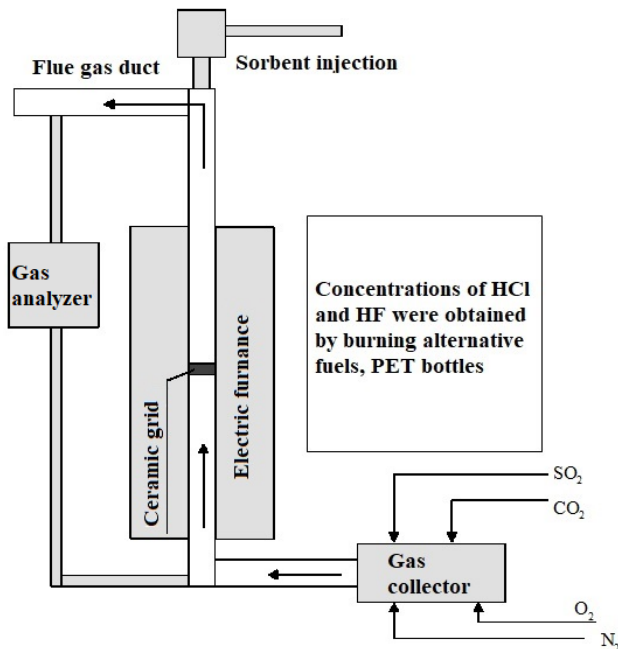


Fig. 9. The schematic diagram of the test stand

Gas concentrations were measured using the GASMET DX-4000 flue gas analyser. This portable gas analyser enables simultaneous measurement of up to 50 chemical compounds present in flue gases, waste gases, process gases and air. Thanks to the use of the "hot" method (the entire measurement circuit is kept at 180°C), it is possible to measure the concentrations of substances that are easily soluble in water, i.e.,  $\text{NH}_3$ , HF or HCl, as well as selectively  $\text{H}_2\text{O}$  and hydrocarbons, which is not possible with the traditional "cold" measurement method. The sample spectrum collected by the analyser is analysed by the patented CALCMET software [17].

### 3. Result and discussion

The application of mechanical activation allowed to obtain sorbents with different average grain sizes and to break down the smooth crystalline surface of the baking soda, causing the formation of pores and cracks. Impact grinding produced a sorbent with an average grain size of

20.76  $\mu\text{m}$ . Electromagnetic field modification produced a product with an average grain size of 30.20  $\mu\text{m}$ .

To more accurately characterise the changes in sorbent properties due to activation, the material's specific surface area per unit mass was determined (BET adsorption method). Table 2 shows the structural properties of mechanically and thermally activated sodium sorbents.

Table 2.  
Structural parameters of the obtained sorbents

No.	Sorbent type	Average grain size, $\mu\text{m}$	Specific surface, $\text{m}^2/\text{g}$
1.	Baking soda	126.00	0.09
2.	Impact activation	20.76	0.20
3.	Impact and thermal 100°C activation	20.76	1.82
4.	Impact and thermal 250°C activation	20.76	5.97
5.	Electromagnetic activation	30.20	0.76
6.	Electromagnetic and thermal 100°C activation	30.20	4.58
7.	Electromagnetic and thermal 250°C activation	30.20	8.64

Mechanical activation by breaking the baking soda into finer cracked and scratched grains allowed for the development of the specific surface of the shredded material. A larger specific surface characterises sorbents subjected to additional thermal activation. This means that the annealing has fulfilled its task, ensuring the formation of pores on the surface of the shredded sorbent grains.

The obtained results of HCl and HF concentration reduction in flue gases with the use of the sorbents presented in Table 2 are presented in Figures 10-15.

The introduction of unmodified baking soda into the flue gas duct allowed to significantly reduce HCl concentration (from 20.77  $\text{mg}/\text{nm}^3$  to 13.20  $\text{mg}/\text{nm}^3$ ) but did not contribute to the reduction of HF concentration. (45  $\text{mg}/\text{nm}^3$ ). In comparison, the use of mechanically modified sodium bicarbonate with a better developed specific surface allowed to reduce the HCl content approximately twice as well (impact mill 8.55  $\text{mg}/\text{nm}^3$ , electromagnetic mill 5.04  $\text{mg}/\text{nm}^3$ ) as a visible reduction of the HF level (impact mill 38.05  $\text{mg}/\text{nm}^3$ , electromagnetic mill 39.46  $\text{mg}/\text{nm}^3$ ).

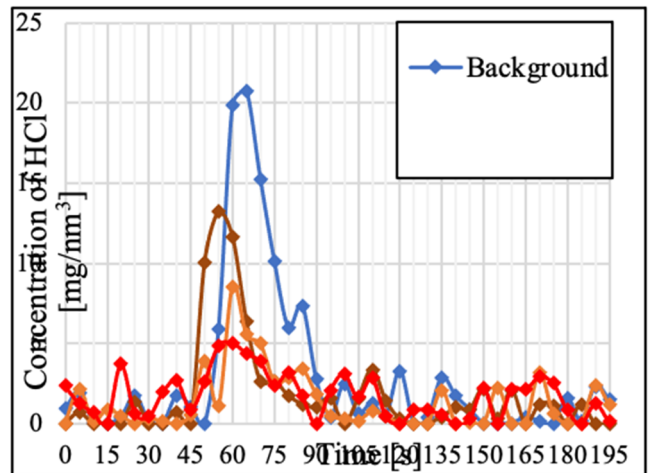


Fig. 10. HCl concentration during testing as a function of time for mechanically activated sorbents

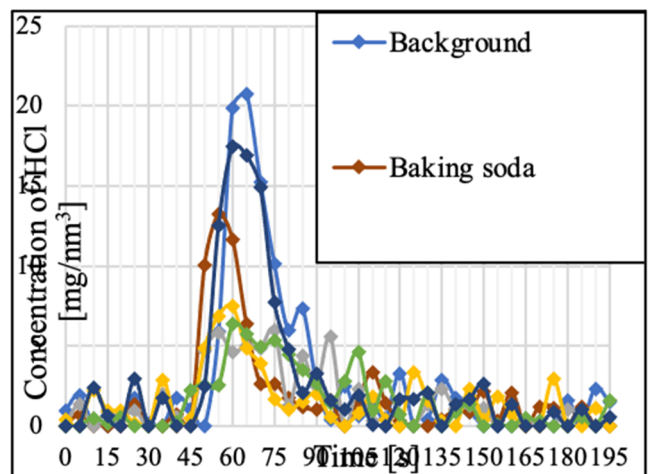


Fig. 11. HCl concentration during testing as a function of time for thermally activated sorbents

The use of additional thermal activation at 100°C had a positive effect on the ability of impact mill-activated sorbents to reduce HCl (from 8.55  $\text{mg}/\text{nm}^3$  to 5.98  $\text{mg}/\text{nm}^3$ ) and HF (from 38.05  $\text{mg}/\text{nm}^3$  to 37.59  $\text{mg}/\text{nm}^3$ ).

The application of additional thermal activation at 100°C resulted in a slight deterioration in the ability of the electromagnetic mill-activated sorbents to reduce HCl (from 5.04  $\text{mg}/\text{nm}^3$  to 6.36  $\text{mg}/\text{nm}^3$ ) while providing a similar concentration of HF (from 39.46  $\text{mg}/\text{nm}^3$  to 39.26  $\text{mg}/\text{nm}^3$ ).

Heating at 250°C did not have the desired effect. In the case of material crushed in an impact mill, the effectiveness

of HCl removal was slightly better ( $7.45 \text{ mg/nm}^3$ ) than that of the material subjected only to grinding ( $8.55 \text{ mg/nm}^3$ ). In comparison, the HF reduction deteriorated (from  $38.05 \text{ mg/nm}^3$  to  $41.48 \text{ mg/nm}^3$ ). Thermal activation at  $250^\circ\text{C}$  of an electromagnetically modified sorbent gave the worst results. Its use allowed the HCl concentration to be reduced ( $17.44 \text{ mg/nm}^3$ ), but to a lesser extent than in the case of unmodified baking soda ( $13.20 \text{ mg/nm}^3$ ). In the case of HF concentration, it can be noticed that the introduction of sorbent caused a significant increase in HF concentration in the tested flue gases ( $132.05 \text{ mg/nm}^3$ ).

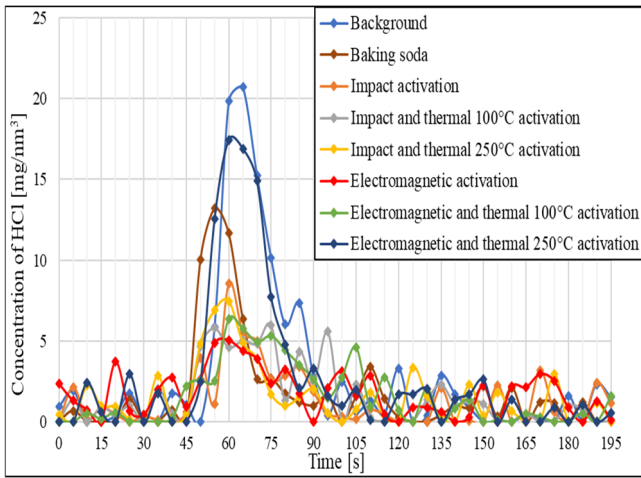


Fig. 12. HCl concentration during testing as a function of time for all sorbents

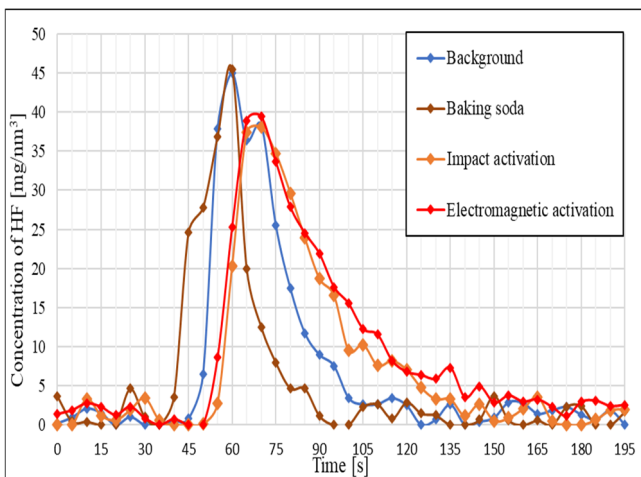


Fig. 13. HF concentration during testing as a function of time for mechanically activated sorbents

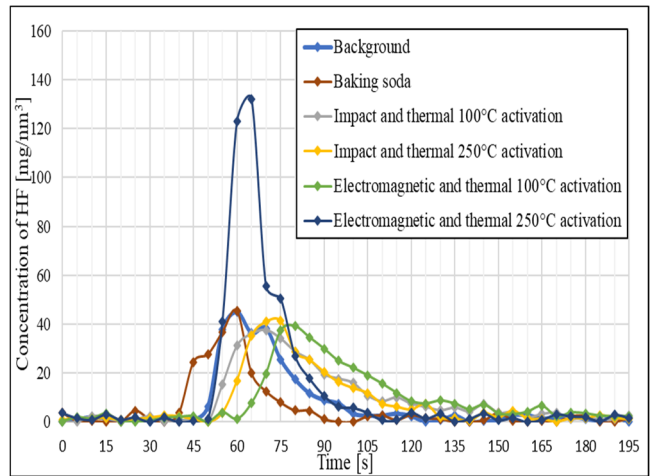


Fig. 14. HF concentration during testing as a function of time for thermally activated sorbents

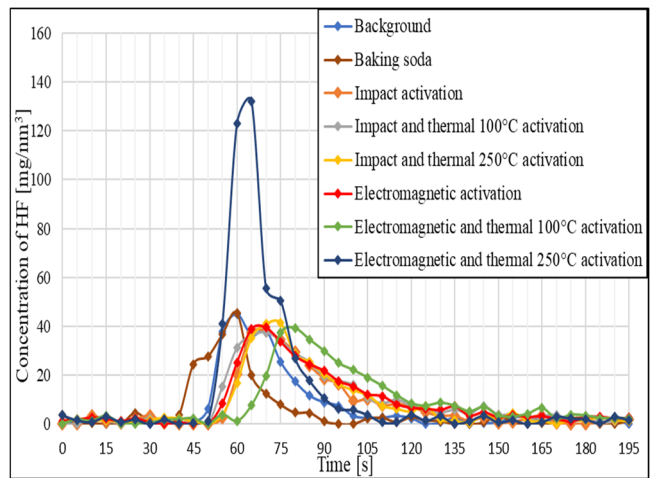


Fig. 15. HF concentration during testing as a function of time for all sorbents

#### 4. Conclusions

Using sodium sorbents allowed for a better flue gas cleaning efficiency from HCl and HF. In the case of the reduction of hydrogen chloride concentration, introducing unmodified baking soda with the largest average grain size of  $126 \mu\text{m}$  improved the flue gas cleaning efficiency by about 30%. The introduction of mechanically and thermally activated sodium sorbents with a much smaller grain size and better-formed pores, increasing the specific surface area of the sorbents, improved this effect approximately twice.



No significant reduction in hydrogen fluoride concentration was observed during an attempt to reduce HF concentration using baking soda. Using the modified sodium sorbents allowed flue gas purification from HF by about 15%. Mechanically and thermally activated sorbents show the development of structural parameters in relation to the base sodium bicarbonate. The specific surface area, as well as the total pore volume and porosity, increased significantly. At the same time, the density decreased, which positively affected the reactivity of the sorbent and, thus, the purification of the flue gas.

Almost all the studied sorbents were allowed to obtain better efficiency of flue gas cleaning from HCl and HF. The only exception was sorbent electromagnetically and thermally activated at 250°C, the use of which caused a threefold increase in the concentration of HF. The presented charts show that the efficiency of the sorbents characterised by the largest specific surface areas, which, according to the theory, were supposed to provide the best results in reducing HCl and HF concentrations, started to drop.

In the case of the impact-activated sorbent and thermally activated at the above-mentioned temperature, the reduction of hydrogen chloride and hydrogen fluoride concentrations was slightly worse than that of the material activated at 100°C, which had about three times lower specific surface area.

In the case of sorbent activated in an electromagnetic mill, flue gas purification efficiency deterioration started with the material heated at 100°C. It intensified during heating at 250°C despite an almost twofold improvement of its specific surface area.

The deterioration of flue gas purification efficiency was most likely the result of the occurrence of sintering pores on the surface of grains of materials after their introduction into hot flue gases at a temperature of 300°C. However, from the results obtained, it can be clearly stated that the process of deterioration of the material's porous structure occurred to a much greater extent in the sorbent with the most developed specific surface area.

From the point of view of process optimisation, the results obtained will contribute to the identification of optimal operating conditions in dry sorbent injection systems. The preparation of sorption materials must consider the optimum development of the specific surface area to prevent the pores on the surface of the sorbent from clogging when injected into hot flue gases.

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