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Management of surgical mask waste to activated carbons for CO₂ capture

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ABSTRACT

The worldwide COVID-19 pandemic has resulted in a huge amount of face masks being used up and thrown away, resulting in increased environmental pollution and infection risks. In our work, we have developed a highly efficient process of neutralizing face mask waste into a useful carbon material. Then, the prepared activated carbon was used for CO₂ adsorption studies. A series of activated carbons from face masks used as a precursor were synthesized using KOH and the activation temperature was in the range of 600–800 °C. All materials were characterized by well-developed porosity. The influence of activation temperature on the textural properties of prepared activated carbons and their adsorption abilities were investigated. The highest CO₂ adsorption was achieved for M₈₀₀ carbon and it was 3.91 mmol/g at the temperature of 0 °C and the pressure of 1 bar. M₈₀₀ carbon exhibited also high selectivity of CO₂ over N₂. Seven equilibrium isotherms were applied to the experimental data to find out the best fit (Langmuir, Freundlich, Sips, Toth, Unilan, Fritz-Schlunder and Radke-Prausnitz isotherms). The presented research provides an environmentally friendly and cost-effective method of recycling waste masks into a valuable product in the form of carbon and its potential use in the absorption of harmful CO₂ influencing the greenhouse effect.

1. Introduction

Nowadays, one of the huge problems which should be resolved is global warming. Carbon dioxide is the primary greenhouse gas emitted as a result of human activities. For CO₂ adsorption, various porous materials are used, including zeolites [1], modified TiO₂ [2], metal organic frameworks MOF [3], carbon spheres [4] or just activated carbons, which are particularly important because they have a very large specific surface, well-developed microporosity, often defined pore morphology, remarkable chemical, thermal properties and good affinity resistance to various types of pollutants [5].

Zeolites are highly ordered microporous crystalline materials. A number of their properties such as pore size and architecture or chemical composition affect their adsorption performance. They were intensively investigated as quite high CO₂ adsorption was achieved [6]. In terms of practical applications, moisture is a big problem to zeolite based adsorbents-H₂O is an important component of flue gas and some other industrial gases, and it competes with CO₂. From a thermodynamic viewpoint, the adsorption of CO₂ in the presence of H₂O is not favored in zeolites [7]. In addition, in the presence of CO₂, the acidic conditions may cause dealumination of zeolite structures, leading to a partial or total destruction of the framework [8].

MOFs are network solids composed of metal ion or metal cluster vertices linked by organic spacers. Although MOFs have very high capacity at high pressures, at atmospheric pressures their capacity is lower as compared to other physical sorbents. Further research is needed to develop MOFs targeting key material properties such as stability, multicule applicability and competitive sorption [9]. In particular, the stability of a framework toward long-term exposure to water vapor is a critical issue in determining its suitability for CO₂ capture from flue gas [10].

Activated carbons have advantages over other CO₂ adsorbents because of their wide availability, low cost and high thermal stability [6]. Owing to their low cost, high surface area, high amenability to pore structure modification and surface functionalization, and relative ease of regeneration, carbon-based materials are considered to be some of the

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most promising adsorbents for capturing CO\textsubscript{2} [11]. In spite of the hydrophobic character of carbon-based adsorbents, their CO\textsubscript{2} adsorption ability is adversely affected by the presence of water vapor [12]. To decrease the cost of adsorbents, cheaper carbon resources such as biomass and waste can be selected. The application of surgical mask waste described in this paper is the way for waste valorization and for utilization of hazardous waste.

The characteristic property of carbon materials is that they have a well-developed internal structure consisting of pores of various sizes and shapes. Depending on the diameter length, these pores are divided into micropores, mesopores and macropores, which play significantly different roles in adsorption processes [13]. Thus, the porous structure of active carbons significantly affects the sorption properties and thus determines the possibilities of their use. A significant advantage of active carbons as adsorbents is the fact that their structure can be easily modified by using various raw materials, binders, carbonization or activation parameters, thanks to which it is possible to obtain several types of activated carbons made of the same raw material.

Currently, there are many methods of capturing and separating carbon dioxide from the flue gas stream, both used, implemented and still being developed. The most commonly used methods are absorption - chemisorption of CO\textsubscript{2} in amine solutions. However, these methods are becoming relatively too costly and ineffective. Among the more modern methods, it is worth mentioning adsorption and membrane technologies, CO\textsubscript{2} separation in the oxygen combustion technology or combustion in a chemical loop (chemical loo-ping). Adsorption methods seem to be particularly promising due to relatively low operating costs, high levels of recovery and purity of the captured carbon dioxide. Among the adsorption methods, several techniques can be listed, due to the method of regeneration of the adsorbent bed, such as [14-19]: TSA - variable temperature adsorption; PSA - pressure swing adsorption, PTSA - pressure swing and temperature swing adsorption; VPSA - pressure swing adsorption with the use of vacuum for regeneration ration; ESA - adsorption with the use of low voltage electric current; RPSA - pressure swing adsorption with a fast pressure jump; URPSA - pressure swing adsorption with an ultrafast pressure jump. The PSA, PTSA and VPSA methods seem to be the most promising, due to the degree of research advancement and the estimated costs of adsorbent regeneration.

Fine particles with large surface area and unique properties are very attractive for many applications. The control of their size, shape, consistency, and composition is necessary and important to ensure their specific commercial applications and meet application requirements [20]. Therefore, recently there has been an increased interest in the production of specific adsorbent materials, characterized by fine or ultrafine particles. As Ragantti and Ammendola [21] mention, these materials can quite easily serve as a substrate for the production of specific adsorbent materials, characterized by fine or ultrafine particles, and composition is necessary and important to ensure their attractiveness for many applications. The control of their size, shape, consistency, and activity on the properties of carbons obtained was systematically investigated. In addition, the influence of the activation temperature and the type of waste on the properties of carbons obtained was systematically investigated. Moreover, the adsorption equilibrium data for the best CO\textsubscript{2} adsorbents were also presented and fitted to Langmuir, Freundlich, Sips, Temkin and Pyzhev, as well as Dubinin-Radushkevich equations. The obtained data can be useful for the design of CO\textsubscript{2} separation process.

2. Materials and methods

2.1. Preparation of activated carbon from a disposable mask

The precursor to the production of activated carbons was disposable face masks manufactured by AP PROPERTY S.A. (Poland). According to the literature the average carbon content of the precursor material – surgical mask was 75.9% [41,42]. A saturated KOH solution was used as the chemical activating agent. After elimination of the wire and rubber strip the major part of face mask has been cut into pieces and treated with an activator at ambient temperature for 3 h. The mass ratio of KOH:
precursor was 1:1. After drying, the material was ground. Carbonization coupled with activation was performed in a tube furnace using temperatures ranging from 600 to 800 °C under nitrogen flow. The sample was heated at a rate of 10 °C min⁻¹ to reach the processing temperature. That temperature was maintained for 1 h. After cooling, the resulting product was washed with distilled water, then with 1 mol/dm³ hydrochloric acid and again with distilled water until the pH was neutral. The resulting material was finally dried in an oven at 200 °C overnight. The obtained carbon (AC) from disposable face masks was named as: M (mask) T (temperature). The temperature was initially changed every 100 °C. After analysis of textural properties it was found that the activation should be carried out every 50 °C.

2.2. Characterisation of carbon materials

The X-ray fluorescence energy dispersion spectrometer (EDXRF) Epsilon3 type (Panalytical, Almelo, The Netherlands, 2011) was used to determine the metal content in the carbon materials.

The textural properties of activated carbons were determined by nitrogen adsorption/desorption at -196 °C. This characterization was carried out on an automated volumetric apparatus Quadrasorb Evo™ Gas Sorption analyzer (Anton Paar, St Albans, UK; previously Quantachrome Instruments, USA, 2014). All samples were out-gassed at 250 °C overnight prior to the measurements. The porous structure was determined from the N₂ sorption isotherms at the temperature of -196 °C. The specific surface areas (S_BET) were calculated using the Brunauer–Emmett–Teller (BET) equation using a relative pressure of 0.05–0.20. The total pore volume (V_mic) was calculated from the nitrogen amount adsorbed at a relative pressure, p / p_0 = 0.99. The micropore volume (V_mic) was determined using the DFT method (density functional theory).

CO₂ adsorption at temperature 0 °C was used to measure micropores with smaller diameters (0.3–1.47 nm). The pore size distribution was determined by the DFT method using the NLDFT model for CO₂ adsorption. The volume of micropores determined from CO₂ measurements was marked as V_mic, CO₂.

The X-ray diffraction (XRD) patterns of the carbon from disposable masks were recorded with an X-ray diffractometer (X'Pert–PRO, Panalytical, Almelo, The Netherlands, 2012) using Cu Kα (λ = 0.154 nm) as the radiation source in the 2θ range 10–80° with a step size of 0.026°.

Scanning electron microscopy (SEM) images of the sample morphology and feature of the samples were performed on a Zeiss Neon 40 EsB and a Zeiss CrossBeam instrument (Carl Zeiss SMT GmbH, Oberchoken, Germany, 2009) at 5.0 kV equipped with a field emission source.

Raman spectroscopy was used to determine the structure of the carbon skeleton of the obtained carbon materials. Raman analysis was recorded on an InVia Raman Microscope (Renishaw PLC, New Mills, Wotton-under-Edge, UK, 2007) with a laser wavelength of 785 nm. The spectrum obtained in the Raman shift range from 800 cm⁻¹ to 2000 cm⁻¹ was analysed. After normalizing the maximum of the G peak to 1, the surface coverage was determined based on the isotherms exhibiting the best fit at different temperatures, utilizing the Clausius-Clapeyron equation:

\[
\frac{\partial \ln (p)}{\partial \frac{1}{T}} = \frac{Q_i}{R} 
\]

where:

- \( Q_i \) – isosteric heat of adsorption [kJ/mol].
- \( R \) – gas constant [kJ/mol K].
- \( \theta \) – degree of surface coverage.
- \( T \) – temperature [K].

In order to determine the isosteric heat of adsorption for the tested materials, the function \( \ln (p) = f (1/T) \) was used for a given degree of surface coverage in the range from 0.005 to 0.035.

3. Results

3.1. Characteristics of the carbons obtained from protective mask

The XRD method is usually used to identify the crystallographic characteristics of materials. Fig. 1 shows the XRD patterns of the carbons.

The diffraction profiles exhibit only two broad bands around 2θ = 23° and 43°, which are associated with diffraction of the (002) and (10) planes, respectively. The (002) denotes the average crystallite thickness and (01) the average graphene sheet diameter [31]. The absence of other signals proved that the activated carbons did not contain any impurities. The broad peaks revealed pre-dominantly an amorphous carbon structure. Similar results were also reported by others [44,45].

The Raman spectra of the carbons in the range of 800–2000 cm⁻¹ are shown in Fig. 2.

The Raman spectra of the carbons were dominated by relatively
sharp D and G bands. The G band was always present for each carbon material, while with an increasing disorder in the graphite structure, another band appeared, denoted as the D band. The tested carbons obtained from masks had two characteristic bands. In the frequency range, approx. 1600 cm\(^{-1}\) (band G) and approx. 1305 cm\(^{-1}\) (band D). The D band could be attributed to the in-plane carbon-carbon stretching vibrations of graphite layers, and the D band was thought to have arisen from the structural imperfection of graphite. The intensity D band was proportional to the number of defects \[31\]. It was noticed that the D band was more intense than the G band, which confirms the poor ordering of the carbon structure and is related to the existence of numerous defects in the structure. Determining the correlation between the location of the G and D bands and their intensity (I\(_G\)/I\(_D\)) allows to characterize the structural properties of carbon materials. The ratio between the D and G band intensities (I\(_G\)/I\(_D\)) indicates the graphitization degree of the material. An increase in the I\(_G\)/I\(_D\) peak intensity ratio indicates a higher degree of carbon graphitization \[46\].

The values of I\(_G\)/I\(_D\) ratios obtained from Raman spectroscopy are shown in Table 2.

On the basis of Raman spectra normalized to the G band, I\(_G\)/I\(_D\) values were estimated. The values of the I\(_G\)/I\(_D\) ratios for all tested carbons are similar to one another and are in the range from 0.67 to 0.77. An increase in the I\(_G\)/I\(_D\) ratio indicates a higher ordering associated with the structural disturbance in these carbons. According to values of the I\(_G\)/I\(_D\) ratio mentioned in Table 2, the M\(_{600}\) (I\(_G\)/I\(_D\) = 0.77) is more ordered than the M\(_{800}\) (I\(_G\)/I\(_D\) = 0.67). The influence of the temperature on the I\(_G\)/I\(_D\) ratio depends on the type of carbon material. Liu et al. \[47\] produced carbon materials from Huolinhe lignite using two different methods. The I\(_G\)/I\(_D\) ratio increased with the temperature for the first method and decreased for the second. The influence of KOH: lumpy bracket mass ratio and the temperature on the I\(_G\)/I\(_D\) ratio was described by Serafin et al. \[31\]. The values of I\(_G\)/I\(_D\) for activated biocarbons were in the range of 0.52–0.76. The higher I\(_G\)/I\(_D\) values were observed in samples obtained at lower and higher temperatures and using the lowest and the highest impregnation ratios of KOH to lumpy bracket. The I\(_G\)/I\(_D\)

### Table 1

| Isotherm model     | Equation                                                                 | Parameters of the isotherm |
|-------------------|--------------------------------------------------------------------------|----------------------------|
| Langmuir          | \( q = \frac{q_{mL} b_s p}{1 + (b_s p)^2} \)                             | \( q_{mL} \) – maximum adsorption on the sorbent surface, [mmol/g] |
| Freundlich        | \( q = k_F p^{m_F} \)                                                   | \( k_F \) – Freundlich constant, [mmol/g] |
| Sips              | \( q = \frac{q_{mS} b_s p^{m_S}}{1 + b_s p^{m_S}} \)                     | \( q_{mS} \) – maximum adsorption capacity, [mmol/g] |
| Toth              | \( q = \frac{q_{mT} b_s p^{m_T}}{1 + b_s p^{m_T}} \)                     | \( q_{mT} \) – maximum adsorption capacity, [mmol/g] |
| Fritz-Schlunder   | \( q = \frac{q_{mFS} b_s p^{m_FS}}{1 + b_s p^{m_FS}} \)                  | \( q_{mFS} \) – Fritz-Schlunder constant, [bar\(^{-1}\)] |
| Sips              | \( q = \frac{q_{mS} b_s p^{m_S}}{1 + b_s p^{m_S}} \)                     | \( q_{mS} \) – maximum adsorption capacity, [mmol/g] |
| Radke-Prausnitz   | \( q = \frac{q_{mRP} b_s p^{m_RP}}{1 + b_s p^{m_RP}} \)                  | \( q_{mRP} \) – Radke-Prausnitz constant, [bar\(^{-1}\)] |
| Fritz-Schlunder   | \( q = \frac{q_{mFS} b_s p^{m_FS}}{1 + b_s p^{m_FS}} \)                  | \( q_{mFS} \) – Fritz-Schlunder model exponent |

### Table 2

| Carbon  | I\(_G\)/I\(_D\) |
|---------|-----------------|
| M\(_{600}\) | 0.77            |
| M\(_{650}\) | 0.76            |
| M\(_{700}\) | 0.70            |
| M\(_{750}\) | 0.72            |
| M\(_{800}\) | 0.67            |

Fig. 1. XRD patterns of carbons from protective masks.

Fig. 2. Raman spectra of carbons from protective masks.
values decreased in the temperature range of 600–850 °C but for 900 °C IG/ID value was similar to Ic/Ib obtained for 600 °C. The surgical mask waste activated carbons were synthetized at the temperature range of 600–800 °C so the decrease of Ic/Ib values is similar to that described by J. Serafin, et al. [31].

Carbon analysis by Raman spectroscopy confirmed the results of XRD studies. The XRD spectra of activated carbons showed that all of them had a nearly amorphous carbon structure. However, on the basis of Scherrer equation the average crystallite thickness and average graphene sheet diameter can be approximately calculated. For the dimension of turbostratic crystallites perpendicular to the graphene sheets, (002) peak data were used. For the dimension in graphene sheet planes, (10) peak data were used. The brighter the peaks, the lower the values. The values were not calculated. They were determined on the basis of the width of the peaks at half height, standing for the degree of graphitization decreasing with the temperature.

The textural properties of the obtained materials were analysed based on the N2 adsorption-desorption studies at −196 °C on carbons (Fig. 2).

The N2 adsorption-desorption isotherms were classified according to the IUPAC as type 1 and 4 isotherms, suggesting that the carbon structure is substantially microporous with the influence of the mesoporous structure [48]. All the isotherms showed a type H3 hysteresis loop. Fig. 4 shows the influence of the activation temperature on the specific surface area, the total pore volume and the volume of micropores determined from N2 adsorption at −196 °C and CO2 adsorption at 0 °C for the carbons.

The Brunauer-Emmett-Teller (BET) surface areas shown in Fig. 4a were quite unique, and the highest value of 969 m²/g was obtained for the carbon M_750, the activation temperature of which was 750 °C. The surface areas in order of the largest to the smallest were M_750 > M_700 > M_800 > M_650 and M_600. The total pore volume was calculated assuming the total N2 under p/p0 = 0.99 and it was found that the highest Vtot value was also obtained for the carbon M_750 and it was 0.635 cm³/g (Fig. 4b). As a result of the analysis of Fig. 4c and Fig. 4d it was found that the volume of micropores with diameters in the range 1.4–2 nm and the volume of micropores (except M_700 carbon) showed an increasing trend with increasing activation temperature.

Fig. 5 shows the distribution of pores with diameters of 0.3–1.4 nm determined with the DFT method based on CO2 adsorption at 0 °C. Fig. 6 presents the same materials and the distribution of pore sizes determined with the DFT method from N2 adsorption measurements at −196 °C. The pore spreading and porous size distribution of the samples (Figs. 5 and 6) were narrowly distributed within the micro-mesopore range.

Traditionally, the pore size distribution is evaluated from the analysis of nitrogen sorption isotherms measured at 77 K. At such a low temperature diffusion of nitrogen molecules into small micropores is very slow and pores below 1 nm cannot be estimated. This problem can be eliminated by using CO2 adsorption at 0 °C. The saturation pressure of CO2 at 0 °C is very high (26141 torr). Therefore low relative pressure measurements necessary for the small micropore analysis are achieved in the range of moderate absolute pressures. At higher temperature and under higher absolute pressures CO2 molecules can more easily access ultramicropores than N2 at 77 K in spite of the fact that molecular critical dimensions of both gases are similar.

On the basis of nitrogen sorption, pore volume of pores with diameter in the range of about 1.1–100 nm can be estimated. Carbon dioxide sorption gives information concerning the pores with diameter in the range of about 0.3–1.1 nm.

The estimation of small micropores (below 1.2 nm) using CO2 adsorption at 0 °C is a common method described and has been utilized by many authors [29,49–51].

In Fig. 5 three particularly intense peaks can be observed at pore diameters of 0.35–0.4 nm, 0.4–0.7 nm, and 0.7–1 nm. These peaks occurred in all carbons regardless of the activation temperature. They show the share of narrow micropores in the activated carbons, the largest share of which was observed in the sample carbonized at the temperature of 800 °C. The micropore volume for sample M_800 determined using carbon dioxide was 0.327 cm³/g. The curves presenting the pore size distribution of narrow micropores seemed similar but the total narrow pores volumes were different. Moreover the big differences were observed for the pores with the diameter of about 0.8 nm (0.7–1 nm).

The analysed activated carbons in their structure, apart from narrow micropores determined with the use of CO2 at 0 °C, also contained micropores with a size of 1.4–2 nm and mesopores as shown in.

Fig. 6. A significant increase in mesopores in the pore range from 2 nm to 5 nm was found for samples M_700 and M_750 compared to other materials where such a large increase in the proportion of mesopores was not observed. The participation of mesopores in the tested materials is visible up to the pore width range of 12 nm, as evidenced by the data presented in Fig. 6.

Particle size for activated carbons was measured. Fig. 7 shows the particle size distribution of activated carbons.

Based on the particle size distribution, activated carbons thus produced were mostly 20–60 µm in diameter with more than 80% of particles of size less than 60 µm. The finer the particle size of an activated carbon, the better the access to the surface area [52].

The above results showed that the temperature of the activation process had a strong effect on the texture of carbons. The carbons with KOH as the activator showed higher microporosity content and higher surface area due to intercalation of active K molecules with carbon lattices and carbon matrix than when using other activators [53–56].

Fig. 8 shows pictures taken with a scanning electron microscope at a magnification of 9,000 magnification in order to observe the surface topography of the materials.

According to the SEM micrograph, the ACs have a highly heterogeneous surface and a wider irregularity. No difference in the morphology of activated carbons prepared at different temperatures was observed. They seemed to be very similar.

The TEM pictures were very similar (Fig. 9) and we were not able to judge on the basis of TEM investigation which activated carbon was more porous. The nitrogen adsorption (Fig. 4) gave more information about porosity. However, TEM investigations confirmed the porosity of the samples.

3.2. CO2 adsorption studies

Fig. 10 shows the results of CO2 adsorption studies. The data presented in Fig. 10 can be used to compare activated carbon samples that were tested up to a pressure of 1 bar at temperatures of 0 °C, 10 °C and
Changing the process temperature affected the amount adsorbed in each sample. For all carbons, the adsorption capacity increased with increasing pressure and decreased with increasing temperature, indicating the adsorption was exothermic in behavior which was expected for physical adsorption [57]. The lower CO$_2$ adsorption capacity resulted mainly from the reduction of interaction forces and binding forces between the adsorbate and the adsorbent [58]. Moreover, this tendency confirms an increase in the energy of CO$_2$ molecules at a higher temperature, which leads to lower adsorption of CO$_2$ by AC [59]. All CO$_2$ isotherms conform to the type I isotherm according to the IUPAC isotherm classification [60]. For carbons prepared from masks, CO$_2$ adsorption isotherms in the range temperatures of 0–20 °C show a similar course, rapidly rising at low pressures and slowing at higher temperatures.
pressures. These isothermal similarities indicate that the adsorption mechanism was the same for all tested activated carbon samples. Temperature change affected the amount adsorbed in each sample, and CO$_2$ uptake decreased with increasing temperature. On the basis of Fig. 10, it was found that the carbon synthesized at 800 $^\circ$C had the highest CO$_2$ adsorption capacity at a pressure of 1 bar and a temperature of 0 $^\circ$C. This adsorption capacity was 3.91 mmol/g at 0 $^\circ$C, whereas at 10 $^\circ$C it was 3.23 mmol/g, and at 20 $^\circ$C it was 2.61 mmol/g. The CO$_2$ adsorption increased with the temperature of the carbonization but at the temperature of 850 $^\circ$C the yield of the carbonization was very low and we could not produce activated carbons at temperatures higher than 800 $^\circ$C.

The Table 3 summarizes the results of CO$_2$ adsorption on activated carbons produced from various carbon precursors. Although the result we obtained for the CO$_2$ adsorption was not the best, it could still be competitive compared to other materials.

In order to analyse how the textural parameters influence the adsorption of CO$_2$ at 0 $^\circ$C, 10 $^\circ$C and 20 $^\circ$C at 1 bar, the relationships between them and CO$_2$ sorption were investigated (Fig. 11).

Fig. 11 shows the effect of the specific surface area (Fig. 11a), the total pore volume (Fig. 11b), the micropore volume determined from the N$_2$ measurements at $-196$ $^\circ$C (Fig. 11c) the volume of the micropores.

Fig. 7. Particle size distribution of activated carbons.

Fig. 8. SEM micrographs of the carbons from protective masks (a) M_600 (b) M_650 (c) M_700 (d) M_750 (e) M_800.
Fig. 9. TEM micrographs of the carbons from protective masks (a) M_600 (b) M_650 (c) M_700 (d) M_750 (e) M_800.
The coefficient of determination $R^2$ is the proportion of the variance in the dependent variable that is predictable from the independent variable. An $R^2$ of 1 means the dependent variable can be predicted without error from the independent variable. A low $R^2$ value indicates no dependency between the variables. On the basis of the $R^2$ values presented in Fig. 11 conclusions were drawn about the influence of $S_{BET}$, $V_{tot}$, $V_{mic N_2}$, $V_{mic CO_2}$ on CO$_2$ adsorption.

There was no correlation between CO$_2$ uptake and the specific surface area and the total pore volume. Moreover, the relevant dependence between CO$_2$ adsorption and micropore volume ($V_{mic N_2}$) was not evidenced. However, the best correlation was obtained between CO$_2$ adsorption and the volume of micropores determined from CO$_2$ measurements at 0 °C. Based on Fig. 5, it was concluded that the carbonaceous materials contained micropores in the pore range from 0.4 to 1 nm which were determined on the basis of CO$_2$ adsorption measurements. Thus, the high CO$_2$ uptake was conditioned by the micropore with the diameter from above-mentioned range.

The effective micropores were mainly responsible for CO$_2$ adsorption on the well-developed activated carbons via a micropore filling mechanism [72]. This was due to the fact that CO$_2$ particles were captured by micropores [73] and the amount of CO$_2$ adsorbed on activated carbons related primarily to narrow micropore volumes [74]. Although the M_750 carbon showed a more developed specific surface than other samples, it did not have the highest CO$_2$ adsorption capacity compared to other carbons. The sample M_800, which showed the greatest development of micropores volume in the range 0.4–1 nm, also showed the greatest adsorption of CO$_2$ at temperatures 0, 10 and 20 °C (Fig. 10).

On the basis of Figs. 5 and 11, the assumption was made that pores with the diameter of about 0.8 nm (0.7–1 nm) are the most important for CO$_2$ adsorption at the pressure of 1 bar.

The correlation of textural parameters and CO$_2$ adsorption at 1 bar was investigated by other authors. Martín et al. [49] showed that CO$_2$ adsorption at 1 bar and the temperature of 25 °C correlated well with narrow microporosity estimated using CO$_2$ adsorption at 0 °C. Although the M_750 carbon showed a more developed specific surface than other samples, it did not have the highest CO$_2$ adsorption capacity compared to other carbons. The sample M_800, which showed the greatest development of micropores volume in the range 0.4–1 nm, also showed the greatest adsorption of CO$_2$ at temperatures 0, 10 and 20 °C (Fig. 10). On the basis of Figs. 5 and 11, the assumption was made that pores with the diameter of about 0.8 nm (0.7–1 nm) are the most important for CO$_2$ adsorption at the pressure of 1 bar.

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grand canonical Monte Carlo (GCMC) and the nonlocal density functional theory [76] that only pores of less than 1 nm can be effective for CO$_2$ adsorption at atmospheric pressure. Wickramaratne et al. [50], Grundy et al. [29], Li et al. [51] proved that CO$_2$ adsorption at 0 $^\circ$C and 1 bar depended on the micropore smaller than 1 nm. These authors utilized CO$_2$ as the adsorbate, temperature of 0 $^\circ$C and p/p$_0$ up to 0.3 for small micropore volume and distribution.

The re-usability of M$_{800}$ as a CO$_2$ sorbent was examined. Twenty adsorption–desorption cycles at a temperature of 0 $^\circ$C were performed. Fig. 12 presents 1st, 10th, 20th adsorption isotherms. No changes in the CO$_2$ adsorption after twenty cycles were found. The highest standard deviation was equal to 0.06. The reproducibility and repeatability of CO$_2$ adsorption on M$_{800}$ activated carbon was proved. M$_{800}$ was a stable CO$_2$ sorbent and it could be regenerated without any loss of its CO$_2$ sorption capacity.

### 3.2.1. Fitting results

The experimental values of CO$_2$ adsorption isotherms presented in Fig. 10 were analysed using seven equations: Langmuir, Freundlich, Sips, Toth, Unilan, Radke-Prausniz and Fritz-Schlunder. Table 4 presents values of the SSE error of CO$_2$ adsorption isotherms for each analysed model. The lowest SSE values were found to determine which model was the best. Based on the squares of the errors (SSE), it was found that the Sips equation gave the highest accuracy of fitting to the experimental data. Using the Sips model, the lowest SSE values were obtained for each CO$_2$ adsorption isotherm at temperatures of 0 $^\circ$C, 10 $^\circ$C and 20 $^\circ$C of the tested carbons, as shown in Table 4.

Table 5 presents the determined parameters of the Sips isotherm for different temperatures of the CO$_2$ adsorption process.

The Sips model was the most suitable for fitting CO$_2$ adsorption on carbons from waste face masks. The Sips isotherm is a combination of the Langmuir and Freundlich isotherms. The difference between the Sips and the Langmuir equation is the heterogeneity factor $n_s$, usually lower than 1. The smaller the value of this parameter, the more heterogeneous the adsorbent surface. If $n_s$ is equal to 1, the Sips equation is reduced to the Langmuir equation and the surface is homogeneous. The values of $n_s$ in the temperature from 0 to 20 $^\circ$C were in the range of 0.663–0.792.

---

**Fig. 11.** The CO$_2$ adsorption at the pressure of 1 bar and at $\blacklozenge$ 0 $^\circ$C, $\blacklozenge$10 $^\circ$C and $\blacktriangledown$ 20 $^\circ$C temperature as a function of (a) $S_{BET}$ (b) $V_{tot}$ (c) $V_{mic\text{ N}_2}$ (d) $V_{mic\text{ CO}_2}$.

**Fig. 12.** Multi-cycle CO$_2$ adsorption isotherms for M$_{800}$ at 0 $^\circ$C, 1st, 10th, 20th adsorption isotherm.
This proves the heterogeneity of the activated carbon surface.

Parameters in the Sips equation such as \( b_s \), \( n_s \), and \( q_{rms} \) are temperature-dependent:

\[
b_s = b_{0s} \times \exp \left( \frac{Q_s}{R \times T_0 \times \left( T - T_0 \right)} \right) ;
\]

\[
q_{ms} = q_{rms} \times \exp \left( \frac{\gamma}{1 + \frac{T}{T_0}} \right) .
\]

In the above equations, \( b_{0s} \) – adsorption affinity, \( n_s \) – heterogeneity coefficient, \( q_{rms} \) – maximum adsorption capacity, \( \gamma \) and \( \alpha \) are constants, \( R \) is the gas constant and \( T_0 \) is the reference temperature, which was assumed in the study to be 0 °C (the lowest process temperature), and \( Q_s \) is the isosteric heat of adsorption with the coverage degree tending to zero. These parameters were determined using graphs showing the dependencies of \( \ln(b_s) \) from \( 1/T \), from \( 1/T \) and \( \ln(q_{rms}) \) from \( T \). The exemplary graphs for the M_750 carbon are shown in Fig. 13.

High values of \( R^2 \) were obtained, which proves a good fit of these dependencies to the linear function. This confirms the correctness of the performed calculations related to the determination of the parameters of the Sips model at the tested temperatures of CO_2 adsorption. The determined temperature-dependent parameters in the Sips equation are presented in Table 6.

### 3.2.2. Heat of adsorption

The isosteric heat of adsorption is important to characterize the interaction between the adsorbent and the adsorbate. A higher value of isosteric heat of adsorption proves a stronger interaction between carbon dioxide and the tested carbons. The isosteric heat of adsorption was calculated from the experimental data obtained at 0–20 °C using the Sips temperature equations and the linear form of Clausius-Clapeyron equation

\[
\ln(p) = - \frac{Q_s}{R} \left( \frac{1}{T} \right) + C
\]

where:

- \( C \) is a constant.
- \( \theta \) – surface coverage.
- \( Q_s \) – isosteric heat of adsorption [J mol^{-1}].
- \( R \) – ideal gas constant [m^3 Pa K^{-1} mol^{-1}].
- \( T \) – temperature [K].

First, the experimental isotherms were fitted to Sips equations to calculate numerically \( \theta \) for a given \( p \) at different temperatures. The isosteres were plotted in coordinates \( \ln(p) \) vs. \( 1/T \) for seven surface coverage (\( \theta = 0.005, 0.010, 0.015, 0.020, 0.025, 0.30, 0.035 \)) and pre-

![Graph showing isosteric heat of adsorption (Qs) vs. 1/T for seven surface coverage](image)

Presented at the Fig. 14. The isosteres gave the information about the energetic homogeneity of the surface in question. At each given \( \theta \), the isostere plot of \( \ln(p) \) as function of \( 1/T \) matches with the linear form of Clausius-Clapeyron equation. Therefore, the isosteric head of adsorption values were calculated from the slopes of the isostere plot:

\[
Q_s = - \text{slope} \times R
\]

The linear fit of all \( f(1/T) = \ln(p) \) showed that the derivative

\[
\left( \frac{d\ln(p)}{d(1/T)} \right)_\theta \text{ was independent from the temperature but the influence of the surface coverage was observed in the temperature range of 0–20 °C. This indicates the homogeneity of the adsorbent surface [54].}

Fig. 15 shows the determined values of the isosteric heat of CO_2 adsorption on the carbon as a function of the degree of surface coverage. The determined values of the isosteric heat of adsorption for carbons did not show any significant variability and ranged from 19 to 25 kJ/mol. These values strongly confirm the physical nature of CO_2 sorption on the carbon materials from the masks. As the degree of surface coverage increased, the isosteric heat of adsorption decreased. The isosteric heat of adsorption decreased as there was less and less interaction of CO_2 with the material. In a common adsorption process, the adsorptive becomes attached to the surface of the adsorbent and forms the adsorbate. If the heat of adsorption of the first layer is comparable, usually slightly lower than the heat of the subsequent layers, it means that physical sorption took place. Therefore, we can hypothesize about physisorption of CO_2 on activated carbons produced from face mask. The values of isosteric adsorption heat determined on the basis of Clausius-Clapeyron equation are consistent with the values of the heat
The isosteric heats of CO$_2$ adsorption are very similar for all activated carbons. The lowest values were obtained for M$_{600}$ and they ranged from 22 to 19 kJ/mol. The highest values of isosteric heats of CO$_2$ adsorption were observed for M$_{800}$ and they ranged from 25 to 21 kJ/mol. The higher heat for M$_{800}$ proved the higher interaction between CO$_2$ and surface of this material. The isosteric heat of adsorption becomes constant at high coverage for M$_{800}$ suggesting a uniform CO$_2$ adsorption on this material from surface coverage of 0.02.

Selectivity adsorption of CO$_2$ over N$_2$ is a significant factor that ought to be taken into account in evaluation of the sorbents for CO$_2$ adsorption. Therefore, nitrogen adsorption measurement at 20°C up to a pressure of 1 bar was performed for the sample M$_{800}$ which had the best CO$_2$ adsorption.

Ideal adsorbed solution theory (IAST) proposed by Myers and Prausnitz [79] is widely used for predicting the adsorption selectivity and mixed-gas adsorption isotherms from pure-component isotherms with a rational accuracy for various systems. In this work, IAST was employed to predict selectivity of CO$_2$ over N$_2$ using single adsorption isotherms of these gases for equimolar CO$_2$ and N$_2$ binary mixture ($S_{EQM}$) according to the following equation [80]:

$$S_{EQM} = \frac{q_{CO_2}}{q_{N_2}}$$

where: $q_{i\text{p}}$ is the adsorption capacity [mmol/g] at the same partial pressure $p$ of CO$_2$ and N$_2$.

Typical flue gas composition in vol% is: 67–72% nitrogen, 8–10% carbon dioxide, 18–20% water steam, 2–3% oxygen for natural gas-fired power plants and 72–77% nitrogen, 12–14% carbon dioxide, 8–10% water steam, 3–5% oxygen for coal-fired boilers [56].

Selectivity for flue gas composition 15% CO$_2$ and 85% N$_2$ ($S_{FG}$) was calculated also on the basis of IAST method using the below equation [81]:

$$S_{FG} = \frac{q_{CO_2\text{p}}}{q_{N_2\text{p}}} \times 0.85 \div 0.15$$

where:

$q_{i\text{p}}$ is the adsorption capacity [mmol/g] of CO$_2$ and N$_2$ at the partial pressure $p$ of CO$_2$ and N$_2$.

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The selectivity was also calculated using the ratios of the Henry’s law constants ($S_{H}$). The Henry’s law constants were estimated from the initial slopes of CO$_2$ ($I_{CO_2}$) and N$_2$ ($I_{N_2}$) adsorption isotherms according to the equation [82]:

$$S_{H} = \frac{I_{CO_2}}{I_{N_2}}$$

### Table 6

| Carbon | $b_0$ [bar$^{-1}$] | $Q$ [kJ/mol] | $n_0$ | $\alpha$ | $\gamma$ | $q_{mo}$ [mmol/g] |
|--------|------------------|--------------|-------|----------|---------|-----------------|
| M$_{600}$ | 0.15 | 26 | 0.66 | 0.39 | 1.35 | 4.97 |
| M$_{650}$ | 0.13 | 27 | 0.68 | 0.37 | 1.68 | 5.88 |
| M$_{700}$ | 0.16 | 27 | 0.76 | 0.41 | 1.44 | 5.92 |
| M$_{750}$ | 0.18 | 28 | 0.70 | 0.42 | 1.51 | 4.99 |
| M$_{800}$ | 0.20 | 29 | 0.69 | 0.38 | 1.45 | 6.21 |

Fig. 13. Dependence of a) the natural logarithm of the bs coefficient of the reciprocal of temperature; b) the ns coefficient of the reciprocal of temperature; c) the natural logarithm of $q_{mo}$ from temperature.
Selectivity calculated using single component gas adsorption capacity for equimolar CO$_2$ and N$_2$ binary mixture is presented in Fig. 16. The CO$_2$/N$_2$ selectivity ratio for the M$_{800}$ carbon decreased to a pressure of about 0.2 bar. The highest CO$_2$/N$_2$ selectivity coefficient at 0.001 bar was 68, while with increasing pressure, the value of the selectivity coefficient decreased, reaching the value of 15 for a pressure of 1 bar. A similar tendency in the CO$_2$/N$_2$ selectivity ratio was noted by Serafin et al. [56].

Table 7 presents the selectivity of CO$_2$ over N$_2$ of activated carbons prepared from different precursors calculated using different methods at the temperature of 25$^\circ$C. The investigations presented in this study were performed at the temperature slightly lower (20$^\circ$C) but there is no much work about selectivity of CO$_2$ over N$_2$ and the most of them were performed at 25$^\circ$C.

Most activated carbons presented in Table 7 had lower selectivity for CO$_2$ over N$_2$ than the M$_{800}$ produced from surgical mask waste. The high selectivity is one of the important requirements of a good adsorbent.

4. Cost of producing activated carbon

The economic evaluation of production of activated carbons from
surgical masks was performed. Activated carbons from surgical masks were produced with chemical activation with potassium hydroxide. The preparation process is described in Section 2.1. On the basis of the obtained data, we made calculations on an industrial scale of production. The initial input of 10,000 kg delivered daily was assumed. The yield of carbon after carbonization was 40% (4000 kg), after cleaning and drying, the final yield was 35% (3500 kg).

The first stage of activated carbon preparation from disposable face masks is treatment with a saturated KOH solution for 3 h. It is well known that weak alkali solutions inactivate viruses. Jeong et al. [92] proved that influenza A virus H1N1 was completely inactivated within 1 min of 0.1 mol/dm³ NaOH treatment. The coronavirus and any other viruses will be completely destroyed in a saturated KOH solution in several seconds. The people who will put the surgical mask waste into the KOH solution should wear personal protective equipment (PPE) for the health workforce during COVID-19 (surgical masks, particulate filter respirators (such as P2 or N95), gloves, goggles, glasses, face shields, gowns and aprons). The cost of PPE is relatively low and there is no need to take it into consideration.

Surgical masks are cut into small pieces with a diameter of 10 mm using an 11 kW cutting machine. After grinding, the material is activated (3 h) with KOH in the ratio of 1 kg of material to 1 l of KOH at room temperature. A 4000 l storage tank is used for the activation process. The material is then transferred to the hopper of a 0.97 × 6.1 m rotary dryer using the parameters established by McCabe et al. [93]. To prevent rusting and reacting with potassium hydroxide, use a stainless steel dryer. The next step is to transfer the material to a 5 × 1.5 × 0.2 m horizontal tube furnace and carbonize it for 1 h at 700 °C under nitrogen atmosphere. After carbonization, the material should be transferred to a rotary cooler, which is suitable for operation at high temperatures. Assuming 4500 kg/day, a 7 m² rotary cooler will be required according to the calculations of McCabe et al. [93] to achieve a temperature drop from 700 °C to room temperature in less than 1 h. The cooled activated carbon should be washed with distilled water to remove residual potassium hydroxide. The washing process will take place in a reactor with a capacity of 8000 L, using 40 L of water per kilogram of carbon. Then, feed the material into an 8000 L reactor with 1 M hydrochloric acid, using 10 L of acid per kilogram of carbon. The water washing process is repeated to remove residual HCl in an 8000 L reactor using 40 L of water per kilogram of carbon. The final step is collecting the carbon on a dewatering sieve and transferring it to a rotary dryer. Assuming a process load of 5000 kg of wet carbon and using the data provided by McCabe et al. [93], a 1.24–2.15 m rotary dryer will be needed. Similarly as before, in order to avoid rusting, it is recommended to be made of stainless steel. Finally, the weight loss from screening is estimated to be up to 5%. Hence, the final weight of the resulting product is 3325 kg/day.

The production cost was calculated assuming 30% efficiency or 3325 kg/day of activated carbon production. The production time is 320 days/year in a three-shift system for 24 h with 3 employees and € 4.20 per hour. An investment of € 4.6 million is required for the activation of potassium hydroxide (Table 8). The annual production cost is estimated to be € 2.24 million (Table 9) with an annual production of 1.064.000 kg. The estimated cost per kilogram of activated carbon would be € 2.10 (Table 10). The project does not include energy recovery devices.

The estimated cost of producing activated carbon from surgical masks is 2.10 €/kg and may compete with other activated carbons obtained from waste materials. Ng et al. [95] obtained activated carbon based on pecan shells and estimated the cost of its production at € 2.34–2.49 per kilogram depending on the type of material activation. On the other hand, Lima et al. [96] prepared activated carbon from the body waste through physical activation and calculated that the cost of production at zero raw material price was € 1.24. Stavropoulos et al. [97] reported the production costs of activated carbon obtained from used tires, wood, and lignite to be 9.81 €/kg, 5.49 €/kg and 4.62 €/kg.
Table 8
Estimated capital costs.

| Equipment                                      | Cost (€) |
|------------------------------------------------|----------|
| Cutting machine                                | 6880     |
| Storage tank                                   | 68,800   |
| Rotary dryer                                   | 130,000  |
| Tube furnace                                   | 176,000  |
| Rotary cooler                                  | 85,000   |
| 2 tanks for water washing                      | 140,000  |
| 1 tank to acid washing                         | 70,000   |
| Rotary dryer                                   | 130,000  |
| Sieve                                          | 2600     |
| Total equipment cost                           | 809,280  |
| Equipment installation                         | 453,000  |
| Instrumentation                                | 349,000  |
| Material transport                             | 397,000  |
| Electrical installation                        | 250,000  |
| Buildings                                      | 250,000  |
| Yard improvements                              | 98,000   |
| Service                                        | 750,500  |
| Land                                           | 49,000   |
| Engineering and supervision                    | 501,400  |
| Construction expense                           | 599,000  |
| Contractor’s fee                               | 98,000   |
| Contingency                                    | 395,000  |
| Total capital costs                            | 4,602,180|

*Capital costs based on different percentages of the total equipment cost according to Peters and Timmerhaus [94].

Table 9
Annual operating costs.

| Item                                | Annual cost (€) |
|-------------------------------------|-----------------|
| Surgical masks                      | 30,000          |
| Potassium hydroxide                 | 220,000         |
| Nitrogen                            | 10,000          |
| Utilities                           |                 |
| Water                               | 30,000          |
| Electricity                         | 65,000          |
| Natural gas                         | 30,000          |
| Labor                               |                 |
| Operating labor                     | 325,000         |
| Maintenance labor                   | 100,000         |
| Supervision                         | 60,000          |
| Fringe benefits                     | 162,000         |
| Supplies                            |                 |
| Operating supplies                  | 40,000          |
| Maintenance supplies                | 54,000          |
| General works                       |                 |
| General and administrative          | 40,000          |
| Property insurance and tax          | 35,000          |
| Depreciation                        | 40,000          |
| Total cost                          | 2,241,000       |

*Annual operating costs for labor, supplies and general work are based on percentages given in Peters and Timmerhaus [94].

Table 10
Summary of costs.

| Cost                              | Value (€) |
|-----------------------------------|-----------|
| Purchased equipment cost          | 809,280   |
| Capital cost                      | 3,792,900 |
| Total fixed capital investment    | 4,602,180 |
| Total annual operating cost       | 2,241,000 |
| Estimated annual production of carbon | 1,528,000 kg |
| Estimated cost for activated carbon | 2.10 per kg |

5. Conclusions

The high-value-added activated carbons produced from disposable face masks have been investigated and described for the first time. The disposable face masks are waste associated with their increased use, especially during a viral epidemic. The obtained carbon was characterized in N₂ and CO₂ sorption investigations and with SEM, XRD, and Raman spectroscopy. XRD results revealed the amorphous nature of the samples, which was confirmed by Raman spectroscopy.

The carbons produced from masks possessed a high surface area and narrow pore distribution which made them a highly efficient candidate for CO₂ adsorbent. The highest CO₂ adsorption was observed on carbon produced at 800 °C. The adsorption of carbon dioxide of this sample at 1 bar was 3.91 mmol/g, 3.23 mmol/g and 2.61 mmol/g at 0 °C, 10 °C and 20 °C, respectively. CO₂ adsorption was most dependent on micropores in the range of 0.4–1 nm. The selectivity of separation for CO₂ over N₂ was calculated based on IAST for equimolar CO₂ and N₂ binary mixture, for flue gas composition and also using the ratios of the Henry’s law constants. The selectivity values on M_800 were the highest so far described in the literature. The overall results suggest that activated carbons produced from disposable face masks can be applied as potential adsorbents for CO₂ separation. Therefore, the approach presented here, which relies on carbonization combined with chemical activation, is an easy method using cheap carbon sources that could be a promising and practical alternative for CO₂ adsorption. That is why it is so important that used face masks could be easily and safely transformed into a full-value product, an effective adsorbent in many processes. Finally, our idea of managing waste masks can be an easy way of decreasing the amount of generated plastic waste.

CRediT authorship contribution statement

Jarosław Serafin: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review and editing, Visualization, supervision. Joanna Sreńszek-Nazzal: Conceptualization, Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review and editing, Visualization. Adrianna Kamińska: Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review and editing, Visualization. Oliwia Paszkiewicz: Investigation, Writing – original draft, Writing – review and editing. Beata Michal-kiewicz: Validation, Data curation, Writing – original draft, Writing – review and editing, Supervision. All authors have read and agreed to the published version of the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Conflicts of interest

The authors declare no conflict of interest.

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