engineering proceedings

MDPI

Proceeding Paper

Development and Synthesis of a Novel Carbon Dioxide-Capturing Polyacrylic Sorbent †

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† Presented at the 6th International Electronic Conference on Applied Sciences, 9–11 December 2025; Available online: https://sciforum.net/event/ASEC2025.

Abstract

The release of CO_2 gas into the atmosphere is one of the most prolific causes of global climate change. To solve this problem, cost-effective technologies are being sought. Polymer membranes are innovative materials that can be widely used in the process of capturing and separating CO_2 gas. In this work, an amine impregnated and amidated solid sorbent (AISS) containing a copolymer (PMMA-co-AA), which consists of acrylic acid (AA) and methyl methacrylate (MMA), and PEPA (polyethylene polyamine), was synthesized. For the first time, sorbents based on homopolymers and copolymers of acrylic acid and methyl methacrylate were compared for their ability to capture CO_2 gas. Other than the synthesis of low swelling AISS, a calculation of its energy consumption, and a comparison of its cyclic capacity with 30% water solutions of monoethanolamine and methyldiethanolamine (MEA and MDEA) were performed. The solid sorbent PMMA-co-AAS showed a higher cyclic capacity than others, corresponding to the order PMMA-co-AAS (23 mg/g) > PAAS (16 mg/g) > MDEA (10 mg/g) > MEA (6 mg/g). The average absorption rate for these sorbents was in the sequence of MEA > PMMA-co-AAS > PAAS > MDEA at 40 °C, and the desorption rates were PMMA-co-AAS > PAAS > MDEA > MEA for these sorbents at 70 °C, correspondingly. When the amount of acrylic acid in the copolymer was varied from 0 to 100%, the copolymer's water absorption capacity ranged from 0.2 to 1359.63%. Among them, the swelling ability of the chosen sorbent prepared from the 10% AA-containing copolymer and PEPA was 0.64%.

Keywords: acrylic copolymers; carbon capture; greenhouse gas; polyamine; sorbents; impregnation

1. Introduction

In recent decades, technologies have been developed around the world to clean up anthropogenic waste dumped into the biosphere [1,2]. The global warming process, i.e., the "greenhouse effect", is caused by the increase in the concentration of carbon dioxide, carbon monoxide, methane, and similar heat-trapping gases in the air during their mining, processing, and combustion. Moreover, the rapid depletion of fossil energy sources and the increase in harmful gases have led to the emergence of many diseases that did not occur in the past, and this process is a serious concern for the whole world [3,4].

CO_2 is captured using MEA or DEA absorbents, Cu(II)-based zeolites, and polymer-based sorbents. Absorbents, typically liquids with 30% amines and 70% water, are widely

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Published: 30 March 2026

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used for CO_2 separation. They react with CO_2, forming new compounds that transfer it into the liquid, and can be regenerated for reuse. Liquid absorbents are adaptable, easy to store, and economical, but their efficiency can decrease over time due to loss of chemical stability. Regeneration requires high energy, mainly due to heating water, and performance can drop if other gases or pollutants are present [5–7]. Ab(d)sorption capacity, ab(d)sorption/desorption kinetics, and desorption properties also have important sorbent properties for industry. CO_2 a(b)dsorption/desorption characteristics vary depending on the type of amine used in the statistics of the chemical sorbent [8]. In general, primary amines have a stronger affinity to absorb CO_2 than secondary amines, and secondary amines have a higher tendency to attract than tertiary ones. However, accordingly, primary amines require higher energy for CO_2 desorption. Therefore, it is now known that secondary amines are the most effective in the purification of CO_2 on an industrial scale due to their highly effective adsorption and desorption rates. Therefore, there is a growing demand for reliable secondary amine-containing solid sorbents. Currently, research is being conducted mainly on secondary amine-containing polyamines [9,10].

Acrylic polymers serve as raw materials in paints, coatings, dentistry, adhesives, fuel-oil additives, and as a gelling agent for battery electrolytes [11–16]. In addition, these polymers are also used as a base for solid sorbents. In recent studies, researchers have focused on modifying polyamine molecules to synthesize secondary amines [17]. An example of such work is tetraethylenepentamine modified with acrylonitrile, which was impregnated into poly(methyl methacrylate) supports [18], in which the properties of modified amines, such as heat resistance, viscosity, and desorption properties, are described in detail. It has been shown that the stability of the CO_2 adsorption/desorption process and, thus, the durability of the sorbent are improved by the thermal stability of the liquid amine, achieved through the reaction between TEPA and acrylonitrile (AN). The viscosity and heat resistance of amines are also important because amine loss can occur during the adsorption/desorption cycle [19,20]. If amines leak from the support during the sorbent's working cycle, the sorbent's CO_2 absorption capacity decreases.

Investigating the works mentioned above, the main purpose of this work is to develop a technology for the synthesis and application of an energy-efficient new solid sorbent that captures CO_2 gas, to contribute a bit to the reduction in the carbon footprint around the World. In this regard, investigations into methyl methacrylate and acrylic acid-based copolymers as solid supports for CO_2-capturing AISS [21] have been conducted. The developed sorbent demonstrates superior sustainability due to its stable structure, which prevents leakage of the impregnated liquid active phases from the solid support. Additionally, it efficiently desorbs absorbed CO_2 in a high-purity, dry state via temperature-swing desorption under vacuum. This process not only increases the purity of the recovered CO_2 but also reduces the need for additional drying steps, making it more energy- and cost-efficient. The sorbent's robust design enables repeated cycles of CO_2 absorption and desorption without significant performance loss, highlighting its potential for long-term industrial applications and environmentally friendly carbon capture solutions.

2. Methodology

A brief detail of acrylate-supported AISS production. In this study, a polycondensation reaction was performed between polyacrylic acid (PAA) and polyethylene polyamine (PEPA) in order to partially amidate the part of primary amines. The other part of the primary amines was bonded with polyacrylic acid, forming polyacrylate, i.e., the amine salt of acrylic acid. PEPA cross-linked polyacrylic acid (present as a comonomer in the copolymer or as a homopolymer constituent) forms a conjugate base and amid bonds.

https://doi.org/10.3390/engproc2026124097

The first function of the study was the synthesis of AISS by reacting with the primary amines of PEPA and PAA and defining physicochemical properties of the obtained solid sorbent; the second function was to compare the CO_2 adsorption/desorption properties of widely used sorbents in industry, such as MEA, MDEA, and the developed AISS.

Spectroscopic measurements: IR spectra were recorded on a Nicolet iS50 FTIR Advanced KBr Gold spectrometer (Thermo Fisher Scientific, Madison, WI, USA) + Nicolet Continuum (Thermo Fisher Scientific) at room temperature within the wave number range of 400–4000 cm^{-1}.

Experimental.

Chemicals used.

Primary reagents for AISS production are stabilized acrylic acid (CAS No. 79-10-7, pure for analysis), methyl methacrylate, bidistilled water, polyethylene polyamine (EC No. 701-266-7, technically clean), and industrial technical alcohol (GOST 18300, 97%). All reagents were used without a cleaning procedure.

Polymerization of polyacrylic acid in a vapor medium

The process of thermal polymerization of acrylic acid was carried out using the device shown in Figure 1. Acrylic acid stabilized by a polymerization inhibitor was boiled in flask 1. The boiling acrylic acid was cooled and partially condensed on the walls of tube 2 (reactor or polymerizer) and subsequently heated under the influence of the heat of the boiling acrylic acid vapors, leading to the initialization in the vapor phase. The non-polymerized acrylic acid was cooled using condenser 3, collected in collector 4, and again poured into a boiling flask using funnel 5. The process is completed before blocking the polymer monomer's vapor path. The polymer was removed from the glass tube and placed in a mortar in the form of debris less than 1 mm in size.

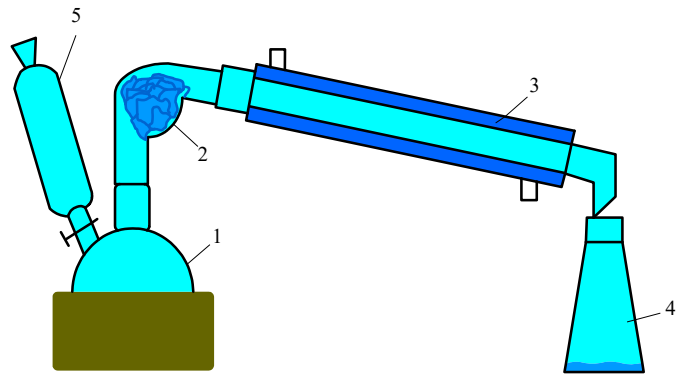


Figure 1. A laboratory setup for the polymerization of acrylic acid in the vapor phase.

Polymerization of poly(methyl methacrylate–co–acrylic acid) copolymer.

Acrylic acid and methyl methacrylate purified from polymerization inhibitor by distillation were copolymerized in the presence of the initiator AIBN. For this, toluene was used as a solvent, and the reaction was carried out in an N_2 atmosphere. The reaction started at 75 °C, and the stirring speed was 180 per minute.

The copolymer was washed in pure ethanol and ultrasonicated three times for 5 min each. The purpose of ultrasonication is to remove residual monomers and open the pores of the copolymer. Analyses of AISS spectroscopic data are given below.

Figure 2 shows the peaks at 1560.3 cm^{-1} and 1435.7 cm^{-1}, which represent the asymmetric and symmetric stretching vibrations of the COO^- group of the salts, i.e., acrylates, respectively. That has resulted from the acid-base reaction of the carboxylic acid on the binary copolymer, and the primary amines of the polyamine. The formed amide bond is evident from the peak at 1629.2 cm^{-1}. However, the double peak (tooth-shaped) characteristic of primary amines at 3280.91 cm^{-1} did not turn into a single peak, i.e., secondary amines,

which is explained by the lower number of carboxyl ions in the copolymer obtained in the ratio 1:9 (AK: MMA) compared to the primary amines in PEPA (in the ratio 1:4.5). See other spectra of the components of AISSs and pure acrylic acid supported AISS (in which that single peak appeared) in Supplementary Materials (Figures S1–S4).

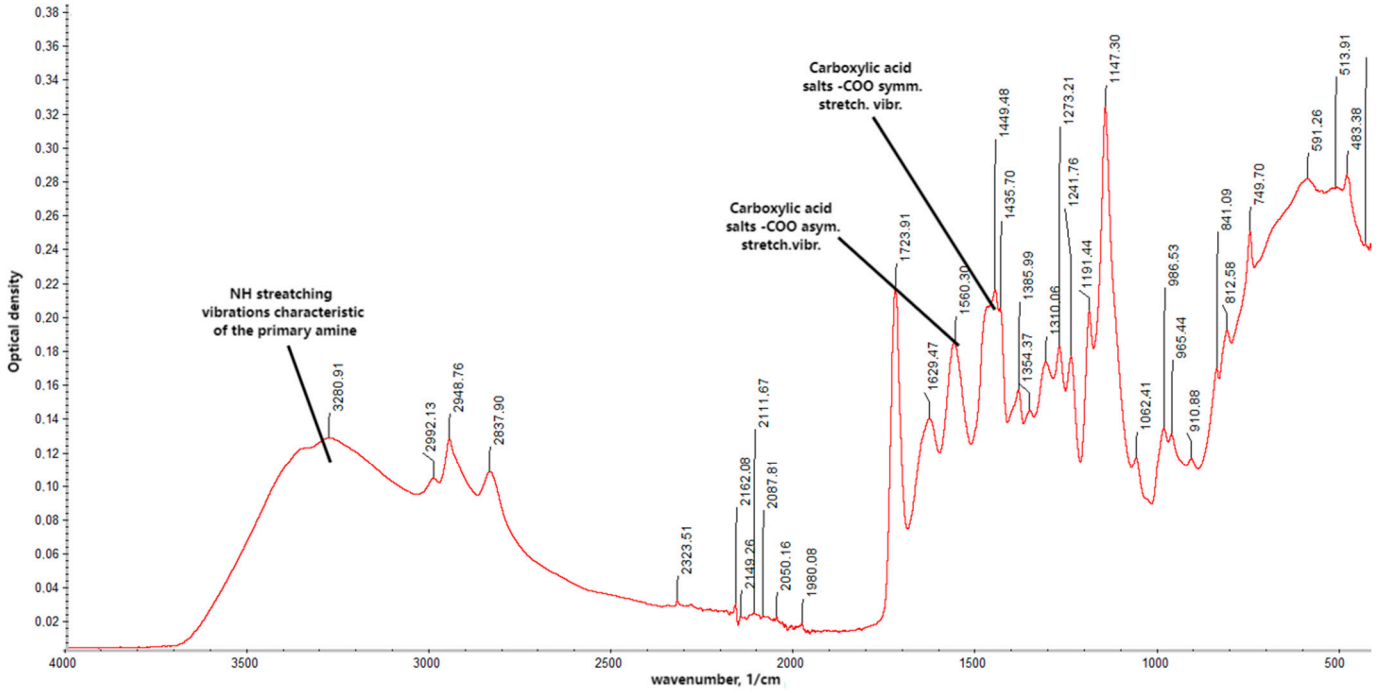
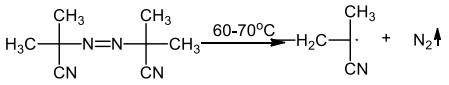


Figure 2. IR spectrum of an AISS based on poly(methyl methacrylate–co–acrylic acid) (PMMA-co-AAS).

3. Results and Discussion

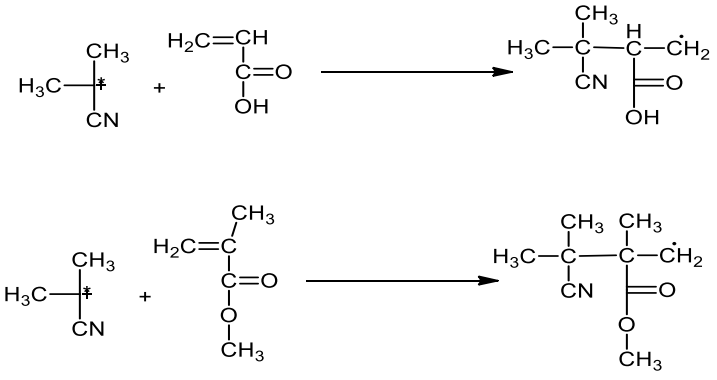
3.1. The mechanism of the reaction equation of the polymerization process in the work carried out is as follows:

Step I: Initiation or chain formation step: decomposition of the azoisobisisobutyronitrile initiator into a free radical



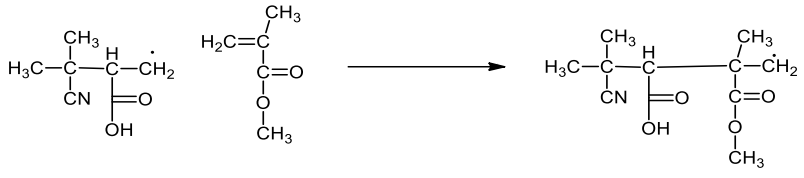
(1)

and it goes with the joining of the double bond of the

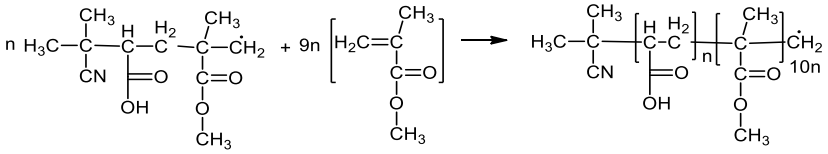


(2)

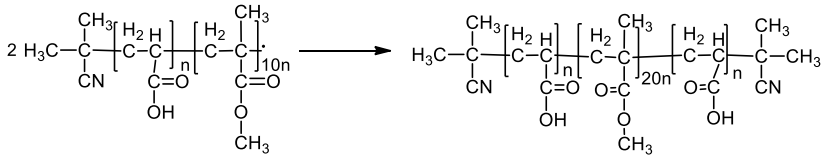
Step II: The stage of growth of the chain. It is formed by the addition of a monomer to a monomer radical in sequence:



(3)

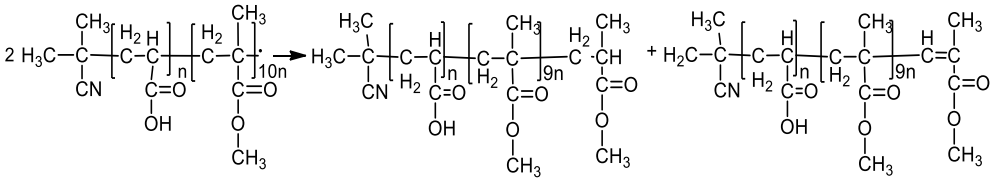


Step III: The stage of breaking the chain. Due to the recombination of the formed macroradicals, these macromolecules form bimolecules with each other, and the nature of their termination does not depend on the composition of the reaction mixture:



(4)

or can also be disproportioned:



(5)

So, the formation of each macromolecule consists of three elementary processes: initiation, growth (propagation), disruption (termination), or transmission (chain transfer). Some macromolecules are formed by breaking, while others are formed by transmission. Their molecular mass is different. The difference in the length of macromolecules depends on the general polymerization and the formation of macromolecules based on the laws of statistics. The sequence of reagents' loading is given in Table 1.

Table 1. Loading mode of reagents.

Name	M, g/mol	Ratio	m, g	*n*, mol	Initial	1st Dosage	2nd Dosage	3rd Dosage
MMA	100.12	10	45.508	0.455		4.551	40.957	
AA	72.063	1	3.276	0.0455		0.328	2.948	
AIBN	164.21		0.821	0.005	0.082			0.739
toluene			49.604		14.881			34.723

3.2. The laboratory measurement facility and procedure.

The cyclic ab(d)sorption capacity of the ab(d)sorbent was determined using the laboratory facilities shown in Figure 3a,b, performing ab(d)sorbsion and desorbsion (regeneration) respectively. The synthesized solid sorbent was compared with 30% of solutions of monoethanolamine (MEA) and methyldiethanolamine (MDEA), which are widely used industrially.

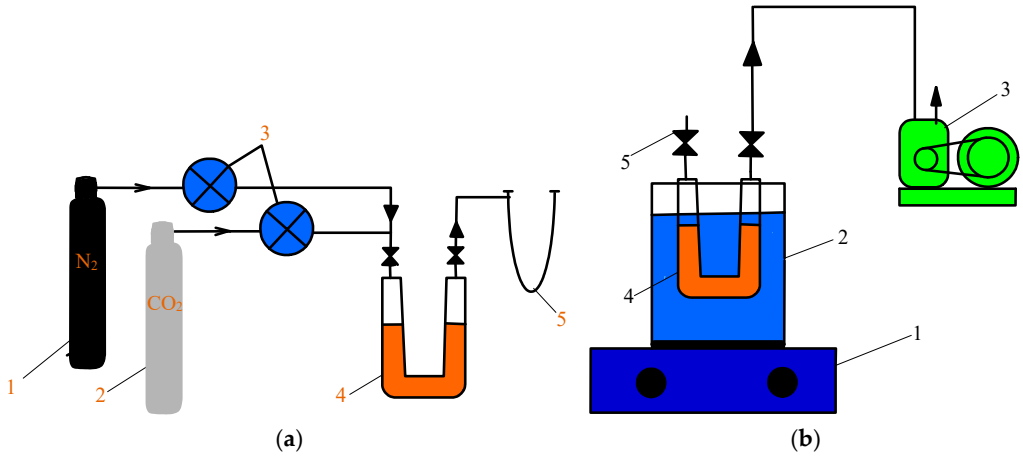


Figure 3. (**a**) Ad(b)sorption laboratory facility. 1—Nitrogen cylinder, 2—Carbon dioxide cylinder, 3—rotameter, 4—chemisorber, 5—hydraulic lock. (**b**) Regeneration laboratory facility. 1—heater, 2—water thermostat, 3—vacuum pump, 4—chemisorber, 5—tap.

The process began with the introduction of a mixture of N_2 and CO_2 gases from 1 and 2 gas cylinders, respectively, with a volumetric flow rate of 0.9–0.91 L/min. The gas flow rate was measured using 3 rotameters. The gas mixture passed through a layer of PMMA-co-AAS (or PAAS, or MEA, or MDEA) in the 4 chemisorbers. A water column (siphon) was used to establish pressure in the reactor.

The desorption, i.e., regeneration process of PMMA-co-AAS, PAAS, MEA, and MDEA saturated with CO_2 gas was carried out in the laboratory apparatus shown in Figure 3b. The CO_2 desorption was conducted under the temperature of 70 °C and the atmospheric pressure, as well as the ab(d)sorption was done under the temperature of 40 °C and pressure 200 mm H_2O.

The synthesized AISSs based on poly(methyl methacrylate–co-acrylic acid) and 30% monoethanolamine and methyldiethanolamine water solutions were each exposed to the same amount of CO_2. Adsorption rate was monitored by weighing the mass of the adsorber containing the solid sorbent every 15, 30, 45, and 60 min. The saturated CO_2 loading and minimum CO_2 loading of the sorbents were determined gravimetrically before and after the ab(d)sorption-desorption processes (see Table 2). The reaction (chemisorption) time for these sorbents was 45–60 min, and the desorption time was 30 min. The CO_2 capacity of these sorbents was measured at low temperature (40 °C for adsorption/absorption) and high temperature (70 °C for desorption) (see Table 2) and is shown graphically in Figure 4. When the maximum carbon dioxide gas absorption of 30% solutions of MEA and MDEA, as well as the synthesized AISSs, was determined, the MEA solution had the highest, the MDEA solution had the lowest, and the synthesized AISSs showed an average absorption rate. All sorbents absorb CO_2 gas by forming a chemical bond, i.e., chemisorption. The absorption capacity of the AISSs was very high in the first 30 min, but then showed very low capacity. After absorption/adsorption (40 °C), the maximum CO_2 loadings of MEA, PMMA-co-AAS, PAAS, and MDEA were 49, 38, 27, and 18 mg/g, respectively. After regeneration, the minimum CO_2 loadings of MEA, PMMA-co-AAS, PAAS, and MDEA were 43, 15, 11, and 8 mg/g, respectively. Similar results were also reported for MEA and MDEA in the literature [22]. The cyclic capacity for CO_2 capture can be calculated from the difference between the maximum CO_2-rich loading and the minimum CO_2-lean loading. This is an important parameter for estimating the working capacity of the sorbent and allows for accurate prediction of the volume and energy requirements in systems designed for CO_2 capture in pilot or full-scale CO_2 capture. The working cyclic capacity of these sorbents was in the order PMMA-co-AAS (23 mg/g) > PAAS (16 mg/g) > MDEA (10 mg/g) > MEA (6 mg/g),

meaning the higher the cyclic capacity, the lower the energy demand for regeneration. The solid sorbent PMMA-co-AAS exhibited a higher cyclic capacity than the others. Here, the low cycle capacity for MEA and MDEA is due to low (70 °C) desorption temperature, which requires about 90–110 °C in application.

Table 2. Carbon dioxide gas capture properties of MEA, MDEA, and PMMA-co-AAS.

Name of Substances	Maximum Saturated Load (Rmax), g/kg	Minimum Load at Recovery (Rmin), g/kg	Cyclic Capacity (ΔR), g/kg
MEA	49 *	43 ***	6
MDEA	18 *	8 ***	10
PAAS	27 **	11 ***	16
PMMA-co-AAS	38 **	15 ***	23

*—60 min, 40 °C, **—45 min, 40 °C, ***—60 min, 70 °C.

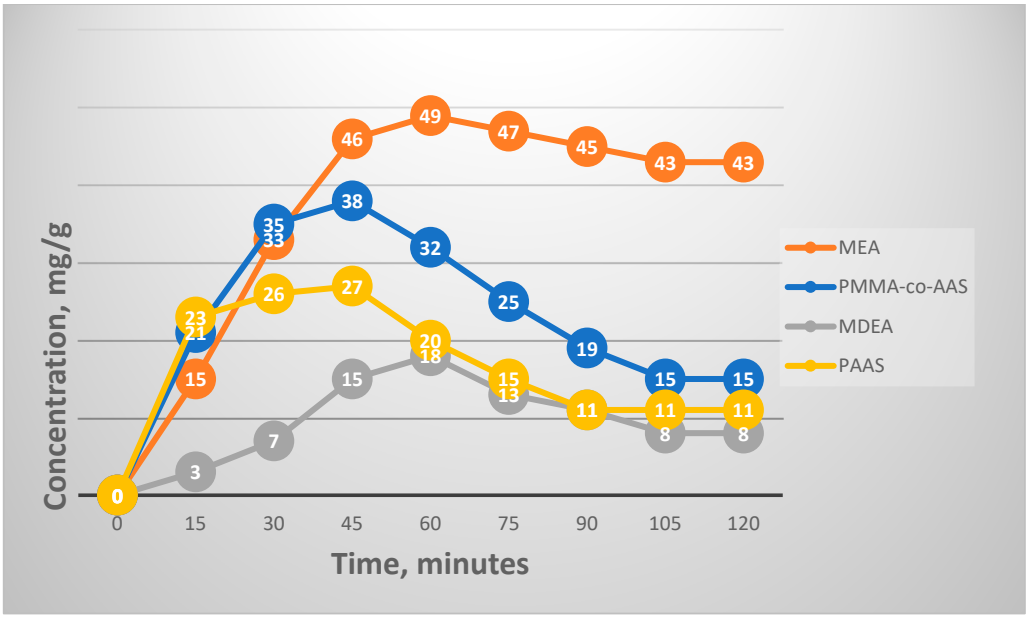


Figure 4. Graphical representation of the change in the concentration of CO_2 gas absorbed by the sorbent during the first absorption/adsorption and desorption cycle. MEA-brown line, PMMA-co-AAS-blue line, PAAS-yellow line, MDEA-grey line.

Since CO_2 absorption is exothermic in nature, according to Le Chatelier's principle, an increase in temperature should reduce the degree of chemisorption. Therefore, the conditions reported in the literature [23] were chosen for the absorption/adsorption and desorption processes. The average absorption rate for these sorbents is in the order MEA > PMMA-co-AAS > PAAS > MDEA at 40 °C, and the desorption rate is in the order PMMA-co-AAS > PAAS > MDEA > MEA for these sorbents at 70 °C.

3.3. Theoretical calculation results on determining the heat of regeneration. The heat capacity values for the sorbent solvent (H_2O)/support (PMMA-co-AAS) are given in Table 3. The heat capacity Cp,s values of the absorbents for MEA/MDEAs are taken from the literature [24].

Table 3. Comparative values of water and polymer supports.

Substance Name	Water	Polymethyl Methacrylate	Polyacrylic Acid	PMMA-co-AAS (90:10)
Heat capacity value, J/(g × K)	4.184	1.931	2.146	1.952

From Table 3, it can be seen that the difference in the heat capacities of the supports, i.e., water and PMMA-co-AAS, is 2.143 times. This indicates the energy efficiency of the obtained solid sorbent.

Currently, aqueous alkanolamines MEA and MDEA are mainly used in industry. In this case, the absorption temperature is on average 40 °C, the pressure is 35–42 kgf/cm^2, and the regeneration temperature is 120 °C, the pressure is 0.65–0.95 kgf/cm^2. The energy efficiency of the synthesized sorbent membranes is that the adsorption temperature is 40 °C, the pressure is 200 mm water above or ≈0.02 kgf/cm^2, the regeneration temperature is 70 °C, and the regeneration pressure can be carried out under atmospheric or vacuum (if high purity CO_2 is required).

3.4. Determination of the swellability of polymers in water. The swellability (B) of polymer samples was determined. The swellability (B) was determined by immersing 0.1 g of polymer samples in 100 mL of distilled water with a pH of 7.00 for 48 h at room temperature (22 °C). After 48 h, the samples were removed from the water, wiped with filter paper, and weighed. (B) was calculated using the following equation [25].

$$B = (Wb - Wq)100/Wq \quad (6)$$

where Wb and Wq are the swelled and dry weights of the polymer, respectively.

Based on the results obtained, the swellability of the samples was calculated using Formula (6). Based on the results of the calculations, a swellability diagram was developed (see Figure 5).

$$Q_{reg} = Q_{des} + Q_{sen} + Q_{steam} \quad (7)$$

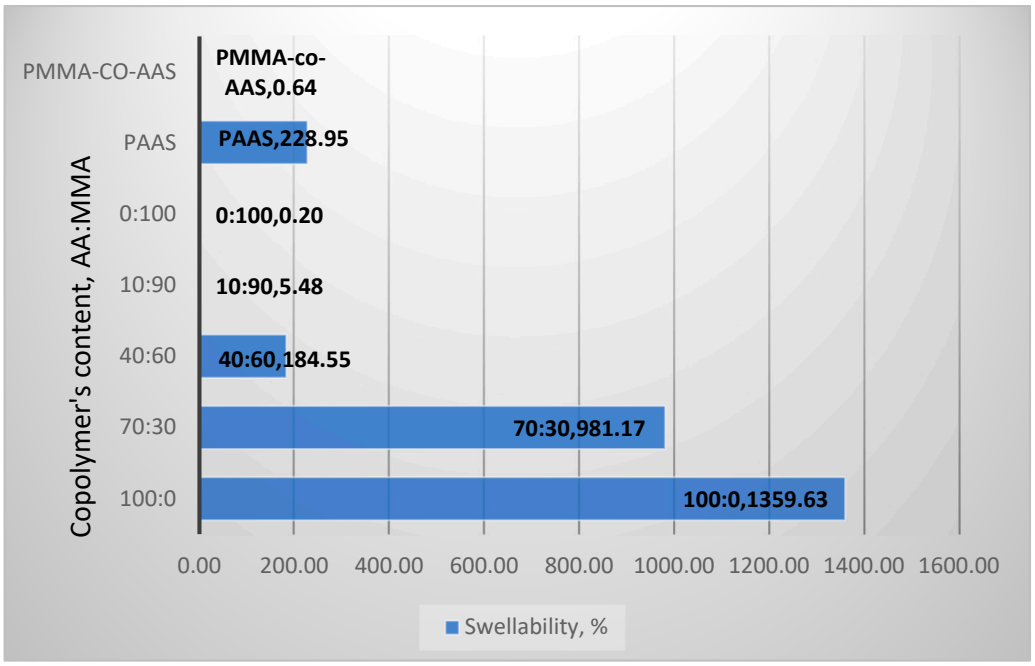


Figure 5. Water swellability of copolymers and AISSs composed of acrylic monomers.

The sensible heat, adsorption heat, vaporization heat, and total regeneration heat in Formula (7) were calculated for the PMMA-co-AAS CO_2 capture system and compared with the aqueous MEA 30%/MDEA system. The energy consumption required for the regeneration process is also evident in the values presented in Table 4. The sensible heat of the sorbent Qsen is the heat or energy that must be supplied to raise the temperature to the desired temperature. While 276.56 J/g of energy is required to raise a 30% MEA solution from 40 °C to 120 °C, for the PMMA-co-AAS sorbent, this value requires 65.149 J/g for the

temperature range 40–70 °C. This means that the energy consumption is 4.25 times less than for the MEA solution and 4.43 times less than for the MDEA solution [26].

Table 4. Heat capacity and sensible heat values of sorbents: theoretical.

Sorbent Name	Temperature Difference, °C	Heat Capacity, J/(g·°C)	Sensation Heat, J/g
MEA 30%	40–120	3.457	276.56
MDEA 30%	40–120	3.607	288.56
PMMA-co-AAS	40–70	2.1716	65.149

4. Conclusions

Thus, a synthesis of a CO_2 gas-absorbing AISS was performed from acrylic acid, i.e., a partial cross-linking by amidation reaction between the primary amines of polyethylene polyamine and the carboxyl groups of polyacrylic acid. Conditions for CO_2 absorption were established for the sorbents synthesized. As a result, the CO_2 adsorption capacity of the obtained AISS was found to be better than that of MEA, while the energy required for regeneration was reduced. In addition to the ability of the AISS obtained to trap CO_2, it exhibited easy CO_2 regeneration at relatively low temperatures (70 °C) and relatively excellent stability in adsorption–desorption cyclic processes. Such an AISS is of great importance for use in the purification of flue gases and other CO_2—containing gas mixtures on an industrial scale. The following can be inferred from a test study conducted to capture CO_2 gas: First, the initial rate of adsorption and desorption increased. Second, the recyclability of PAAS and PMMA-co-AAS was shown to be superior to that of MEA and MDEA water solutions. Finally, an efficient and energy-saving CO_2 AISS was obtained.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/engproc2026124097/s1, Figure S1. IR spectrum of polyacrylic acid; Figure S2. Polyethylenepolyamine IR spectrum; Figure S3. IR spectrum of a AISS based on polyacrylic acid; Figure S4. IR spectrum of poly(methyl methacrylate-co-acrylic acid).

Author Contributions: Conceptualization, S.S.k.K. and O.S.M.; methodology, S.S.k.K. and O.S.M.; validation, S.S.k.K. and O.S.M.; formal analysis, O.S.M.; data curation, S.S.k.K.; writing—original draft preparation, S.S.k.K. and O.S.M.; writing—review and editing, S.S.k.K. and O.S.M.; visualization; supervision, S.S.k.K. and O.S.M.; project administration, S.S.k.K. and O.S.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format, they are available from the corresponding author upon reasonable request.

Acknowledgments: We would like to express our deep gratitude to "Shams Neft Mahsuloti" LLC for supplying chemicals for this research work and "Al Know How" LLC for providing practical and laboratory technical assistance.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

MEA	Monoethanolamine
MDEA	Methyldiethanolamine
CO_2	Carbon dioxide
PAAS	Polyacrylic acid-supported sorbent
PMMA-co-AAS	Poly (methyl methacrylate-co-acrylic acid) supported sorbent

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