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Cu_2BaSnS_4/Polyanilin Kompozitlerinde CO_2 Adsorpsiyonu: Sinerjik Bir Yaklaşım

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ÖZ

İletken polimerlerin kalkojenit bazlı malzemelerle birleştirilmesi, CO_2 adsorpsiyon performansını artırmada umut verici umutlar göstermiştir. Bu makalede, yeni bir Polianilin/Cu_2BaSnS_4 (PANI/CBTS) kompoziti, Cu_2BaSnS_4 gözenekleri içinde anilinin polimerizasyonu ile sentezlenmiş ve CBTS'nin PANI'de CO_2 alımı ve salınımı davranışı üzerindeki sinerjik etkisi araştırılmıştır. Tüm kompozit malzemeler çeşitli CBTS içerikleriyle hazırlanmış ve %5 CBTS ilavesi en umut verici sonuçları göstermiştir. BET yüzey alanı üzerinden yapılan çalışmalar, CBTS'nin PANI'ye eklenmesinin kompozit malzemenin gözenekliliğini yüzey özellikleriyle birlikte optimize ettiğini ve bozulmamış PANI'ye göre CO_2 adsorpsiyon kapasitesinin artmasına neden olduğunu ortaya koymuştur. Bu arada, Taramalı Elektron Mikroskobu (SEM) ve X-ışını Kırınım (XRD) analizleri CBTS partiküllerinin kompozit içinde homojen dağılımını ve CBTS'nin kristal bütünlüğünü doğrulamıştır. CO_2'nin adsorpsiyon-desorpsiyon döngüleri, CBTS ilavesini takiben alımında keskin bir düşüş gösterdi çünkü PANI'nin mikro ve mezo gözenekleri CBTS partikülleri tarafından bloke edildi ve adsorpsiyon-desorpsiyon sürecini engelledi. Bununla birlikte, elde edilen PANI/CBTS5 kompoziti, CO_2 yakalama uygulamaları için nispeten umut verici bir performans göstermiş ve adsorban malzemelerde etkili ve sürdürülebilir gelişim arayışları için yeni yollar açmıştır.

CO_2 Adsorption in Cu_2BaSnS_4/Polyaniline Composites: A Synergistic Approach

Research Article

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ABSTRACT

The incorporation of conductive polymers with chalcogenide-based materials has shown promising prospects in enhancing CO_2 adsorption performance. In this paper, a novel Polyaniline/Cu_2BaSnS_4 (PANI/CBTS) composite was synthesized by the polymerization of aniline inside the pores of Cu_2BaSnS_4 for investigating the synergistic effect of CBTS incorporation on CO_2 uptake and release behavior in PANI. All composite materials were prepared with varied content of CBTS, and addition of 5% of CBTS showed the most promising results. The studies via BET surface area revealed that introduction of CBTS in PANI optimizes the porosity of composite material along with its surface properties and results in enhancement of CO_2 adsorption capacity over pristine PANI. Meanwhile, the Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) analysis confirmed the homogeneous dispersion of CBTS particles in the composite and the crystalline integrity of CBTS. The adsorption-desorption cycles of CO_2 showed a sharp decrease in uptake following the addition of CBTS because the micro- and mesopores of PANI were blocked by CBTS particles, impeding the adsorption-desorption process. However, the as-received PANI/CBTS5 composite demonstrated relatively

promising performance for CO_2 capture applications and opened up new avenues for the pursuit of effective and sustainable development in adsorbent materials.

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1. Introduction

Since the Industrial Revolution, the intensive use of fossil fuels has led to severe air pollution and a rapid increase in CO_2 emissions, significantly contributing to global climate change. Reducing CO_2 emissions is crucial to mitigating environmental impacts and ensuring the sustainable use of resources (Nunes, 2023). One of the most effective strategies to reduce anthropogenic CO_2 emissions involves exploring cost-effective and scalable CO_2 capture technologies at emission sources. Among the various CO_2 capture techniques, sorption (absorption and adsorption), membranes, biological conversions, and cryogenic methods have emerged as prominent approache (Song et al., 2019; Lei et al., 2020; Zhang et al., 2020) s. Currently, aqueous amine solutions are the most used absorbents for capturing CO_2 from industrial emissions. However, despite their effectiveness, amine-based processes face challenges, particularly in operation and regeneration, which can raise economic and environmental concerns (Zaker et al., 2023).

Solid sorbents have garnered significant attention due to their selective adsorption of gases such as CO_2, H_2, and CH_4 (Gupta et al., 2015). Various classes of solid materials, including zeolites, silicas, porous carbons, porous organic polymers, covalent organic frameworks, and metal-organic frameworks, have been developed for CO_2 capture and conversion applications (Dziejarski et al., 2023). In recent years, transition metal sulfides have gained extensive research interest due to their excellent stability, unique band structures, and semiconductor properties, making them promising candidates for optoelectronic applications (Cao et al., 2023). One such material, copper barium tin sulfide (CBTS), has emerged as a potential photocathode material for CO_2 reduction due to its favorable conduction band position and suitable bandgap (~1.9 eV). CBTS, a quaternary compound semiconductor, is known for its exceptional light absorption capacity, low cost, environmental friendliness, and abundant availability (Khattak et al., 2019). Recent studies have considered CBTS films as a promising photovoltaic material for high-efficiency thin-film solar cells (Guo et al., 2019). Although CBTS has mainly been investigated for photovoltaic applications, its band structure and semiconductor properties make it a promising candidate for photocatalytic CO_2 reduction and hydrogen production. Despite these advantages, CBTS has not been sufficiently explored in the field of photocatalytic fuel production, particularly for short-chain carbon-based fuels such as CO, CH_4, H_2, and CH_3OH. The main challenge lies in the high carrier recombination rates and low photoelectric conversion efficiencies inherent to single semiconductor materials (Cao et al., 2023). To overcome these limitations, strategies such as incorporating co-catalysts, forming heterojunctions, modifying surface passivation layers, and adjusting material morphology have been explored (Cao et al., 2023).

Polyaniline (PANI) has gained significant research attention due to its simple synthesis method and high environmental stability. PANI has a decomposition temperature of approximately 400°C and contains abundant amine groups, which enhances its potential for CO_2 capture (Khalili et al., 2016). The nanostructures of PANI (such as nanowires, nanofibers, nanotubes, and nanorods) support rapid gas diffusion within the structure due to their small diameters, large surface areas, and high penetration depth for gas diffusion, thereby increasing CO_2 adsorption capacity (Fratoddi et al., 2015; Khalili et al., 2016). CO_2 molecules interact chemically with the surface of PANI through the partial formation of carbonates and bicarbonates, improving gas capture efficiency (Khalili et al., 2016). To increase the affinity of nanomaterials to CO_2 and thereby improve the separation efficiency, composites with polymers such as polyethyleneimine (PEI) and PANI have been synthesized. PANI, one of the well-known conductive polymers, has recently been loaded onto MIL-101(Cr), resulting in a significant increase in CO_2 selectivity against N_2 (Aliev et al., 2016). Although the amount of adsorbed CO_2 decreased slightly compared to pure MIL-101(Cr) (for example, the amount adsorbed at 1 bar decreased from 3.35 mmol/g to 2.26 mmol/g when PANI was loaded), CO_2 selectivity improved significantly. This decrease could be attributed to the substantial reduction in porosity due to the large amount of PANI loaded onto MIL-101(Cr); the BET surface area of MIL-101(Cr) decreased from 3580 m^2/g to 631 m^2/g after PANI loading. Additionally, a recent study reported that increasing the amount of PANI loaded onto mesoporous SBA-15 also enhanced CO_2 capture capacity (Boukoussa et al., 2018). These findings suggest that modifying nanomaterials with PANI is a feasible strategy to enhance CO_2 capture performance.

While PANI has previously been introduced into materials like MIL-101(Cr) to promote CO_2 selectivity, its combination with CBTS for CO_2 adsorption has not been documented. In this work, we concentrate on the influence of CBTS integration on the CO_2 uptake performance of PANI. The composite was created by polymerizing aniline inside the pores of CBTS, resulting in a hybrid material with possibly increased adsorption capabilities. By injecting 5% CBTS into PANI, we systematically investigated the influence of this alteration on CO_2 adsorption capabilities based on BET surface area analysis. This study intends to give insights into the synergistic impact of PANI and CBTS in composite materials and provides a fresh viewpoint on their potential for effective CO_2 capture applications.

Although both CBTS and PANI have individually demonstrated promising properties for CO_2 capture and photocatalytic applications (Khalili et al., 2016; Boukoussa et al., 2018; Khattak et al., 2019; Cao et al., 2023), their integration into a single composite material has not yet been reported. This study is the first to investigate the synergistic interaction between CBTS and PANI for CO_2 adsorption applications. By leveraging the high surface functionality and conductive polymeric nature of PANI (Khalili et al., 2016; Boukoussa et al., 2018) with the favorable band structure and chemical stability of CBTS (Khattak et al., 2019; Cao et al., 2023), the designed composite is expected to overcome the intrinsic limitations of the individual components, such as low photoelectric conversion efficiency in CBTS (Cao et al., 2023) and reduced porosity in PANI-based systems. Furthermore, the strategy of

polymerizing aniline within the CBTS framework not only provides improved CO_2 adsorption capacity but also offers a new approach for tuning the structural and textural properties of hybrid sorbents. This novel design concept broadens the application scope of CBTS beyond its traditional photovoltaic role and provides a rational pathway for developing multifunctional hybrid materials for next-generation CO_2 capture technologies.

2. Material and Methods

All compounds used in the production of the materials were used without any purification process. Copper chloride ($CuCl_2$) (Sigma Aldrich), Barium sulfate ($BaSO_4$) (Across Organics), Stann chloride ($SnCl_4$) (TCI Chemical) and Thiocarbamide ($CS(NH_2)_2$) (Across Organics) were supplied from commercial sources. In the production of PANI, Aniline hydrochloride (Thermo-scientific, purity: 99%) was used as monomer, ammonium persulfate (Acros Organics APS, purity: 98+%) was used as initiator, and sulfuric acid (95-98% wt) supplied by Sigma Aldrich was used as solvent. Water used in the synthesis process was purified with Millipore Milli-Q system (Millipore Inc., Ω = 18 MΩ cm).

2.1. Synthesis of CBTS

CBTS was produced using a conventional solution-based synthesis process that we used in our previous study (Güngör et al., 2024). First, the precursor solution was prepared by dissolving $CuCl_2$, $BaSO_4$, $SnCl_4$ and $(CS(NH_2)_2)$ in ethylene glycol solvent at a stoichiometric molar ratio=2:1:1:8. Then, the solution was transferred to a Teflon-coated stainless-steel autoclave to be heated at 200 °C 24h to ensure the formation of the basic kesterite phase. The obtained product was washed with ethanol and water using the centrifugation method and dried in a 90 °C oven (Ali et al., 2021; Levcenko et al., 2021) (Figure 1A).

2.2. Synthesis of PANI

The Aniline monomer (1 mmol) was dissolved in 10 mL of 1 M perchloric acid ($HClO_4$) solution and stirred for 10 min. In a separate beaker, ammonium persulfate (APS) (1 mmol) was added to 100 mL of 1 M $HClO_4$ and stirred for 10 min. APS solution was slowly added dropwise to the aniline solution under constant stirring at room temperature. The start of the polymerization reaction was understood when the solution turned dark green. The reaction mixture was cooled to 4 °C and left to react for 24 h to obtain pure PANI. The obtained product was collected by centrifugation, washed with ethanol and distilled water, and dried at 70 °C for 24 h (Chiou et al., 2008) (Figure 1B).

2.3. Synthesis of PANI/CBTS

The PANI synthesis method was followed with minor changes to synthesize PANI: CBTS nanocomposites prepared by doping 5% CBTS by mass (w/w, %)). Aniline and CBTS powder in 10 mL of 1 M $HClO_4$ were sonicated for 30 min. The APS solution prepared in another beaker was added

dropwise to the CBTS solution to ensure polymerization. The reaction mixture was cooled to 4°C and left to react for 24 h. The resulting product was added to ethanol and washed with distilled water and dried at 70 °C for 24 hours (Figure 1C). The obtained nanocomposites were coded as PANI: CBTS5.

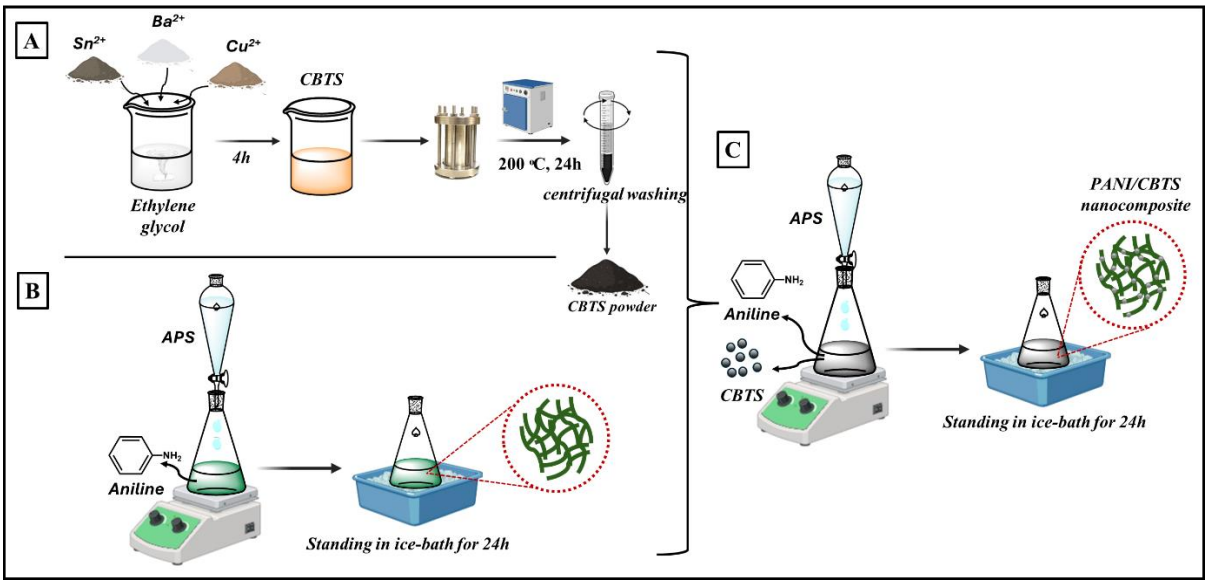

Figure 1. Schematic representations of the synthesis of (A) CZTS, (B) PANI, and (C) PANI/CBTS 10 nanocomposites.

3. Results and Discussion

The structural, morphological, and surface properties of the synthesized materials were thoroughly analyzed to evaluate their appropriateness for CO_2 collection and energy storage applications. The synergistic combination of PANI and CBTS in the PANI/CBTS5 composite was specifically examined to assess the impact of the material's microstructure, crystallinity, and surface characteristics on its overall performance. The findings derived from SEM, XRD, and BET investigations provide significant insights into the materials' structure, emphasizing their potential for advanced functional applications.

Figure 2 presents SEM images for the investigated materials. The SEM micrographs below show that the PANI nanomaterials exhibit a homogenous morphology with diameters within the range of 45-65 nm, as shown in Figure 2a. Such nanostructures form a cable-like structure that stretches along the longitudinal axis towards micrometer-scale lengths, hence increasing the porosity of the material. In Figure 2b, CBTS consists of well-defined crystalline particles with uniform and small dimensions. While in some regions, there has been the appearance of sphere-like particles; this is mostly attributed to the contribution of the solvent elements during the process of synthesis. The PANI/CBTS5 composite exhibited a homogenous dispersion of CBTS on the surface of PANI nanomaterials; Figure 2c indicates such dispersion. It is considered an essential factor for enhancement in the electrical conductivity of the material and its active surface area. While there is minor aggregation of particles in some regions, this does not detract from the general morphology of the composite.

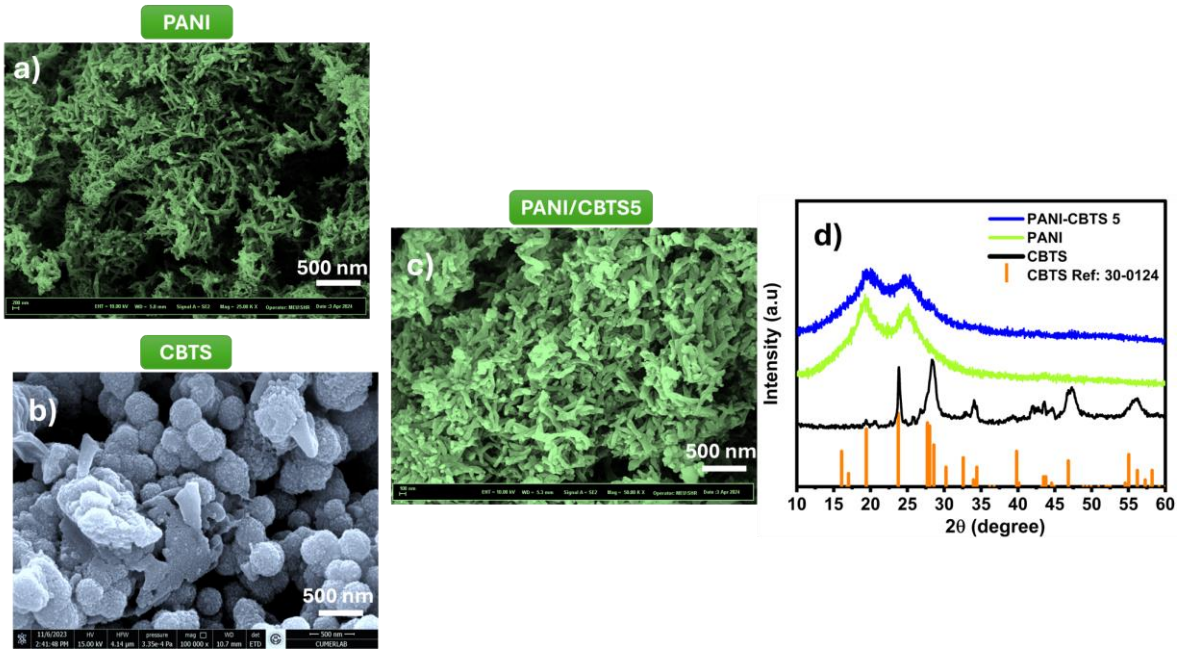

Figure 2. SEM images of PANI (a), CBTS (b), PANI/CBTS5 (c) and XRD graph (d).

Figure 2d shows the XRD patterns of the materials. The PANI XRD pattern exhibited a broad peak at $2\theta \approx 20°$, which confirms its amorphous nature because of the irregular structure of its molecular chain. In contrast, the XRD pattern of CBTS reveals sharp, strong diffraction peaks at 2θ positions of 27.4°, 45.5°, and 53.7° corresponding to characteristic crystallographic planes of CBTS. Such peaks fit well in the JCPDS PDF card, No. (30-0124), evidence for the highly crystalline nature of CBTS in support of the successful synthesis route (Crovetto et al., 2020; Ali et al., 2021; Teymur et al., 2022). The XRD pattern of the PANI/CBTS5 composite indicates the broad peak related to the amorphous nature of PANI and pronounced peaks typical of the crystalline phase of CBTS. This surely confirms that CBTS has been successfully incorporated into the PANI matrix. In addition, changes in peak intensities indicate homogeneous incorporation of material phases in this composite.

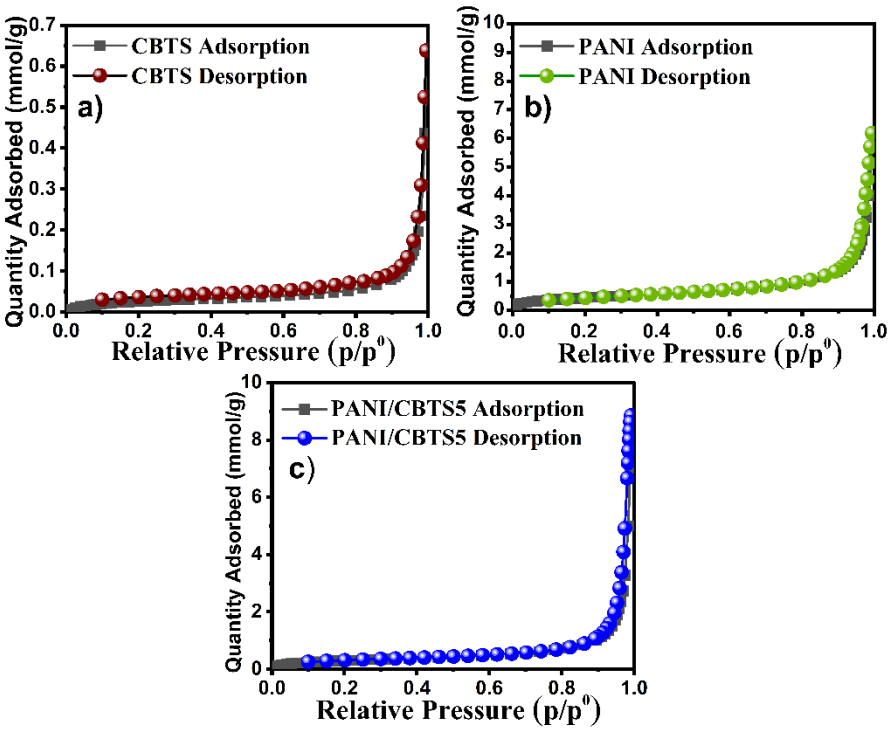

Figure 3. BET analysis results of PANI (a), CBTS (b) and PANI/CBTS5 nanomaterials.

The surface area and porosity of the materials in Figure 3 were studied by BET analysis. The PANI demonstrated a surface area of 35.3 m^2/g, aligning with its porous characteristics, and the pore size distribution was mostly mesoporous. For CBTS, the surface area was measured as 2.2 m^2/g, showing its thick crystalline structure and restricted porosity. The PANI/CBTS5 composite revealed a surface area of 24.35 m^2/g, somewhat lower than that of pure PANI. This decrease may be due to the uniform distribution of CBTS particles over the PANI surface, which may partly block PANI's pores (Zhou et al., 2018; Yu et al., 2023). Despite this, the active sites generated by CBTS particles greatly boost the composite's electroactive surface area. BET adsorption-desorption isotherms for all three materials (PANI, CBTS, and PANI/CBTS5) are typical of a Type-3 isotherm, which is suggestive of weak adsorbate-adsorbent interactions and implies the existence of non-porous or mesoporous structures. This sort of isotherm likewise corresponds with the measured surface areas and supports the structural features of the materials. The Type-3 isotherm behavior further demonstrates that the materials offer surface features suited for applications such as CO_2 collection and energy storage, where adsorption dynamics and active surface properties play significant roles (Liu et al., 2023).

Table 1. BET surface area and Horvath-Kawazoe (HK) cumulative PANI and CBTS nanocomposite pore volume.

Material	BET surface area (m^2/g)	HK cumulative pore volume (cm^3/g)
PANI	35.33	0.1391
CBTS	2.20	0.0107
PANI/CBTS5	24.35	0.1845

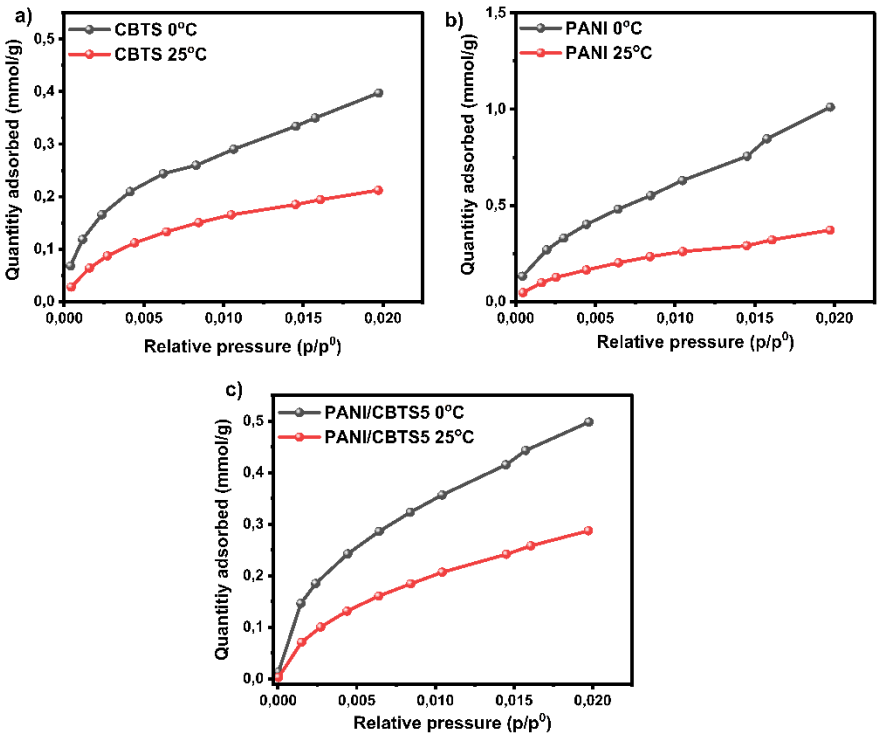

Figure 4. CO_2 adsorption analysis at 0°C and 25°C of PANI (a). CBTS (b) and PANI/CBTS5 (c) nanomaterials.

The CO_2 adsorption performance of PANI. CBTS. and PANI/CBTS5 nanomaterials was evaluated at 1 bar pressure using a surface area and porosity analyzer (BET). The corresponding results are provided in Figure 4 and Table 2. Among the materials. PANI. which exhibited the highest surface area. demonstrated superior CO_2 adsorption capacity at both investigated temperatures. Conversely. in alignment with the BET analysis. the PANI/CBTS5 nanomaterial showed a significant reduction in CO_2 adsorption. decreasing by approximately 50% at both temperatures. This decline can be attributed to the CBTS nanomaterial adhering to the micro- and mesopores of PANI. effectively blocking these pores and hindering the adsorption-desorption process (Liu et al., 2023). Overall. the CO_2 adsorption performance of the synthesized materials suggests their potential suitability as effective adsorbents for CO_2 Capture System (CCS) technologies.

Table 2. CO_2 adsorption analysis at 0°C and 25°C of PANI and CBTS nanocomposite pore volume.

Material	CO_2 Adsorption Analysis at 0°C mmol/g	CO_2 Adsorption Analysis at 25°C mmol/g
PANI	1.0110	0.3711
CBTS	0.3976	0.2122
PANI/CBTS10	0.4737	0.2568

4. Conclusions

In this work. Cu_2BaSnS_4/Polyaniline (PANI) composites were effectively synthesized and analyzed to examine their potential for CO_2 capture. The findings revealed that the addition of PANI into Cu_2BaSnS_4 improved both the surface area and porosity. considerably enhancing the CO_2 adsorption capability. BET surface area examination validated the advantageous porosity of the PANI/CBTS5 composite. while SEM and XRD tests demonstrated the homogeneous dispersion of CBTS particles on the PANI nanomaterial surface. These structural modifications led to increased CO_2 adsorption. especially under low-pressure conditions. which are critical for practical CO_2 capture applications.

The CO_2 adsorption performance of PANI. CBTS. and PANI/CBTS5 nanomaterials was evaluated at 1 bar pressure using a BET surface area and porosity analyzer. Among the materials. PANI. which exhibited the highest surface area. demonstrated superior CO_2 adsorption capacity at both investigated temperatures. Conversely. the PANI/CBTS5 nanomaterial showed a significant reduction in CO_2 adsorption approximately 50% at both temperatures. This decline is attributed to the CBTS nanomaterial adhering to the micro and mesopores of PANI. effectively blocking these pores and hindering the adsorption-desorption process.

Overall. the synergy between Cu_2BaSnS_4 and PANI provides a feasible method for creating materials with enhanced CO_2 adsorption capabilities. The synthesized materials demonstrated their potential suitability as effective adsorbents for CO_2 Capture System (CCS) technologies. Future research should focus on evaluating the long-term stability and reusability of the composite. as well as its performance under real-world conditions. Additionally. a comparative analysis of the CO_2 adsorption capabilities of PANI/CBTS composites with other known materials could yield valuable insights for further improvements. This study contributes to the development of sustainable and effective materials for CO_2 capture. which may play a crucial role in mitigating climate change by addressing industrial CO_2 emissions.

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Statement of Conflict of Interest

SGC declares that the study complies with all applicable laws and regulations and meets ethical standards.

Credit Authorship Contribution Statement

SGC single-handedly planned and designed the synthesis and modification of Cu_2BaSnS_4 (CBTS) and performed the synthesis experiments. SGC alone collected and analyzed the experimental data. SGC solely planned and designed the CO_2 uptake studies. SGC independently designed and supervised the BET, SEM, and XRD analyses. The manuscript was written entirely by SGC.

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