ORIGINAL PAPER

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Upcycling sugar beet waste into sustainable organo-nanocatalysis for carbon dioxide fixation and cyclic carbonate synthesis: a research design study



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Abstract

Environmental pollution is a major global issue due to the increase of various pollutants all over the world. Enhancing pollutant remediation strategies for environmental sustainability necessitates increasing the efficiency of conventional methods or introducing innovative approaches. Nanotechnology, particularly carbon-based nanomaterials, offers substantial promise due to their high surface area and absorption potential. Concurrently, organocatalysts have emerged as sustainable and versatile alternatives to traditional metal-based catalysts in modern chemical research. This study highlights the synthesis and application of organo-nanocatalysts derived from biomass, specifically a spherical carbon nanocatalyst synthesized from sugar beet pulp. This novel green catalyst, characterized by high selectivity and efficiency, successfully converts epoxides and CO₂ into valuable cyclic carbonates under solvent-free conditions. The hydroxyl groups on the Sugar Beet-derived Carbon NanoSphere (SCNS) surface act as Bronsted acid sites, facilitating epoxide activation via hydrogen bonding. The integration of carbon-based nanomaterials and organocatalysis represents a promising, sustainable solution for pollutant remediation and green chemistry advancements.

Keywords Organo-nanocatalyst, CO₂ fixation, Cyclic carbonates, Sugar beet, Heterogeneous catalyst, Brønsted acid

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Introduction

The pressing demand for sustainable and eco-friendly chemical processes has spurred the creation of innovative catalysts and methods to effectively harness carbon dioxide (CO₂) as a raw material in chemical synthesis (Song et al. 2017). Carbon dioxide is a greenhouse gas that exists in significant quantities in our atmosphere. Interestingly, scientists view it as a promising and renewable carbon resource for creating valuable chemicals through sustainable processes (Dibenedetto and Nocito 2020; Gür 2022). Due to the increase in CO_2 emissions as a common industrial waste gas and the excessive production of this gas, CO₂ recycling and utilization methods have become one of the most popular and interesting topics for researchers in scientific investigations (Allen et al. 2009; Barnes et al. 2019; Kiat et al. 2019; Gulzar et al. 2020; Nasirov et al. 2022 Feb). Hence, carbon dioxide capture and storage (CCS) and carbon dioxide capture and utilization (CCU) are viewed as crucial strategies for mitigating the issue of climate change (Roy et al. 2023). CO_2 , an abundant, non-toxic, and inexpensive greenhouse gas, can be likened to a concealed treasure in the atmosphere (Wickramasinghe et al. 2021). Scientists have successfully discovered various ways to convert this unwanted gas into valuable organic compounds, effectively transforming a problem into a solution. This process can produce various useful substances such as formic acid (Sun et al. 2022; Verma et al. 2021), hydrocarbons (Chen et al. 2018), alcohols (Erickson et al. 2020; Li et al. 2024), aldehydes (Liu et al. 2018; Ren et al. 2017), and even carbonates (Pal et al. 2022; Das et al. 2022). In essence, it is akin to molecular-level recycling. A frequently employed approach to stabilize and recycle carbon dioxide involves the formation of five-membered cyclic carbonates. These compounds can be synthesized either by combining CO_2 with diols (Gérardy et al. 2019) and epoxides (Marciniak et al. 2020) or by converting glycerol (Okoye and Hameed 2016) into glycerol carbonate and its derivatives (Scheme 1).

OH

diols

HO





Scheme 1 Variation of cyclic carbonate synthesis routes using carbon dioxide

One promising approach for the chemical fixation of CO_2 is the synthesis of cyclic carbonates from epoxides through a catalytic process (Scheme 2).

Cyclic carbonates are versatile building blocks used in various industrial applications, including as solvents, monomers for polymers, electrolytes in lithium-ion batteries, and as renewable raw materials in a wide range of chemical reactions, pharmaceutical/fine chemical intermediates, and in many biomedical applications (Rollin et al. 2021; Lopes et al. 2020). The synthesis of cyclic carbonates from epoxides and CO₂ offers a sustainable alternative to traditional methods that rely on toxic and environmentally harmful reagents (Rehman et al. 2021). Hence, a diverse range of catalysts (homogeneous/heterogeneous) has been devised to enhance the conversion of carbon dioxide into cyclic carbonates. These catalysts include metal halides (Natongchai et al. 2022), metalorganic complexes (Prasad et al. 2022), metal-organic frameworks (Helal et al. 2023), metal oxides (Kulal et al. 2019), organocatalysts (such as organic salts and ionic liquids) (Zhang et al. 2021), porous materials (Torabi et al. 2023), and hydrogen bond donor compounds (Silva 2019). In recent years, organocatalysts have emerged as efficient and environmentally benign catalysts for the synthesis of cyclic carbonates from epoxides and CO₂ (Ala'a et al. 2022; Li et al. 2023). Organocatalysts are organic molecules that can activate substrates and facilitate chemical reactions without the need for transition metals or other toxic components (Sinibaldi et al. 2019). The use of organocatalysts in the synthesis of cyclic carbonates offers several advantages, including high selectivity, mild reaction conditions, and easy recyclability (Cokoja et al. 2015).

Plant biomass, which is composed of a carbon framework, holds great potential as a source material due to its abundance of carbon, affordability, accessibility, and recyclability. The physical and chemical properties of carbon materials derived from biomass have garnered significant interest, primarily because of their remarkable characteristics, including a large specific surface area and a porous structure (Chen et al. 2014; Singh et al. 2020; Tang et al. 2018; Vinayagam et al. 2020). The diverse range of applications for carbon nanomaterials derived from biomass extends to numerous fields, encompassing supercapacitors, batteries, fuel cells, sensing and bio-imaging, catalysis, polymer processing (as nano fillers), drug delivery systems, water purification, thermal insulation materials, and electro-catalysis (Tiwari et al. 2022; Zaytseva and Neumann 2016).

Upcycling, or creative reuse, involves converting byproducts, waste materials, or unwanted items into new materials or products of superior quality or value compared to the original. This process differs from traditional recycling, which typically entails breaking down materials to produce new items of similar or lower quality. The primary objective of upcycling is to minimize waste by repurposing existing materials. This strategy reduces the need for new raw materials, thereby decreasing energy consumption, air and water pollution, and greenhouse gas emissions (Zhang et al. 2022). Upcycling represents a crucial step toward a more sustainable and regenerative design culture, resulting in end products that are cleaner, healthier, and often more valuable than the original materials. The upcycling of agricultural by-products offers a promising pathway for sustainable development and environmental conservation. For instance, sugar beet pulp, a by-product generated in large quantities by sugar factories, presents a unique opportunity for valorization (Niskanen et al. 2024).

Sugar beet (*Beta vulgaris*), a globally cultivated agricultural crop, primarily serves as a source of sucrose for the sugar industry. During sugar beet processing, sucrose extraction results in the formation of a



Scheme 2 Cycloaddition of CO 2 to epoxide

significant byproduct called sugar beet pulp. Traditionally considered waste, this residual material retains a portion of glucose. Remarkably, sugar beet pulp can be utilized as a valuable glucose source, playing a pivotal role as a precursor in the synthesis of carbon structures (Singh et al. 2020). Agro-industrial wastes, like sugar beet, are valuable sources of renewable materials, which can be converted into valuable products such as catalysts. Biomass-based catalysts are known to be renewable sources of energy, eco-friendly, non-toxic, cost-effective, and biodegradable which could be a suitable alternative to conventional catalysts (Tang et al. 2018). In our study, we present the design and synthesis of a spherical carbon nanocatalyst derived from sugar beet pulp (SCNS). This catalyst demonstrates efficacy in converting epoxides and CO₂ (an industrial pollutant) into cyclic carbonates (Scheme 3).

The significant advantage of this approach lies in its worldwide applicability. Sugar beet cultivation and sugar production take place in various countries, spanning diverse climates and conditions. Consequently, the abundance of glucose-rich sugar beet waste is not confined to specific regions. This renders the production of spherical carbon from sugar beet waste an accessible and environmentally sustainable choice for researchers and industries globally. By converting sugar beet factory waste into an organo-nanocatalyst for the synthesis of cyclic carbonates, we aim to demonstrate the potential of utilizing agricultural waste for the development of sustainable catalytic processes. The focus of this study is to develop a method for the preparation of a cost-effective catalyst from sugar factory wastes to turn CO_2 into cyclic carbonates as valuable products of this reaction.

One of the key attributes of the SCNS catalyst is its high porosity and morphological stability, achieved through the calcination process. This process aligns the structure of the final catalyst more closely with that of carbon. The catalyst's efficiency and performance are enhanced during the reaction process. Additionally, the presence of hydroxyl groups on the SCNS catalyst's surface enables it to donate hydrogen bonds, which promotes reactivity toward producing the cyclic carbonates due to the –OH group on the surface of SCNS that could polarize and accelerate the epoxide ring opening (Khodabakhshi et al. 2021 Jan; Alassmy et al. 2020; Xie et al. 2019).

Therefore, the metal-free oragno-nanocatalyst with high activity is an excellent candidate for the heterogeneous catalytic chemical fixation of CO_2 without environmental pollution.

Multiple techniques exist for the synthesis of carbon spheres, including chemical vapor deposition pyrolysis (Du et al. 2019), hydrothermal processes (Wu et al. 2021), and solvothermal methods (Huo et al. 2023). Notably, hydrothermal carbonization (HTC) stands out as an exceptional thermochemical approach. HTC efficiently transforms large sugar molecules into valuable products, offering an economical, straightforward, and environmentally friendly route for preparing uniform carbon nanospheres (Nieves et al. 2016).



Scheme 3 The synthetic procedure of the sugar beet-derived carbon nanosphere (SCNS) catalyst

Experimental methods

Materials

All reagents and chemicals were used without further purification. Sugar beet pulp was obtained from a sugar beet factory in Khorasan-e Razavi, Iran. Sulfuric acid (95–97%) (H₂SO₄) used in this study was purchased from Sigma-Aldrich and Merck. Deionized water with a resistivity of 18.2 M Ω cm⁻¹ was used in all experiments.

Synthesis method

Preparation of carbon nanospheres by hydrothermal process

Sugar beet pulp is a valuable precursor that can be utilized in the synthesis of activated carbon nanospheres through a hydrothermal process. In a typical procedure, 5 g of sugar beet pulp, serving as the carbon source, was combined with 17 mL of deionized water and subjected to sonication for a duration of 10 min. Following this, 0.6 mL of H_2SO_4 was added into the resulting solution, which was subsequently transferred into a Teflon-coated stainless-steel autoclave with a capacity of 50 mL. The autoclave was then maintained at a temperature of 200 °C for a period of 40 h. Once the autoclave had cooled to room temperature, the product was filtered, washed with deionized water until colorless water was obtained (to remove remaining organic pollutants), and dried at 100 °C for a duration of 24 h. The resulting carbon nanospheres, derived from sugar beet pulp (referred to as SCNS), were obtained by subjecting the as-synthesized carbon nanospheres to calcination at a temperature of 850 °C for a duration of 2 h under a nitrogen atmosphere. The heating ramp rate employed during this process was 5 °C per minute. The calcination process was employed in the synthesis of this catalyst for dual purposes: firstly, to activate and develop its pore structure, and secondly, to stabilize the carbon surface.

Catalytic cycloaddition of carbon dioxide with epoxides in the presence of SCNS

The reaction involving the combination of CO_2 and epoxides was carried out in a 50-mL high-pressure stainless-steel reactor that was equipped with a magnetic stirrer bar. To the stainless reactor, epoxy styrene (8 mmol, 1.312 g) was added along with a mixture of (0.8 mmol, 0.025 g) and SCNS catalyst (0.01 g). The reaction mixture was heated to 85 °C in a sealed reactor under a 5-bar pressure of CO_2 . This heating process was conducted in an oil bath directly on a stirred/ hot plate at a speed of 400 rpm. Subsequently, the CO_2 was gradually released from the reactor after reducing the temperature of the reactor. After the completion of the reaction, the catalyst was separated by simple filtration and washed with ethyl acetate (3×10 mL). The catalyst was dried at 100 °C overnight in preparation for the next run. Following the extraction process of the product with ethyl acetate, the crude product was purified by column chromatography using ethyl acetate/n-hexane (3:10) (0.148 g, 91% yield).

Results and discussion Catalyst characterizations

The morphology and structure of the synthesized SCNS catalyst were verified in the current investigation through the utilization of FE-SEM (field emission scanning electron microscopy) and TEM (transmission electron microscopy) techniques. Furthermore, the elemental composition of the synthesized SCNS catalyst was determined using EDX (energy-dispersive X-ray spectroscopy) analysis. The functional groups present on the surface of the synthesized SCNS catalyst were examined using FT-IR (Fourier transform infrared) spectroscopy. The Raman spectrum was recorded at the ambient temperature using a Renishaw RM2000 Raman microspectrometer using 532 nm laser irradiation. X-ray powder diffraction (XRD) was conducted using a Bruker D4 X-ray diffractometer (Ni-filtered Cu KR radiation, 40 kV, 30 mA). The surface area, pore volume, and average pore size of samples were measured by Brunauer-Emmett-Teller (BET) and the Barrett-Joyner-Halenda (BJH) approaches used by the Toosnano, GasSorb I instrument using nitrogen physical absorption. The thermal stability of the SCNS catalyst was confirmed by conducting TGA (thermogravimetric analysis) to monitor the weight change as the sample was heated at a constant rate by an SDT Q600 V20.9 Build 20 instrument.

To study the morphology of the synthesized sugar beetderived carbon nanospheres (SCNS) catalyst, FE-SEM and TEM analyses were utilized. It is evident from Fig. 1a that the prepared SCNS exhibited size-tunable spherical morphology, and the nanospheres had excellent dispersity. Further analysis with TEM depicted the presence of spherical SCNS with a diameter of about 500 nm, as well as the uniform structure of the catalyst (Fig. 1b). The recycled SCNS catalyst underwent SEM analysis, and the findings are depicted in Fig. 1c, revealing negligible alterations in catalyst morphology.

The presence of carbon and oxygen as major elements in the SCNS catalyst was confirmed through the use of an SEM instrument equipped with an EDX detector. The elemental composition of the SCNS catalyst was determined, and the result of this analysis is shown in Fig. 2.

FT-IR spectroscopy of sugar beet pulp (Fig. 3a), as-prepared SCNS catalyst (Fig. 3b), SCNS catalyst (after calcination) (Fig. 3c), SCNS catalyst after vacuum furnace (Fig. 3d), and the recycled SCNS catalyst after 5 catalytic cycles (Fig. 3e) was employed to confirm the functional groups on the surface of the samples, their chemical structure, and to compare the changes made in each step. The identification of prominent absorption peaks



Fig. 1 SEM images of the SCNS catalyst (a), TEM image of the SCNS catalyst (b), and SEM images of the recycled catalyst after 5 catalytic cycles (c)

ranging from 3400 to 2800 cm⁻¹, which correspond to the stretching vibrations of the –OH and –CH groups, respectively, serves as a confirmation of the chemical composition of sugar beet pulp (Fig. 3a).

The bands between 1600 and 1800 cm⁻¹ are typically associated with identifying different types of pectin, and the absorption bands at 1730–1745 cm⁻¹ and 1630–1650 cm⁻¹ are indicative of free and esterified carboxyl groups, respectively. Additionally, the distinct absorption peak observed at 1740 cm⁻¹ in sugar beet pulp is attributed to the stretching vibration of acetyl and uronic ester groups originating from hemicelluloses or the ester linkage of carboxylic groups present in ferulic and p-coumaric acids of lignin (Yang et al. 2018). The broad peaks at 1058 cm⁻¹ are attributed to ether linkages (C–O–C) from lignin or hemicellulose, while the peak at 870 cm⁻¹ corresponds to beta-glycosidic linkages of the glucose ring in cellulose (Sidi-Yacoub et al. 2019). After calcination, which was performed to achieve the spherical carbon structure of the catalyst and eliminate polar functional groups, FT-IR analysis was conducted, and the results are presented in Fig. 3c. A comparison of the spectra before and after calcination, as shown in Fig. 3b and c, reveals structural changes in the catalyst, including a reduction in the intensity of the absorption bands. In the FT-IR spectrum of the SCNS catalyst, the presence of C–H stretching vibration bonds is evident at 2888 cm⁻¹, while the peaks at 3443 and 1629 cm⁻¹ correspond to the stretching vibration and bending mode of the –OH group, respectively (Fig. 3c). Although calcination removes some functional groups,



including hydroxyl groups, it does not eliminate them entirely. Some hydroxyl groups remain, influencing the surface chemistry of the activated carbon. These residual functional groups can engage in adsorption and interact with reactants during the catalytic process. By comparing the absorption bands associated with the hydroxyl functional group in the spectra (Fig. 3a, b, c and d), changes in intensity can be observed, reflecting the structural evolution of the catalyst at different steps. In spectrum d, which corresponds to the catalyst after treatment in a vacuum furnace, a broad absorption band appears in the 3200-3500 cm⁻¹ range. Although the intensity of this band has decreased compared to the previous step, it has not been entirely eliminated. The final spectrum presented pertains to the recycled catalyst after five catalytic cycles. A comparison with the spectrum of the catalyst's final structure reveals only minor changes in the intensity of the absorption bands in certain regions (Fig. 3e).

Raman spectroscopy was used to understand the degree of graphitization of sugar beet pulp-derived carbon nanosphere catalyst. The Raman spectra of SCNS catalyst show two remarkable peaks centered at approximately 1300 (D band) and ~1540 cm⁻¹ (G band). The D band can be attributed to the presence of sp³ hybridized carbon atoms, while the G band is an indicator of conjugated sp² carbon atoms. Both D and G bands are almost broad, meaning a rather amorphous structure (Fig. 4) (Gür et al. 2022; Zhang et al. 2019).

The crystalline structure of the SCNS catalyst and recycled SCNS catalyst was analyzed using the XRD technique, with the findings displayed in Fig. 5a and b. The XRD pattern reveals two clear diffraction peaks located at precise angles, $2\theta = 25^{\circ}$ and 44° , which can be attributed to the (0 0 2) and (1 0 0) reflections of the amorphous structure of the SCNS catalyst, respectively (Lu et al. 2018). Additionally, XRD analysis was performed on the recycled catalyst. As shown in Fig. 5b, the crystal structure of the SCNS catalyst remains largely unchanged.

The nitrogen adsorption-desorption technique was employed to determine the specific surface area of the SCNS catalyst (Fig. 6). According to the IUPAC classification, BET analysis reveals a type IV isotherm, typical of mesoporous materials with pore diameters ranging from 2 to 50 nm. A notable characteristic of this isotherm is the sharp rise in adsorption at higher relative pressures, due to capillary condensation within the mesopores. The hysteresis loop in the graph indicates a discrepancy between the adsorption and desorption paths, suggesting a type H1 hysteresis. The corresponding average surface areas, pore volumes, total pore volume, mean pore diameters, and adsorption average pore diameter of the SCNS catalyst are provided in Table 1. The significant specific surface area, approximately 1464 m^2/g , is attributed to the chemical activation of the SCNS catalyst following the hydrothermal process, which greatly influences the interaction between epoxides and CO₂.

The BJH method was employed to inspect the pore size distribution (PSD) of the SCNS catalyst as shown in Fig. 7. The BJH diagram confirms that the SCNS catalyst possesses a mesoporous structure with a high surface area, making it suitable for catalytic and adsorption applications.



Fig. 3 AQ FT-IR spectrum of sugar beet pulp (a), As-prepared SCNS catalyst (before calcination) (b), SCNS catalyst (after calcination) (c), SCNS catalyst after vacuum furnace (d), and recycled SCNS catalyst after 5 catalytic cycles (e)

The thermogravimetric analysis (TGA) was employed to investigate the thermal stability of the SCNS catalyst. The analysis was conducted within a temperature range of 25 to 700 °C, with a heating rate of 10 K min⁻¹. The obtained results, as depicted in Fig. 8, indicate two significant weight losses. The first weight loss, occurring between 50 and 150 °C, can be attributed to the removal of adsorbed water molecules from the surface of SCNS. This accounts for a reduction in weight of approximately 10%. The second weight loss, observed between 150 and 550 °C, is attributed to the complete decomposition of the organic components present in SCNS. This decomposition leads to a weight reduction of around 30%. Furthermore, the analysis reveals that the collapse of carbon within the SCNS structure occurs at temperatures exceeding 550 °C.

Catalytic performance of SCNS catalyst in carbon dioxide fixation

Following the confirmation of the successful synthesis of the SCNS catalyst, we proceeded to conduct experimental investigations on its catalytic activity in the CO_2 fixation reaction. The objective of this study was to explore the chemical fixation of CO_2 , as a safer alternative to using toxic reagents like phosgene, by introducing epoxides and a catalytic quantity of the co-catalyst tetrabutylammonium bromide (TBAB). The aim was to synthesize five-membered cyclic carbonates, as depicted in Scheme 4.

It has been demonstrated that halide ions, as cocatalysts, are required to improve the performance of the heterogeneous catalyst in the CO_2 fixation reaction (Jin et al. 2019). Information regarding the optimum



Fig. 4 Raman spectrum of the SCNS catalyst



Fig. 5 XRD pattern of SCNS catalyst (a) and recycled SCNS catalyst after 5 catalytic cycles (b)



Fig. 6 N₂ adsorption and desorption isotherms of the SCNS catalyst

 Table 1
 Specific surface area (SBET), pore volume, and mean pore diameter of SCNS catalyst

Sample	BET surface area (m ⁻¹	Total pore volume (cm ³ g ⁻¹)	BJH adsorption cumulative volume of pores (cm ³ g ⁻¹)	Mean pore diameter (nm)	Adsorption average pore diameter (Å)
SCNS catalyst	1464	0.36	1.01	3.38	33.81



Fig. 7 BJH adsorption of SCNS catalyst



Scheme 4 The chemical fixation of CO₂ with epoxides in the presence of SCNS catalyst

reaction conditions and the assessment of the catalytic performance of SCNS in the production of cyclic carbonates through CO_2 cycloaddition with styrene oxide (SO) was acquired as a representative reaction. Various factors such as solvent impact, catalyst quantity, and reaction temperature were examined to enhance the reaction conditions, and the outcomes are presented in Table 2.

In the initial examination, the catalytic activity of SCNS in solvent-free conditions at 100 °C and 10 bar CO_2 pressure in the absence of TBAB was analyzed for efficient synthetic CO_2 fixation (Table 2, entry 1). Additionally, the CO_2 fixation to cyclic carbonates with SO was evaluated under the same reaction conditions in the presence of TBAB but without the SCNS catalyst (Table 2, entry 2). These outcomes indicated that the utilization of both the catalyst and co-catalyst was crucial for achieving effective performance and high efficacy throughout the reaction process. In the next experiment, the obtained result showed that the presence of the SCNS catalyst along with TBAB

led to a good yield of the cyclic carbonate in a short period (Table 2, entry 3). Furthermore, an examination was conducted on the impact of various solvents on the production of the related product (Table 2, entries 4-8). The DMF solvent exhibited the highest product yield among the solvents employed, owing to its Co-catalytic contribution in the reaction process. Moreover, this solvent generally offers a favorable environment for the reaction. Conversely, water proved to be less efficient as a solvent in this reaction medium compared to other solvents. Not only was it unsuitable for the reaction, but it also resulted in the formation of by-products. Although the solvents exhibited favorable results, the desired product was still achieved with a significant yield in the absence of any solvent (Table 2, entry 3). The optimization of product yields, and the acceleration of the reaction rate are heavily influenced by the quantity of catalyst used. The range of catalyst loading that resulted in the highest yield of the desired product was determined to be between 0.01 and 0.02 g. However, a decrease in product yield was observed

Entry	Catalyst (g)	Co-catalyst	Solvent	Temp (°C)	Time (h)	Yield (%)	P (bar)
1	0.01	-	_	100	24	Trace	10
2	-	TBAB	-	100	24	< 20	10
3	0.01	TBAB	-	100	4	91	10
4	0.01	TBAB	H ₂ O	85	4	60	10
5	0.01	TBAB	<i>n</i> -hexane	85	4	70	10
6	0.01	TBAB	Toluene	85	4	66	10
7	0.01	TBAB	CH ₂ Cl ₂	85	4	78	10
8	0.01	TBAB	DMF	85	4	88	10
9	0.005	TBAB	-	100	4	45	10
10	0.005	TBAB	-	100	8	62	10
11	0.02	TBAB	-	100	4	91	10
12	0.01	TBAB	-	Rt	24	36	10
13	0.01	TBAB	-	50	24	61	10
14	0.01	TBAB	-	50	10	49	10
15	0.01	TBAB	-	75	8	65	10
16	0.01	TBAB	-	85	5	91	10
17	0.01	TBAB	-	85	4	91	10
18	0.01	ТВАВ	-	85	4	91	5
19	0.01	TBAB	-	85	4	83	4
20	0.01	TBAB	-	85	4	41	3
21	0.01	TBAB	-	85	8	23	2
22	0.01	TBAB	-	85	10	12	1

Table 2	Optimization o	f reaction conditio	ns for the c	ycloaddition of CO	y with styrene oxide in t	ne presence of SCNS cata	alyst ^a
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^a TBAB (0.8 mmol), styrene oxide (8 mmol), and in the case of solvent use, the volume used is 5 mL

when the catalyst loading fell below 0.01 g (Table 2, entry 9). Additionally, the impact of moderate temperatures (25–85 °C) (Table 2, entries 12–17) and the pressure of CO₂ (1–10 bar) on the formation of cyclic carbonate was investigated (Table 2, entries 18–22). Elevating the temperature to a specific degree enhances the effectiveness of the reaction by boosting gas permeability and elevating catalytic activity. The optimal conditions for achieving the highest yield of the desired product were found to be at a temperature of 85 °C and a pressure of 5 bar (Table 2, entry 18).

The expansion of the formation of five-membered cyclic carbonates through the coupling of carbon dioxide with various epoxide substrates was achieved by understanding the optimal reaction conditions. The results presented in Table 3 demonstrate that the conversion of CO_2 to cyclic carbonates using epichlorohydrin, which includes electron-withdrawing groups such as oxygen, chlorine, and bromine atoms, yielded the desired products in a short period of time and with excellent isolated yields (Table 3, entries 2, 3, 6). The SCNS catalyst exhibited satisfactory activity in the production of cyclic carbonates with long alkyl chains in the epoxide (Table 3, entry 4). However, when internal epoxides with a high steric hindrance effect were used, the corresponding cyclic carbonates were obtained with lower yields compared to similar reactions.

Furthermore, the primary techniques employed for determining the structure elucidation of the products were FT-IR, ¹H NMR, and ¹³C NMR spectroscopy, as stated in the supporting information. Research indicates that catalysts with a significant presence of hydroxyl groups create Brønsted acid sites, which are highly effective in activating the oxygen atom within the epoxide ring (Yingcharoen et al. 2019; Liang et al. 2017; Zhu et al. 2018). Additionally, TBAB serves as the nucleophilic group, facilitating the activation of the carbon atom within the epoxide ring (Sun et al. 2014).

Initially, the epoxy ring underwent activation via a hydrogen bond with the hydroxyl group on the SCNS catalyst's surface to facilitate the cycloaddition reaction with CO_2 (I). Following this, the Br⁻ anion, functioning as a nucleophilic group, attacked the less hindered carbon of the epoxide, resulting in the opening of the ring (II). The hydroxyl groups present on the catalyst's



 Table 3
 Cycloaddition of CO 2 with various epoxides in the presence of SCNS



Scheme 5 The proposed mechanism of catalyzed CO₂ fixation reaction in the presence of SCNS

surface then facilitated the connection of carbon dioxide to the catalyst through hydrogen bonding, bringing the reactants into close proximity. Subsequently, the bromo-alkoxide, generated from the interaction between the oxygen anion of the opened epoxide and CO_2 (III), underwent transformation into cyclic carbonate by eliminating the bromine anion (IV) (Scheme 5).

To assess the reusability and renewability of the SCNS catalyst, the model reaction was conducted

under optimized conditions as outlined in Table 2, entry 18. After each run, the reaction mixture was cooled to room temperature, and ethyl acetate was added to the mixture. By employing a simple filtration process, the catalyst was separated from the reaction mixture, followed by washing with diluted ethyl acetate (2×5 mL). Subsequently, the catalyst was dried under vacuum at 80 °C overnight to prepare it for the next execution. Remarkably, the SCNS catalyst exhibited consistent catalytic activity and selectivity over



Fig. 9 Recycle of the SCNS catalyst in the CO₂ fixation reaction

Entry	Catalyst	Temp. (°C)	P (bar)	Time (h)	Yield (%)	Ref.	
1	[HEBim][Asp]	140	20	12	93.5	(Yue et al. 2019)	
2	[HOBU]I	80	10	7	90	(Fanjul-Mosteirín et al. 2019)	
3	Amb-OH-i910	80	10	18	66	(Alassmy et al. 2020)	
4	Fe/ZIF	120	7	8	97	(Hu et al. 2020)	
5	lignin-ChCl-PABA	110	10	3	90	(Xiong et al. 2020)	
6	Zn complex (Zn(HBA) ₂)	80	1	36	81	(Ge et al. 2022)	
7	Poly (HCO ₃ -OH-2)	100	1	48	86	(Jiang et al. 2023)	
8	C4-500 (chitin)	150	30	15	>90	(Polidoro et al. 2022)	
9	Sugar beet-derived carbon nanosphere	85	5	4	91	This work	

Table 4 Comparison of SCNS catalyst with reported catalysts in the cycloaddition of CO₂ into styrene oxide

five consecutive cycles, indicating its commendable durability in the reaction, as depicted in Fig. 9.

The comparative evaluation of the as-synthesized catalyst's efficiency was carried out against different catalytic systems reported in the literature for the conversion of CO_2 with epoxides under various catalytic conditions (Table 4). It is a well-established fact that well-preserved catalysts exhibit excellent catalytic activity. However, it has been observed that SCNS, being a sustainable, cost-effective, readily available, environmentally friendly, and impressive catalyst, demonstrates higher efficiency in the CO_2 fixation reaction compared to almost all the catalytic systems documented in Table 4.

Conclusions

In conclusion, the utilization of organo-nanocatalyst that derived from sugar beet waste has proven to be a promising and efficient approach for the synthesis of cyclic carbonates through the chemical fixation process. The structure of the nanocatalyst was confirmed by several complementary techniques such as FE-SEM, TEM, EDX, FT-IR, Raman, XRD, BET, and TGA. The presence of hydroxyl functional groups on the catalyst's surface (act as Brønsted acid) enhances its catalytic activity by establishing hydrogen bonds with reactants, leading to increased reaction rates. The analysis findings indicate that the organo-nanocatalyst's effectiveness in facilitating the desired reactions is attributed to its specific surface area, which is a result of the organo-nanocatalyst's high porosity. The successful production of corresponding five-membered cyclic carbonates under mild reaction conditions, including solvent-free conditions and a moderate temperature of 85 °C, underscores the potential of this organo-nanocatalyst for sustainable and environmentally friendly chemical transformations. Furthermore, the SCNS catalyst can be successfully reused for at least five repeated cycles without an appreciable loss of activity in $\rm CO_2$ epoxidation to cyclic carbonates. Overall, this study highlights the value of utilizing bio-based catalysts in catalysis for advancing green chemistry practices and achieving efficient synthesis of valuable products.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s40712-024-00178-4.

Additional file 1: Supplementary Materials.

Acknowledgements

The authors express their appreciation for the partial funding received from the Research Council of Ferdowsi University of Mashhad, which supported this study (Grant No. p/3/58887).

We appreciate the Microsoft Copilot assistant for providing editorial support for this manuscript.

Authors' contributions

We strongly encourage authors to include author contributions and recommend using CRediT for standardized contribution descriptions. For example, Fateme Moazen: Investigation, Methodology, Data curation, Writing-Original draft preparation. Hossein Eshghi: Supervision, Conceptualization, Writing-Reviewing and Editing. Hossein Torabi: Writing-Reviewing and Editing.

Funding

The Research Council of Ferdowsi University of Mashhad supported this study (Grant No. p/3/58887).

Availability of data and materials

The supporting file was uploaded to the confirmation of the structure of the products. The products were described in the experimental section through the analysis of their FT-IR, 1 H NMR, and 13 C NMR spectra.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

All of the authors agree with the publication.

Competing interests

The authors declare that they have no competing interests.

Received: 18 June 2024 Accepted: 4 September 2024 Published online: 20 September 2024

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