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*Research Article*

An Efficient Perlite/Fe₃O₄ Magnetic Nanocatalyst for Carbon Dioxide Fixation Under Solvent-free Conditions at Atmospheric Pressure of CO₂

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CO₂ fixation,
Magnetic nanocatalyst,
Epoxide,
Solvent-free.**ABSTRACT**

The increasing reliance, on fuels has led to an increase in the levels of carbon dioxide causing concerning outcomes like climate change and global warming. In response to this problem, there is a solution; CO₂ fixation. In this study, Fe₃O₄ became changed with the mineral perlite to create magnetic nanomaterials. Fe₃O₄/Perlite magnetic nanomaterials had been created the use of a co-precipitation technique. In this research, we have presented Perlite/Fe₃O₄ magnetic nanocatalyst for the transformation of carbon dioxide and epoxides into cyclic carbonates without using solvents at atmospheric pressure of CO₂. The Perlite/Fe₃O₄ magnetic nanocatalyst and products were examined using analysis methods like FTIR, FE-SEM, VSM, EDAX, XRF, NMR, and TGA analysis. The analysis showed the success development of Fe₃O₄/Perlite with the favored magnetic nanoparticles on its surface. The catalyst exhibited excellent efficiency in generating cyclic carbonate through the activation of CO₂ and epoxide. Among the epoxides, 1-Chloro-2,3-epoxypropane completed the best conversion fee of 98%, finishing the system in a fast 4.5 h. This approach offers advantages, including mild reaction conditions, solvent-free, the atmospheric pressure of CO₂, high yields, simple workup process, inexpensive catalyst, environmentally friendly, short reaction times, and convenient catalyst removal using an external magnet. The catalyst can be reused multiple times, up to 5 cycles, without a significant decrease in its efficiency.

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1. Introduction

People, societies, industries, etc., use fossil fuels for energy, and this has caused an increase in the amount of carbon dioxide, which contributes to climate change. Indiscriminate emissions and rapid growth of carbon dioxide have caused the greenhouse effect. Carbon dioxide is a gas that absorbs and emits heat. [1]. Reducing the produced carbon dioxide is an important and necessary issue for which various methods have been proposed so far. One of the options is to convert carbon dioxide into fine chemicals. Referring to the process of formation of cyclic carbonates through the addition reaction of epoxides with carbon dioxide in the presence of a catalyst [2]. Fabrication of cyclic carbonates using catalytic conversion of carbon dioxide not only reduces greenhouse gases but also increases economic value by producing valuable chemicals. Use in lithium batteries, synthesis of polycarbonates, important intermediates in the synthesis of ethylene glycol, and polyurethane, pharmaceutical precursors, and polar solvents are the various applications of cyclic carbonates.

[3-6]. Numerous heterogeneous and homogeneous catalysts have been employed for CO₂ fixation. However, uniform (homogeneous) Catalysts come with drawbacks, such as challenges in separation and recovery from the solvent. Thus, researchers are Oriented to utilizing heterogeneous catalysts, considering their native advantages. Diverse catalysts, including silicates, metal complexes, modified polymers, metal-organic frameworks, and ionic liquids, have been applied in CO₂ fixation processes [7-11]. Despite this, employing catalysts and co-catalysts at room pressure and temperature proves to be mighty for cycloaddition reactions of cyclic carbonates. A challenge that is still significant for researchers. Inventing and designing nanocatalytic systems that can easily absorb and stimulate CO₂ [12-15].

perlite is a hydrous rhyolitic stone with a pearly luster. Perlite is an economical, and naturally occurring volcanic substance, a type of mineral oxide mixture comprising approximately 70–75% SiO₂ and 12–15% Al₂O₃. Perlite is distinguished by its elevated surface area, significant porosity, robust adsorption capabilities, environmentally conscious features, exceptional resilience to heat and chemicals, substantial porosity, lightweight, cost-effectiveness, minimal toxicity, and ease of handling. The highly porous composition and silica content of perlite open up possibilities for diverse applications in industries such as refractory, metallurgical, paint, petrochemical, oil, building, and gas. It finds utility in insulation, refrigeration, heating tanks, moisture control, and as an absorbent [16-20]. Iran has abundant reserves of perlite. There are perlite mines in the center, west, and east of Iran. According to the available reserves and the technical characteristics of Iran's perlite, there is the potential to become one of the world's largest perlite suppliers.

On the one side, the chemical, electrical, structural, and magnetic properties of nanomaterials, and on the other side, the great potential of nanomaterials to create new and innovative products in many fields, which have attracted the attention of many researchers in the last 20 years. Due to these interests, nanomaterials have continuously progressed over the years, and now nanomaterials are made with complex compositions, adjustable surface charges, diverse shapes and morphologies, precisely defined dimensions, and appropriate physical and chemical properties [21-22]. The unique properties of nanoparticles made from magnetite (Fe₃O₄) have garnered interest. Magnetite nanoparticles have properties such as high surface-to-volume ratio, biocompatibility, and superparamagnetic properties, which lead to their wide applications. In fields such as water and

wastewater treatment from pollutants, drug delivery, medical imaging, and catalyst design for various organic reactions, nano-sized magnetic materials with high magnetic properties, chemical stability, minimal toxicity, wide surface selectivity, and spreadability have been used. In different solvents, a significant number of surface active sites, availability, recyclability, and ease of separation under an external magnetic field are distinguished. Fe₃O₄ exhibits a high level of chemical reactivity and has shown excellent performance as a catalyst in numerous reactions. These materials stand out as essential solid supports for loading heterogeneous catalysts. Functionalization of Fe₃O₄ by creating regions of chemical coordination enhances conjugation and ensures chemical stability [23-25]. Primarily because of their characteristics, such as their ability to remain chemically stable, lack of toxicity, profitable efficiency, and uncomplicated manufacturing processes, silica-coated magnetite nanosupports have attracted considerable attention in the field of various solid nanomaterials. Due to the profitable use of these materials, this focus mainly originates from the industrial sectors. Furthermore, their magnetic separation offers an alternative to laborious filtration and centrifugation techniques, ultimately saving time, energy, and catalysts [26-31].

Based on our previous research in the catalytic properties of perlite and functionalized perlite, herein, we introduce an effective heterogeneous nanocatalyst based on perlite/Fe₃O₄ for use in the CO₂ fixation reaction. The perlite/Fe₃O₄ composite nanocatalyst has been employed to directly convert CO₂ and epoxides into cyclic carbonates in conditions of gentle reaction. Notably this catalytic system implements CO₂ fixation under low pressure of CO₂ in the absence of solvents, making the present procedure both environmentally sustainable

and economical, while maintaining its simplicity and effectiveness.

2. Experimental procedure

2.1. Materials and method

Acquiring expanded perlite from Alvand Metal Chemistry CO, Qazvin, while other chemicals procured from Aldrich and Merck chemical firms were utilized without any further purity adjustments.

2.2. Characterization and instrumentation

Characterization of the catalyst involved the use of various analytical techniques. Field emission scanning electron microscopy (FE-SEM) and Energy-dispersive X-ray spectroscopy (EDS) were conducted on an electron microscopy MIRA3TESCAN-XMU with detector SAMX. Thermogravimetric analysis (TGA) was carried out with Perkinelmer TGAL001. Fourier transform infrared (FT-IR) spectra were obtained using a Bruker Tensor 27 spectrometer, with absorbencies reported in cm⁻¹. The chemical analysis of perlite was accomplished by XRF (Philips 1480). Using a Bruker DRX-300 AVANCE, the ¹H and ¹³C NMR spectra were logged at a frequency of 300 MHz. Melting point was conducted using an Electrothermal 9100 apparatus. VSM device Model force-MDKF made by a Magnetic Danesh Pajoh Co, was carried out.

2.3. Purification and customization of expanded perlite powder

To remove impurities in perlite. The amount of 10 g of perlite was suspended in 200 ml and stirred for 20 hours at room temperature. Then the mixture was filtered and washed four times with ethanol. After that, it was heated with 4 N sodium hydroxide solution for 1 hour. To remove sodium hydroxide, after being filtered, the product underwent four thorough washings with distilled water. The color of perlite at this stage was gray. It was dried in the oven



Figure 1. (a) Perlite/ Fe_3O_4 magnetic nanocatalyst (b) Perlite/ Fe_3O_4 magnetic nanocatalyst with an external magnet, (c) perlite (d) perlite with an external magnet

for 24 hours at a temperature of 130 °C and its color changed to white [32].

2.4. Synthesis of Perlite/ Fe_3O_4 nano-magnetic

A co-deposition method (carried out in an inert argon atmosphere) was investigated for the synthesis of magnetic nanoparticles supported by perlite containing Fe_3O_4 . After purification, 6 g of perlite was dispersed in 100 ml of deionized water along with a solution containing 2 mmol of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 4 mmol of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$. The mixture was stirred vigorously at 80 °C for 30 min. Then, under an argon atmosphere and with continuous stirring, 10 ml of 8M ammonium hydroxide (NH_3) solution was added dropwise to induce precipitation. The reaction was continued for another 30 min and then terminated by turning off the heat and allowing the system to cool to room temperature. The resulting blackish brown perlite- Fe_3O_4 composite was separated using magnetic separation. To remove residual impurities and unreacted precursors, the perlite- Fe_3O_4 composite was subjected to a three-step washing process with deionized water and ethanol, facilitated by an external magnet. Finally, the product was dried [33-34].

2.5. Process for the synthesis of cyclic carbonates with carbon dioxide fixation

The reaction vessel was initially purged with carbon dioxide (CO_2) to create an inert atmosphere for the subsequent cyclic carbonate synthesis.

In a flask, 0.014g Perlite/ Fe_3O_4 nanocomposite catalyst and 0.012g tetrabutylammonium bromide (TBAB) as co-catalyst were thoroughly mixed together. After that, the reaction vessel was carefully

closed. Next, 2mmol of epoxide was introduced into the reaction mixture with caution. The reaction commenced when the flask was submerged in an oil bath adjusted to a specific temperature.

At the same time, CO_2 infiltrated the reaction container, and stirring was executed. The evolution of the reaction was monitored through TLC (Thin Layer Chromatography) employing a blend of *n*-hexane and ethyl acetate in a 3:1 ratio as the eluent, and the outcomes were detected under UV. Following the completion of the reaction, the mixture gradually reached ambient temperature. Two segments of water, each measuring 3 ml (2×3 ml), were used to extract TBAB. The impure product was meticulously refined by passing it through column chromatography on silica gel using a blend of *n*-hexane and ethyl acetate.

3. Results and discussion

3.1. Characterization of Perlite and nano-magnetic Perlite/ Fe_3O_4

Nano-magnetic Perlite/ Fe_3O_4 catalyst was identified by using analytical methods, consisting of X-ray fluorescence analysis (XRF) and Thermogravimetric-differential thermal analysis (TGA-DTA). Fourier-transform infrared spectroscopy (FT-IR), Field emission scanning electron microscopy (FE-SEM), and energy-dispersive spectrometry (EDS).

3.2. X-ray fluorescence analysis (XRF) of perlite

The XRF of the perlite is demonstrated in Table 1. The results show that silica (70.29%) and alumina (12.78%) are the main components of perlite [35].

3.3. FT-IR spectra analysis of the Perlite and nano-magnetic Perlite/ Fe_3O_4

The FT-IR absorption peak of perlite, as illustrated in Figure 2a, the sharp peak at approximately 1633 cm⁻¹ shows the bending vibration of the -OH group.

Table 1. The chemical composition of XRF expanded perlite

Constituent:	SiO ₂	Al ₂ O ₃	K ₂ O	Fe ₂ O ₃	Na ₂ O
Percentage:	70.29	12.78	5.36	1.63	3.35
Constituent:	CaO	MgO	TiO ₂	Other	Total
Percentage:	1.03	0.14	0.154	5.26	100

The two peaks shown in the region 792 and 1059 cm⁻¹ both correspond to Si-O-Si vibration, but one of them is related to the symmetric stretching and the other is asymmetric [36]. The peak at 460 cm⁻¹ corresponds to the bending vibration of Al-O-Al.

In Figure 2b, The FT-IR of Fe₃O₄/Perlite Indicates peaks at around 471, 793, and 1041 cm⁻¹ have appeared. These three spectra with a slight shift also appeared in the infrared spectrum of perlite, which indicates the presence of silica and alumina in perlite and Nano-magnetite perlite composite. The Peak around 570 cm⁻¹ is attributed to the Fe-O which confirms the presence of nano-Fe₃O₄ in the fabricated composite. Broad bands around 3438, and 3430 cm⁻¹ belong to stretching vibration of the OH group for Si-OH, H-O-H, and Al-OH, and of perlite and Nano-magnetic Perlite/Fe₃O₄. These Outcomes confirm the fabrication of Nano-magnetic perlite/Fe₃O₄ composite [33-34].

3.5. FE-SEM analysis of the nano-magnetic Perlite/Fe₃O₄ catalyst

The regular and uniform appearance of the Nano-magnetic Perlite/Fe₃O₄ catalyst (a, b) is depicted in Figure 4, similar to the sleek surface of a wooden stick or leaf. This noticeable characteristic is marked by an abundance of condensed and densely distributed pores. The perlite-Fe₃O₄ nanoparticles exhibit spherical and hemispherical shapes, with particle sizes ranging from 18 to 23 nm [33-34].

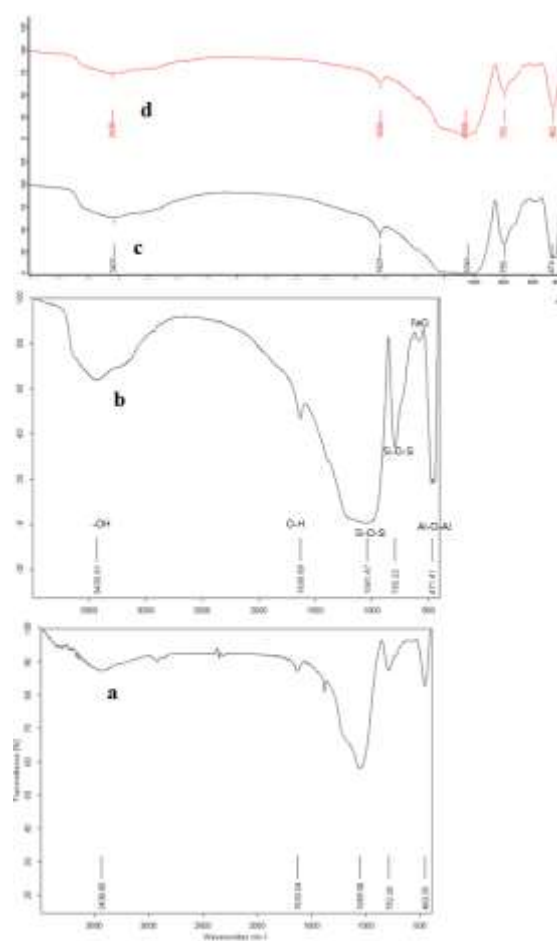


Figure 2. FT-IR (a)- of perlite, (b)- Perlite/Fe₃O₄, (c) 1st Perlite/Fe₃O₄, (d) 5th recovered Perlite/Fe₃O₄

3.6. SEM-EDX analysis of the nano-magnetic Perlite/Fe₃O₄ catalyst

The SEM-EDX chemical analysis of perlite/Fe₃O₄ nanomaterials is presented in Fig 5. As anticipated in Perlite/Fe₃O₄, the quantitative results show the detection of components such as O, Si, Al, and Fe. According to the EDX analysis, (Table2. EDX Quantitative Results) the Nano-magnetic sample of Perlite/Fe₃O₄ exhibited a composition comprising Silicon (20.65 w %), Oxygen (51.32 w %) Aluminium (5.16 w %), and Iron (22.87 w %) the range of (250-700°C) probably result from the thermal decomposition of molecular water in the restricted cell pores, the dehydroxylation of perlite, the removal of oxygen carrier groups, and transformation in the iron oxide phase [35,37].

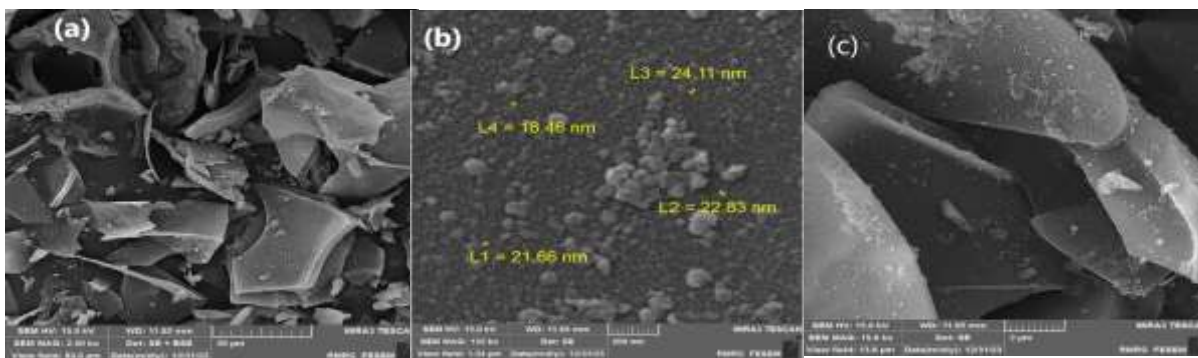


Figure 4. FE- SEM image of the Nano-magnetic Perlite/Fe₃O₄ (a, b, c) (Nanoparticles with an average size of 22 nm)

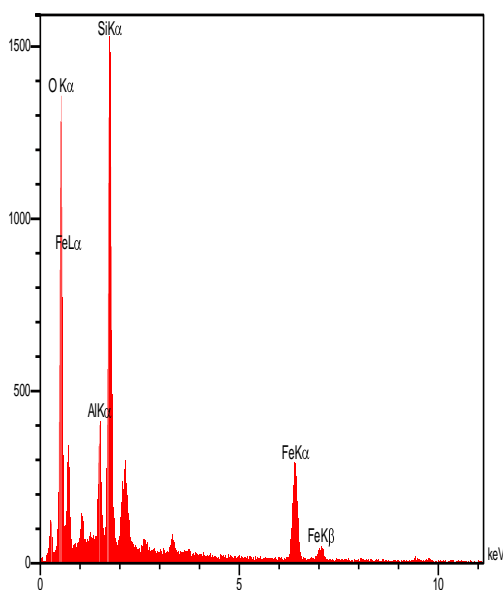


Figure 5. EDX analysis of Fe₃O₄/Perlite catalyst

Table 2. EDX Quantitative Results

Element	W%	A%
O	51.32	70.60
Al	5.16	4.21
Si	20.65	16.18
Fe	22.87	9.01
	100.00	100.00

3.7. TGA-DTA analysis of the nano-magnetic Perlite/Fe₃O₄ catalyst

The TGA-DTA for the Perlite/Fe₃O₄ nanocatalyst is depicted in Figure 6. In the temperature range (0-250 °C) weight losses of perlite/Fe₃O₄ Nanocomposite correspond to the evaporation of water molecules, the elimination of moisture absorbed by the perlite surface, and the existence of solvents adsorbed to the outer layer of the catalyst. The weight losses within

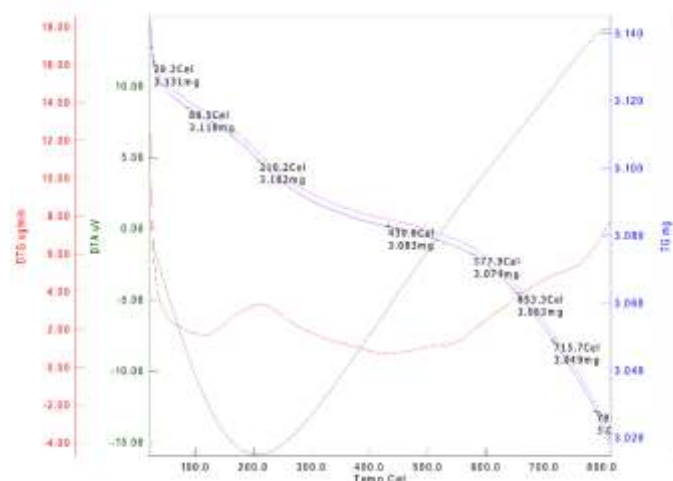


Figure 6. TGA-DTA curve of the Fe₃O₄/Perlite catalyst

3.8. VSM analysis

The Figure 7 displays the magnetic loop of perlite-magnetite nano particles. The VSM test reveals that the made perlite/Fe₃O₄ shows superparamagnetic behavior. Also, the magnetic reaction hints at the capacity to simply separate these nano particles with an outside magnet force. This results are consistent with previous research on this topic [38].

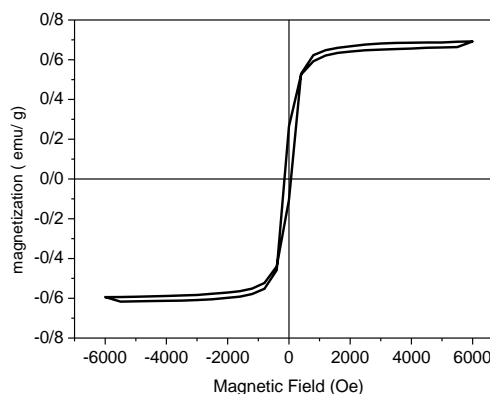
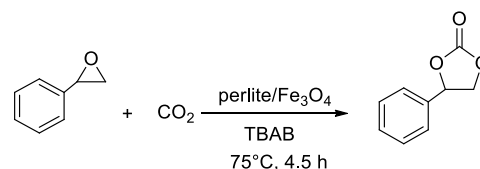


Figure 7. VSM curve of Fe₃O₄/Perlite catalyst

This paper aims to study the catalytic activity of Perlite/Fe₃O₄ nano-magnetic as a catalyst. Moreover, the research investigated the formation of cyclic carbonates through the addition reaction of epoxides with carbon dioxide in the presence of a catalyst (insertion of CO₂ into styrene oxide catalyzed by Perlite/Fe₃O₄ as a controlled reaction to synthesize cyclic carbonate). The utilization of Nano-magnetic Perlite/Fe₃O₄ as a catalyst resulted in a specific production yield. At 90°C without perlite/Fe₃O₄ nano-material as a catalyst, the model reaction did not take place under solvent-less conditions (Table 3, Entries 1) [39]. CO₂ fixation did not occur without the use of TBAB as a co-catalyst in this reaction (Table 3, Entries 2). Without TBAB, this reaction is impossible. The findings emphasize the crucial role of TBAB as a nucleophile in the reaction. Increasing the perlite/Fe₃O₄ nanocatalyst to 0.014 g and TBAB to 0.04 mmol led to a significant improvement in reaction efficiency at room temperature, as shown in (Table 3 Entries 3-6). Increasing the amount of catalyst to 0.018 g and TBAB to 0.045 mmol did not have a noticeable effect on the reaction efficiency (Table 3 Entries 7). When the temperature is increased to 60°C, there is a notable increase in the reaction yield and, as a result, its efficiency improves significantly (refer to Table 3, entry 8). The optimized conditions included using 0.014 g of Perlite/Fe₃O₄ catalyst, epoxide (2.00 mmol), TBAB (0.04 mmol), CO₂ (1 atm) for 4.5 h at 75°C (Table 3, Entries 9). perlite/Fe₃O₄ nanocatalyst has great advantages for the chemical CO₂ fixation. Because perlite is a highly porous compound [40], it has the ability to absorb CO₂. The ability to absorb carbon dioxide in the catalyst is an important feature that plays a very important role in the conversion of CO₂ to cyclic carbonates. Activation of carbon dioxide, nano-magnetite perlite catalyst activates CO₂ by creating hydrogen bonds and facilitating its conversion and reaction

efficiency. Another important feature of this catalyst is that it activates the epoxide ring by establishing a hydrogen bond with oxygen and makes the ring more reactive for nucleophilic attack [41]. Notwithstanding, owing to its morphology, high surface area, and porosity, the Perlite/Fe₃O₄ catalyst demonstrated remarkable efficiency when the same reaction conditions were applied. The final product was fabricated in exceptional yield after 4.5 hours. The outcomes reveal that the Perlite/Fe₃O₄ multi-active site nanocatalyst plays a vital role in capturing and integrating CO₂ into epoxides, facilitating the production of cyclic carbonates within the CO₂ fixation. The optimal conditions for the Synthesis of styrene carbonate from styrene oxide and CO₂ were determined by checking different temperatures, times, and catalyst amounts. This research explored the synthesis of cyclic carbonate using Epoxide derivatives under identical conditions. At 75°C and CO₂ pressure of 1 atm, the most desirable result was detected with the Epichlorohydrin derivative. This process achieved a notable 98% product yield over a span of 4.5 hours. (Table 4, Entries 1–4).



Scheme 1. Model reaction




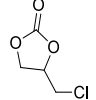
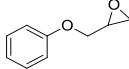
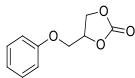
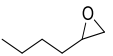
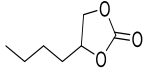
Table 3. Optimization of reaction conditions

Entry	Catalyst	Amount of Catalyst(g)	TBAB	Tim(h)	T(°C)
1	No catalyst	0	0	7	90
1		0			
2	perlite-Fe ₃ O ₄	0.012	0	6	r.t
1	(CO ₂ balloon)	0			
3	perlite-Fe ₃ O ₄	0.005	0.02mmol	6	r.t
1	(CO ₂ balloon)	57			
4	perlite-Fe ₃ O ₄	0.01	0.02mmol	6	r.t
1	(CO ₂ balloon)	61			
5	perlite-Fe ₃ O ₄	0.01	0.04mmol	6	r.t
1	(CO ₂ balloon)	67			
6	perlite-Fe ₃ O ₄	0.014	0.04mmol	6	r.t
1	(CO ₂ balloon)	70			
7	perlite-Fe ₃ O ₄	0.018	0.045mmol	6	r.t
1	(CO ₂ balloon)	71			
8	perlite-Fe ₃ O ₄	0.014	0.04mmol	5	60
1	(CO ₂ balloon)	83			
9	perlite-Fe ₃ O ₄	0.014	0.04mmol	4.5	75
1	(CO ₂ balloon)	98			
10	perlite-Fe ₃ O ₄	0.014	0.04mmol	4	80
1	(CO ₂ balloon)	96			

3.8. Catalytic optimization

The quantity of Perlite/Fe₃O₄ catalyst was assessed between 0.004 and 0.03 g. In the synthesis of cyclic carbonate noticeable change was not observed when the amount of Perlite/Fe₃O₄ catalyst was increased from 0.014 and 0.03 g. Therefore, the optimal quantity for the synthesis of cyclic carbonates is 0.014 g.

Table 4 Synthesis of cyclic carbonate

Entry	Epoxide	Product	Time(h)	Yield ^b (%)
1			4.5	96
2			4.5	98
3			4.5	95
4			4.5	90

^a Reaction conditions: Epoxide (2.00 mmol), CO₂ (1 atm) Perlite/Fe₃O₄ (0.014g), TBAB (0.04 mmol).

^b Isolated yield

3.7. ¹H NMR, ¹³C NMR, of isolated products

4-Chloromethyl-1,3-dioxolan-2-one:

(light-yellow liquid)⁴², IR:1797 (C=O) cm⁻¹, ¹H NMR (300 MHz, CDCl₃) δ(ppm): 3.77 – 3.57 (m, 2H), 4.33 (dd, J = 8.8, 5.8 Hz, 1H), 4.51 (t, J = 8.5 Hz, 1H), 4.93 – 4.84 (m, 1H). ¹³C NMR (75 MHz, CDCl₃) δ(ppm): 43.58, 67.00, 74.21, 154.11.

4-(phenoxymethyl)-1,3-dioxolan-2-one:

(White solid)^{42,43,44}, mp:99-100°C, (lit.^{37,39}99–100°C) IR:1796 (C=O) cm⁻¹, ¹H NMR (300 MHz, CDCl₃) δ(ppm): δ 7.57 – 6.98 (m, 2H), 6.99 – 6.68 (m, 3H), 5.21 – 4.85 (m, 1H), 4.64 – 4.39 (m, 2H), 4.30 – 3.99 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ(ppm): 161.33, 152.36, 129.72, 122.00, 112.23, 72.06, 66.38, 66.26.

4-phenyl-1,3-dioxolan-2-one:

(White solid)^{42,43}, mp:53-55°C (lit.³⁷ 51–53°C), IR: 1811 (C=O) cm⁻¹, ¹H NMR (300 MHz, CDCl₃)

δ(ppm): 8.12 – 6.85 (m, 5H_{Ar}), 5.54 (t, J = 7.5 Hz, 1H), 4.66 (t, J = 8.1 Hz, 1H), 4.37 – 3.93 (m, 1H). ¹³C NMR (75 MHz, CDCl₃) δ(ppm): 155.14, 135.92, 129.67, 129.18, 126.04, 78.11,71.26.

4-Butyl-1,3-dioxolan-2-one:

(Yellowy-orange oil)⁴³, IR: 1793 (C=O) cm⁻¹, ¹H NMR (300 MHz, CDCl₃) δ(ppm): 0.80 (t, J = 7.1 Hz, 3H), 1.17-1.31 (m, 4H), 1.48-1.66(m, 2H), 3.90 (t, J = 7.7 Hz, 1H), 4.40 (t, J = 8.1 Hz, 1H), 4.55 (dd, J = 13.3, 7.0 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ(ppm): 158.76, 76.81, 69.42, 33.47, 24.00, 19.63, 13.75.

3.8. Mechanism of catalytic CO₂ fixation

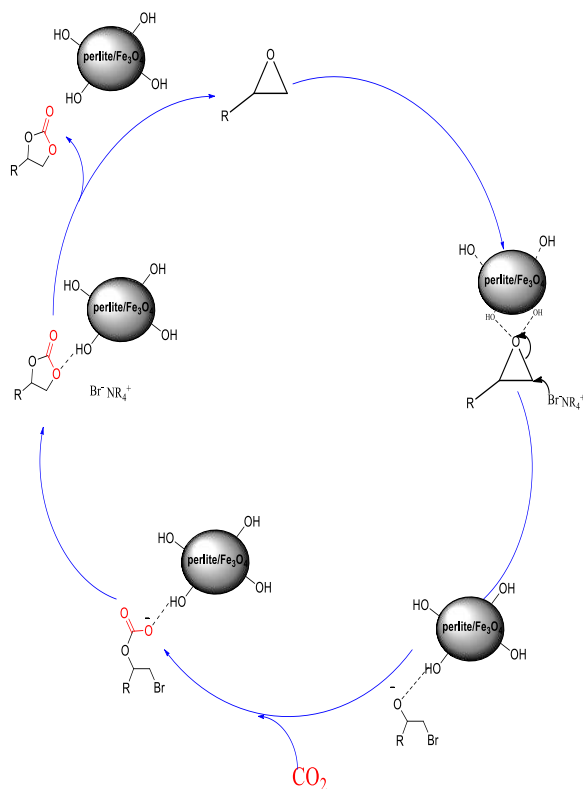
Scheme 2 proposed mechanism for CO₂ fixation by utilizing a perlite/Fe₃O₄ nanocatalyst and a co-catalyst of tetrabutylammonium bromide (TBAB).

CO₂ and epoxide ring activation: The hydroxyl groups on the perlite/Fe₃O₄ catalyst form hydrogen bonds with CO₂ and epoxide ring, making them more susceptible to nucleophilic attack. Therefore, the less sterically hindered carbon atom from epoxide are attacked through the bromide anion, which is released from the co-catalyst (TBAB). Then ring opening and CO₂ Insertion: The C-O bond in the epoxide ring breaks, allowing the oxygen atom to bond with the carbon atom of CO₂. An intramolecular S_N2 reaction occurs. The oxygen atom from the newly formed carboxyl group displaces the halide anion, Finally, a five-membered carbonate ring is formed.

3.9. Catalyst reusability

The proficiency of Perlite/Fe₃O₄ to be reused as a catalyst was evaluated in the CO₂ fixation reaction. The catalyst was washed three times with deionized water and ethanol. Then, Perlite/Fe₃O₄ was subjected to drying at 140°C for 5 h. Perlite/Fe₃O₄ was employed for at least 5 cycles in the CO₂ fixation reaction with Epichlorohydrin without any significant decrease in yield. The FT-IR peaks of the 1st and 5th recovered Perlite/Fe₃O₄ are presented in

Fig.2(c, d). All the features of the peaks, including sharpness, intensity, and regions, are the same in both peaks and well-preserved.



Scheme 2. Proposed mechanism for the synthesis of cyclic carbonate in the presence of the expanded perlite.

The results propose that there are no changes in the chemical structure of the Perlite/Fe₃O₄ even after 5 runs. We did not have a noticeable drop in efficiency until the fifth cycle. But a significant drop was observed in the sixth cycle.

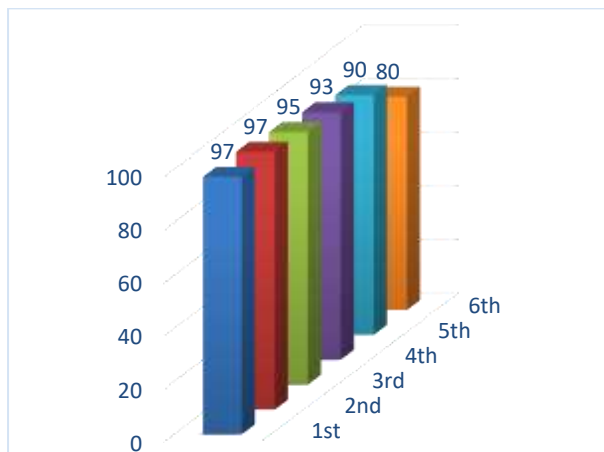


Figure 7. Perlite/Fe₃O₄ catalyst reusability

4. Conclusion

We have successfully done the transformation of carbon dioxide and epoxides into cyclic carbonates without using solvents at the atmospheric pressure of CO₂. The prominent characteristics of this method include solvent-free conditions, easy, fast, and clean separation of catalysts, atmospheric pressure of CO₂, easy workup, short reaction time, and a clear protocol. Furthermore, the Perlite/Fe₃O₄ catalyst is environmentally friendly due to its ability to be recovered up to 5 times while maintaining high performance. The capabilities and efficiency of this catalyst under solvent-free conditions and atmospheric pressure inspire optimism for a brighter future in carbon dioxide reduction.

Table 5. Comparison of various silica-based catalysts for the CO₂ fixation.

En	Catalyst	T(°C)	Pressure (bar)	Time (h)	Yield	Ref
1	AlPcMCM-41/n-Bu ₄ NBr	110	40	2	31	[45]
2	Mesoporous titanium-silicate	50	8	48	70	[46]
3	Ti-SBA-15-pr-Ade	120	6.9	8	88.9	[47]
4	Ti-mSiO ₂	120	16	6	91.6	[48]
5	Perlite/Fe ₃ O ₄	75	1	4.5	96	This work

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