

Study on the desorption of CO₂ from MEA aqueous solution over iron-titanium mixed oxide catalyst synthesized by microwave irradiation treatment

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Abstract: The energy-intensive regeneration of amine-based solvents remains a major bottleneck in CO₂ capture from fossil-fuel combustion flue, which is due to the high thermal consumption and slow desorption kinetics. To address these challenges, this study aims to develop a cost-effective, high-performance solid-acid catalyst to accelerate CO₂ desorption from CO₂-rich monoethanolamine (MEA) solutions while reducing energy input. A series of FeTiO₂-M catalysts were synthesized via a microwave-assisting co-precipitation method and the as-prepared catalysts were characterized by XRD, FT-IR, Py-IR, XPS, NH₃-TPD, and N₂ adsorption-desorption to elucidate structure-activity relationships. Results indicate that the Fe₂₅Ti₇₅O₂-M catalyst achieves a 36% increase in the average CO₂ desorption rate and a 25.7% reduction in relative heat duty at 0.4 wt. % loading and 90 °C, with certain stable activity maintained over five cycles. The enhanced performance is attributed to synergistic combination of abundant Brønsted and Lewis acid sites, activated surface hydroxyls by sulfate groups, and enlarged mesoporous surface area that facilitates carbamate diffusion. These findings demonstrate that rational design of bimetallic solid-acid catalysts can substantially improve CO₂ desorption efficiency and energy savings, therefore providing a scalable and energy-efficient strategy for amine regeneration in industrial-level CO₂ capture applications.

Keywords: CO₂ desorption, MEA, Iron-titanium oxide, Acid site, Mesoporous structure

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30 1. Introduction

31 Global energy demand is continuously rising with the rapid development of the global
32 economy ^[1]. This trend is further intensified by the slow deployment of low-emission technologies
33 and the continuing heavy reliance on fossil-fuel combustion ^[2,3]. Consequently, the dramatic
34 increase in emissions of carbon dioxide and other greenhouse gases has made global warming severe
35 challenge to human survival and development ^[4,5]. Carbon Capture, Utilization and Storage (CCUS)
36 is recognized as a critical technology for achieving net-zero carbon emissions from fossil energy
37 and one of the most viable solutions for limiting atmospheric CO₂ releases, with its core objective
38 being to facilitate the transition toward net-zero CO₂ emissions ^[6,7].

39 Currently, CO₂ capture technologies are primarily categorized into pre-combustion
40 decarbonization, in-combustion CO₂ capture, and post-combustion CO₂ capture, based on the
41 integrated position and circulation of the CO₂ separation process within the power system ^[8,9].
42 Chemical absorption is currently one of the most promising and widely applied technologies for
43 CO₂ capture due to its high efficiency, mature process, and strong adaptability to existing industrial
44 systems. It offers high selectivity and large absorption capacity, especially when using amine-based
45 solvents. Compared to other methods, chemical absorption has rich engineering experience and is
46 relatively easy to scale up. However, it suffers from high regeneration energy consumption,
47 equipment corrosion, and solvent degradation, which significantly limit its large-scale deployment
48 ^[10-12]. Based on this, researchers have conducted extensive research efforts to accelerate CO₂
49 desorption from amine solvents while simultaneously reducing regeneration energy consumption.
50 For instance, recent studies have achieved notable progress in lowering regeneration energy through
51 the development of biphasic absorbents ^[13] and blended primary amine systems ^[14,15]. Nevertheless,
52 inherent limitations remain for absorbent modification alone. As the current benchmark solvent for
53 amine scrubbing, the 30 wt.% MEA aqueous solution exhibits an actual regeneration energy
54 consumption exceeding the theoretical minimum by 0.93 GJ/t·CO₂ ^[16-18], demonstrating substantial
55 optimization potential in thermal desorption regeneration. Additionally, this system experiences
56 volatile amine losses with associated carcinogenic risks ^[19]. While elevated temperature and reduced
57 pressure enhance CO₂ desorption rates, employing tailored heterogeneous catalysts proves to be
58 more effective in balancing high desorption kinetics with low regeneration energy consumption
59 ^[20,21].

60 In recent years, the application of solid acid catalysts (SACs) has emerged as a promising
61 strategy to reduce energy requirement and increase CO₂ desorption capacity for the regeneration of
62 MEA solvent [22,23]. Using solid acid catalysts for CO₂ desorption can reduce the energy barriers
63 related to the C-N bond breaking in carbamate and speed up the proton transfer rate in protonated
64 amines [24]. Metal oxides are typical SACs, offering simple preparation routes, low cost, and robust
65 catalytic activity [25]. Li et al. [26] fabricated a solid-acid catalyst by one-step urea-hydrolysis of
66 sepiolite (SEP) to anchor SO₄²⁻/ZrO₂ (SZ), yielding SZ@SEP composites with tunable SZ/SEP
67 ratios. Among them, SZ@SEP-1/2 exhibited the highest density of Brønsted and Lewis acid sites.
68 When 2 g of SZ@SEP-1/2 was suspended in 5 M MEA at 88 °C, the CO₂ desorption rate rose by
69 92 % and the relative heat duty dropped by 48.6 % versus blank thermal regeneration, and the
70 catalyst retained stable activity over 20 cycles, confirming its potential for energy-efficient CO₂
71 capture. Rao et al. [27] prepared a ternary Fe/SiO₂/biochar solid-acid catalyst via one-pot pyrolysis
72 of wheat flour, SiO₂ and FeCl₃. In 30 wt. % MEA at 90 °C the best sample (FSC₂) raised the CO₂
73 desorption amount by 35 % and cut the relative heat duty by 34 % versus blank thermal regeneration,
74 and activity loss after five cycles was only 6.1 %. The performance gain is ascribed to abundant
75 Brønsted/Lewis acid sites and a reversible Fe (II)/Fe (III) redox couple that jointly accelerate
76 carbamate/bicarbonate cleavage and proton transfer, offering an energy-efficient and stable route
77 for amine solvent regeneration. Jin et al. [28] prepared black TiO₂ nanoparticles by NaBH₄ reduction
78 of P25, and the resulting material functions simultaneously as a photothermal converter and a solid-
79 acid catalyst. Surface bridging hydroxyls supply Brønsted acid sites, while Ti³⁺/Ti⁴⁺ centers act as
80 Lewis acid sites, accelerating carbamate and bicarbonate decomposition during CO₂ desorption
81 from MEA solution at ≈ 88 °C under 7-sun irradiation. The above results indicate that nanostructured
82 SACs can significantly improve the absorption and desorption performance of the system [29]. In
83 addition, some metal-organic framework-based solid acid catalysts have been reported for CO₂
84 desorption processes due to their advantages of high porosity, selectivity, flexible structure, and ease
85 of modification/functionalization [30,31]. However, their industrial application is limited by either
86 high production cost or insufficient catalytic efficiency. Therefore, it is of significance to develop
87 cost-effective and highly active solid acid catalysts that are inexpensive yet highly effective for
88 promoting the regeneration of amine-rich solutions [32].

89 Studies had shown that metal-oxide nanoparticles combine physical and chemical promotion,

90 and they physically broke bubbles via Brownian motion, enlarged the interfacial area and lowered
91 mass-transfer resistance, while their surfaces abound in Lewis and Brønsted acid sites helped to
92 promote CO₂ reactions, leading to markedly higher CO₂-desorption rates [33,34]. However, their
93 modest specific surface area, irregular pore size and rapid deactivation limit their use at high
94 temperatures and in aqueous amine systems [35]. Relative to single-metal ions, the differing electron
95 affinities of elements in mixed-metal oxides strongly alter the electronic coordination environment
96 around multiple metal ions [36]. Among the reported candidates, iron-based catalysts had been
97 identified as exceptionally efficient for CO₂-catalyzed desorption, benefiting from their abundant
98 acidic sites and the facile electron transfer mediated by the Fe(II)/Fe(III) redox cycle [27]. On the
99 other hand, our research group had demonstrated the catalytic promotion of microwave-irradiated
100 FeOOH catalysts for the regeneration of CO₂-rich MEA solutions, highlighting the significant
101 impact of a large specific surface area and abundant Lewis acid sites on catalytic performance. Ti⁴⁺
102 (74.5 pm) has been shown to be a promising dopant due to its ionic radius being comparable to that
103 of Fe³⁺ (69 pm) and its half-filled (d⁵) electron configuration [37]. And in the study by Jiang et al. [38],
104 the improvement of the absorption properties of MEA and MDEA solutions by different
105 nanoparticles was examined. The findings indicated that TiO₂ particles exhibited the most
106 significant enhancement factor, followed by MgO with Al₂O₃, and SiO₂ particles demonstrated the
107 least enhancement. Additionally, Geng [39] et al. systematically demonstrated the significant catalytic
108 effect of a mesoporous, high-acid-density SO₄²⁻/ZrTiO_x solid acid catalyst on CO₂ desorption in a
109 90 °C, 5 mol MEA system. In this study, metal-element doping was employed to mitigate mesopore
110 collapse induced by high-temperature calcination, while dual active sites were introduced to
111 synergistically accelerate CO₂ desorption from amine-rich solutions.

112 Microwave irradiation enables rapid and uniform heating of the reaction medium, inducing an
113 explosive surge in nucleation that favors products with small particle sizes and narrow size
114 distributions [40,41]. On the other hand, the volumetric heating characteristic of microwaves allows
115 the interior of the adsorbent to heat up instantly, markedly shortening CO₂ desorption time, lowering
116 regeneration energy consumption, and improving microwave energy utilization efficiency-an
117 advantage that also applies to catalysts [42,43]. Therefore, microwave irradiation treatment is expected
118 to refine the particle size of Fe-Ti composite catalysts, enlarge their specific surface area and acid-
119 site density, and consequently enhance their performance for CO₂ desorption from MEA solution.

120 In this study, a series of FeTiO₂-M-20 catalysts were prepared by a co-precipitation-microwave
121 method to evaluate their performance in CO₂ desorption from CO₂-rich MEA solutions. Five
122 catalysts with different Fe³⁺:Ti⁴⁺ molar ratios-0:100, 25:75, 50:50, 75:25 and 100:0, were
123 investigated. In addition, the catalyst exhibiting the highest activity was further subjected to five
124 absorption–desorption cycles to examine its stability and potential influence on subsequent CO₂
125 absorption. Comprehensive physicochemical characterization, including XRD, FT-IR, Py-IR, XPS,
126 and NH₃-TPD, was performed to elucidate the structure-activity relationships on governing the
127 catalytic behavior.

128 2. Experimental

129 2.1 Catalyst preparation

130 All reagents were of analytical grade (AR) and used directly without further purification.
131 Initially, predetermined amounts of Fe(NO₃)₃·9H₂O and Ti(SO₄)₂·7H₂O were dissolved in 150 mL
132 of deionized water and vigorously stirred for 1 h. Subsequently, 2 mol/L NH₃·H₂O was added
133 dropwise into the mixed solution until the pH reached 9-10. The resulting suspension was then
134 immediately filtered without an aging step and thoroughly washed. The obtained wet precipitate
135 was subjected to microwave irradiation at power level P₃₀ for 20 min using a domestic microwave
136 oven (EG823MF3-NW, Midea, China), followed by calcination in static air at 400 °C for 5 h to
137 yield the final catalysts. These materials were denoted as Fe_xTi_{100-x}O₂-M, where *x* represents the
138 molar ratio of Fe³⁺/(Fe³⁺+Ti⁴⁺) in the precursor solution prior to the titration and precipitation. Five
139 catalysts were prepared analogously and labeled as TiO₂-M, Fe₂₅Ti₇₅O₂-M, Fe₅₀Ti₅₀O₂-M,
140 Fe₇₅Ti₂₅O₂-M and Fe₂O₃-M, respectively.

141 2.2 Catalytic activity Test

142 X-ray diffraction (XRD) patterns were collected on a Rigaku Smart Lab diffractometer (Japan
143 Science) using Cu K α radiation ($\lambda = 0.15406$ nm) over $2\theta = 20-80^\circ$ at a scan speed of $2^\circ/\text{min}$ under
144 40 kV and 40 mA. N₂ adsorption-desorption isotherms were acquired with a Micromeritics ASAP
145 2460 analyzer at 77 K, and specific surface areas and pore-size distributions are obtained through
146 Brunauer-Emmett-Teller (BET) calculations and Barrett-Joyner-Halenda (BJH). X-ray
147 photoelectron spectroscopy (XPS) was performed on a Thermo-Fisher Scientific Escalab 250 Xi
148 (USA) with 150 W monochromated Al K α radiation (1486.6 eV). Binding energies of Fe 2p and O
149 1s were referenced to the C 1s peak at 284.8 eV. Fourier-transform infrared spectra (FTIR) were

150 recorded on a Nicolet iS5 spectrometer (4 cm^{-1} resolution) from 400 to 4000 cm^{-1} . Acid-site
151 characterization by pyridine-IR (Py-IR) was carried out via a Thermo Nicolet 380 spectrometer.
152 Samples were firstly evacuated at $105\text{ }^{\circ}\text{C}$, cooled to ambient temperature, and then exposed to a 20
153 mL/min He flow saturated with pyridine for 2 h to ensure full acid-site coverage. Subsequently, the
154 cell was evacuated for 30 min at $150\text{ }^{\circ}\text{C}$ and $250\text{ }^{\circ}\text{C}$, and spectra were collected at each temperature.
155 Particle-size distributions were determined in triplicate with a Malvern 2000 analyzer. CO_2 -TPD
156 was carried out on a Micromeritics Auto Chem II 2920 (USA). The sample was heated to $200\text{ }^{\circ}\text{C}$
157 ($10\text{ }^{\circ}\text{C}/\text{min}$, 30 min hold) in 30 mL/min He to remove moisture, cooled to $50\text{ }^{\circ}\text{C}$, saturated with 40
158 mL/min high-purity CO_2 for 60 min, and then purged with 30 mL/min He for 30 min to eliminate
159 physisorbed CO_2 . Desorption was monitored from 50 to $500\text{ }^{\circ}\text{C}$ at $10\text{ }^{\circ}\text{C}/\text{min}$ under 30 mL/min He.

160 **2.3 Absorption and desorption experiments**

161 CO_2 uptake and multi-cycle desorption experiments were conducted in a custom-designed
162 semi-intermittent apparatus. During the absorption phase, a continuous stream of pure CO_2 (1.5 L
163 /min) sequentially passed through a calibrated mass-flow meter, a three-way valve, and a wash bottle
164 before entering into a 500 mL three-neck flask containing 30 wt.% MEA solution. The system was
165 maintained at $40 \pm 1\text{ }^{\circ}\text{C}$ under constant magnetic stirring at 300 r/min [20,44]. To monitor the
166 absorption progress, the solution of 1-1.5 mL was periodically withdrawn every 2-3 min using a
167 pipette and collected in sealed vials for subsequent analysis. A bypass loop was integrated into the
168 gas line to allow controlled interruption of CO_2 flow, which kept closed during absorption to
169 maintain a stable gas supply, and was opened during sampling to divert the gas stream and
170 temporarily suspend gas-liquid contact. This setup enabled the safe withdrawal of 1-2 mL of CO_2 -
171 loaded amine solution from the reactor headspace without disturbing the bulk system. After each
172 sampling, the bypass valve was closed to restore CO_2 flow, and absorption continued until the MEA
173 solution reached a CO_2 loading of 0.50 mol CO_2 per mole of amine, as measured by Chittick titration.

174 CO_2 -desorption experiments were conducted in a 1 L three-neck flask fitted with a thermometer,
175 condenser and sampling port. In each run, 2 g of catalyst was added to the CO_2 -loaded MEA solution
176 and the mixture was maintained at $90 \pm 1\text{ }^{\circ}\text{C}$ with magnetic stirring at 400 r/min [45,46]. The oil bath
177 was insulated with black thermal cotton to minimize the heat loss and ensure an accurate
178 temperature control. When the MEA temperature reached $40\text{ }^{\circ}\text{C}$, an initial aliquot of 1-1.5 mL was
179 withdrawn for Chittick titration to determine the CO_2 loading. Subsequently, samples were collected

180 every 5 min initially, with intervals extended to 15 min between 30 and 60 min, and to 30 min
 181 between 60 and 120 min. Electrical power consumption was recorded simultaneously by an energy
 182 meter. The CO₂ loading (α) was calculated from the volumes of CO₂ evolved and HCl consumed
 183 during the Chittick titration, as seen in Eq. (1):

$$184 \quad \alpha = \frac{V_{CO_2} - 2V_{HCl}}{V_{HCl} \times \zeta} \quad (1)$$

185 Here, α denotes the CO₂ loading, V_{CO_2} represents the volume of CO₂ evolved during the
 186 analysis, V_{HCl} corresponds to the volume of hydrochloric acid consumed at the titration endpoint
 187 (indicated by the complete colour change of the amine solution), and ζ is the gas-phase correction
 188 factor computed from $\zeta = \frac{(T+273.15) \times 22.4}{273.15}$.

189 For the catalytic desorption of CO₂ from MEA phase, the catalyst was compressed into a thin
 190 flake, encased in a fine-pore gauze, and positioned at the center of CO₂-rich MEA solution. Each
 191 experiment involved approximately twelve sampling steps, leading to a MEA volume reduction of
 192 20-30 mL, which occupies 4-6% of the initial 500 mL. As reported in the literature, the CO₂
 193 desorption rate is primarily determined by the MEA⁺COO⁻ concentration in the loaded solution [47].
 194 Consequently, this minor solvent loss does not alter amine concentration and is therefore considered
 195 kinetically negligible [48].

196 Herein, CO₂-desorption profiles were constructed by plotting the CO₂ loading (mol/mol)
 197 versus residence time (min) from the initial saturated value of 0.5 mol/mol to the equilibrium lean
 198 loading in the MEA solvent. Each loading datum was obtained in duplicate (or more) to guarantee
 199 an average absolute deviation (AAD) below 5%. The figures-of-merit used to appraise CO₂-
 200 desorption performance—namely, the molar amount of CO₂ released (n_{CO_2}), the mean desorption rate
 201 (r), the absolute heat duty (HD), the relative heat duty (RHD), the cyclic capacity and the composite
 202 desorption parameter (DP), are defined and computed in Eqs. (2-7), as detailed below:

$$203 \quad n_{CO_2} = C \times V \times (\alpha_{rich} - \alpha_{lean}) \quad (2)$$

$$204 \quad r = \frac{n_{CO_2}}{dt} = \frac{C \times V \times (\alpha_{rich} - \alpha_{lean})}{dt} \quad (3)$$

$$205 \quad HD = \frac{Q_{input}}{n_{CO_2}} = \frac{kJ/\Delta t}{mol/\Delta t} \quad (4)$$

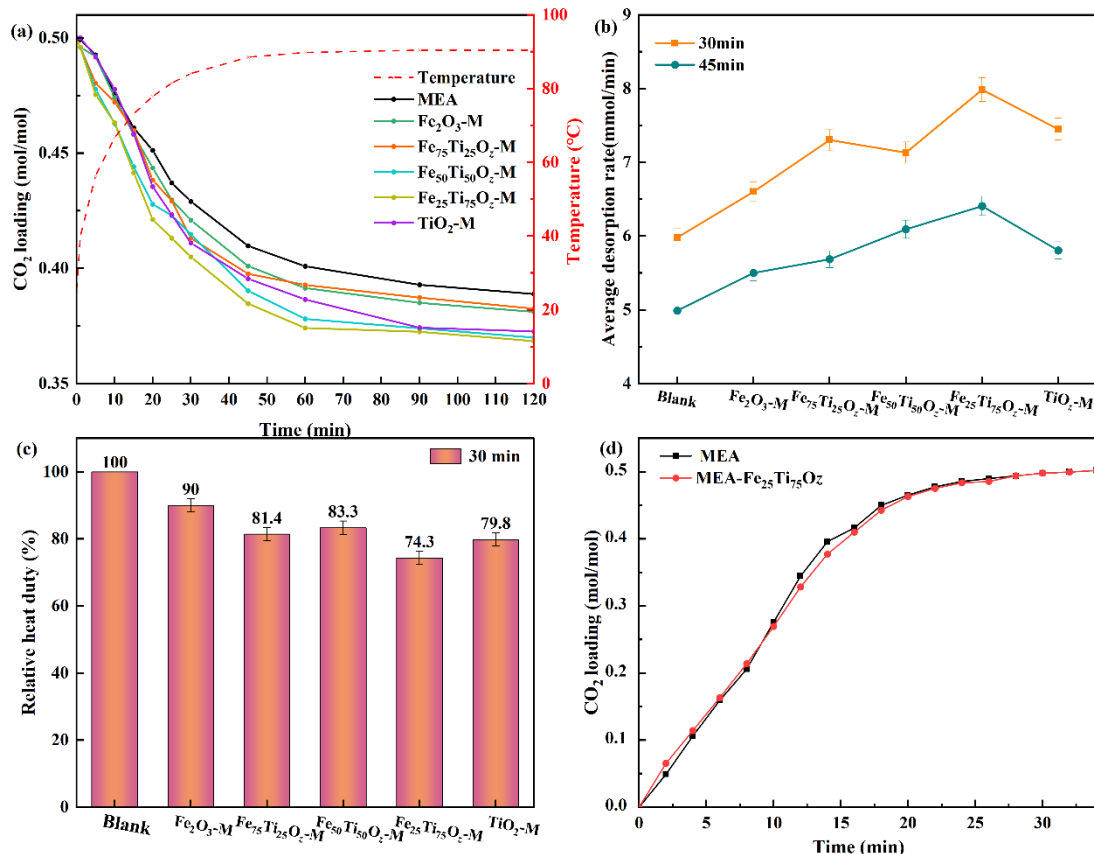
$$206 \quad RHD = \frac{HD_i}{HD_{baseline}} \times 100\% \quad (5)$$

$$207 \quad \text{Cyclic capacity} = C \times (\alpha_{rich} - \alpha_{lean}) \quad (6)$$

$$208 \quad \text{Desorption parameter} = \frac{\text{initial desorption rate} \times \text{cyclic capacity}}{\text{heat duty}} \quad (7)$$

209 **3.Results and discussion**

210 **3.1 Catalytic Desorption and Absorption Performance Evaluation**



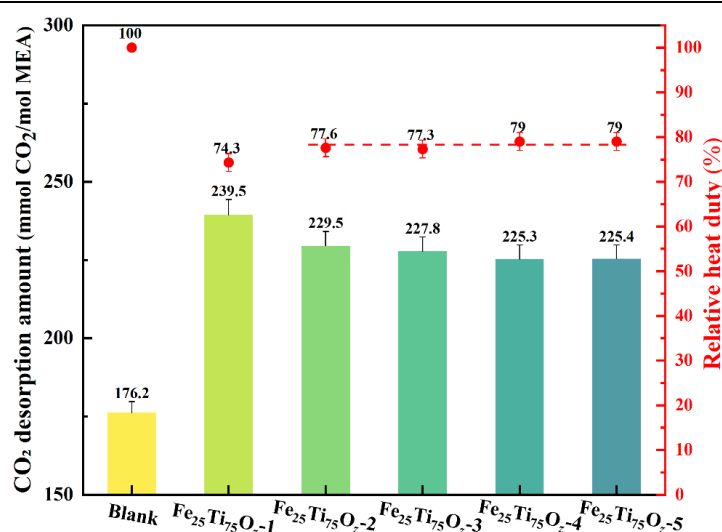
211
212 Fig.1. Catalytic desorption performance: CO₂ desorption profiles (a), average desorption rate (b), relative heat duty
213 (c), and catalytic effect of Fe₂₅Ti₇₅O₂-M on CO₂ absorption (d).

214 The catalytic CO₂ desorption performance of the five as-synthesized catalysts was evaluated
215 in 5 M MEA solution and benchmarked against a blank run (Fig. 1(a)). Between 40 °C and 90 °C
216 all solids accelerated the reduction of CO₂ loading, the steepest decrease occurred within the first
217 30 min of the 120-min test, indicating an extremely rapid desorption rate. The rate declined
218 progressively from 30-60 min and levelled off from 60-120 min. Consequently, the average
219 desorption rate (ADR) over the initial 30 min and the relative heat duty were used as the main
220 metrics of catalytic activity. As shown in Fig. 1(b), the 30-min ADR displays a volcano-type trend
221 with increasing Fe/Ti molar ratio, peaking at Fe/Ti = 25/75, where the ADR is 36% higher than that
222 of the uncatalyzed system. Correspondingly, the RHD values (Fig. 1(c)) indicate that Fe₂₅Ti₇₅O₂-M
223 reduces energy consumption by 25.7%, ranking superior to TiO₂-M, Fe₇₅Ti₂₅O₂-M, Fe₅₀Ti₅₀O₂-M,

224 and Fe₂O₃-M. These results demonstrate that Fe₂₅Ti₇₅O₂-M not only accelerates CO₂ desorption but
 225 also substantially lowers the energy requirement for solvent regeneration, underscoring its potential
 226 for cost-effective and energy-efficient CO₂ capture. To verify whether the acidic solids impair
 227 absorption, absorption tests were conducted, as seen in Fig. 1(d). In 5 M MEA the catalysts slightly
 228 enhance the rate in the first 10 min, but the final CO₂ loading (0.50 ± 0.01 mol mol⁻¹) is identical to
 229 that without catalyst, confirming that the absorption equilibrium is unaffected. Table 1 summarizes
 230 the cyclic capacities and desorption parameters for the first 30 min. As the Fe content decreases, the
 231 cyclic capacity increases and the heat duty decrease. Among the tested catalysts, Fe₂₅Ti₇₅O₂-M
 232 exhibits the highest composite desorption parameter, with an RDF of 258.8% relative to the blank,
 233 highlighting its superior combination of desorption efficiency, energy savings, and operational
 234 stability for practical CO₂ capture applications.

235 Table 1. Desorbed amount, heat duty and desorption parameter of CO₂ within the first 30 min from MEA solution
 236 over the as-prepared catalysts.

Catalysts	Cyclic capacity (*10 ³ mol CO ₂)	Heat duty (kJ/mol)	Desorption parameter [(10 ⁻³ mol CO ₂) ³ /L ² kJ mol]
Blank	0.176	1185.18	0.0017
TiO ₂ -M	0.223	934.4	0.0036
Fe ₂₅ Ti ₇₅ O ₂ -M	0.240	871.6	0.0044
Fe ₅₀ Ti ₅₀ O ₂ -M	0.214	975.9	0.0031
Fe ₇₅ Ti ₂₅ O ₂ -M	0.219	953	0.0034
Fe ₂ O ₃ -M	0.198	1054.4	0.0025



237
 238 Fig. 2. Cyclic stability test of Fe₂₅Ti₇₅O₂-M catalyst: CO₂ desorption amount and relative heat duty over five
 239 consecutive regeneration cycles.

240 Catalyst stability is a key property for realizing catalyst recycling and cost reduction. The
241 $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ catalyst was subjected to five consecutive CO_2 desorption cycles in an amine-rich
242 solution at a catalyst-to-MEA mass ratio of 0.4 wt. %. After each cycle, the wrapped sheet-like
243 catalyst was removed, repeatedly rinsed with deionized water to eliminate residual amine on the
244 surface, dried, and then reused in the next cycle. As shown in Fig. 2, the fresh $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ catalyst
245 achieved a CO_2 desorption amount of 239.5 mol over per mol MEA and a relative heat duty of
246 74.3 %. After five cycles, it retained approximately 95% of its initial desorption capacity, and the
247 relative heat duty increased only slightly to 79%, indicating minimal loss in catalytic activity. The
248 XRD and FT-IR patterns of the fresh and spent $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ catalysts are presented in Fig. 3. After
249 five cycles, the spent catalyst retains an amorphous phase similar to that of the fresh sample, except
250 for the emergence of weak $\text{TiO}_2\text{-M}$ diffraction peaks, likely arising from interactions between Fe
251 and Ti species during use. Compared with the fresh catalyst, the FT-IR spectrum of the spent
252 $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ shows enhanced bands at 3420 cm^{-1} and 1630 cm^{-1} , attributed to surface-adsorbed
253 water, along with a slightly intensified band at 1407 cm^{-1} corresponding to sulfated species ^[49].
254 Notably, a new signal at 1078 cm^{-1} in the spent catalyst is probably due to MEA adsorbed on the
255 surface ^[50]. In addition, the diffraction peak around 500 cm^{-1} shifts slightly, indicating lattice inter-
256 doping and strong mutual interactions, consistent with the XRD observations. Although the spent-
257 catalyst exhibits minor structural rearrangement and adsorption of a small amount of MEA during
258 cycling, it still retains 95% of original catalytic CO_2 desorption capacity with the relative heat duty
259 increasing from 74.3% to 79% in the fifth test, indicating that the as-prepared $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ catalyst
260 possesses a certain degree of stability, and its abundant hydroxyl groups and sulfate species may
261 play a positive role in promoting carbamate decomposition. In addition, Table 2 presents a
262 comparison of CO_2 desorption performance over various solid acid catalysts for CO_2 -rich MEA
263 amine solutions. From Table 2, it can be seen that the as-prepared $\text{Fe}_{25}\text{Ti}_{75}\text{O}_z\text{-M}$ catalyst effectively
264 increases the CO_2 desorption amount and reduces the consumption of renewable energy at $90\text{ }^\circ\text{C}$,
265 which is better than most of the solid acid catalysts reported in Table 2.

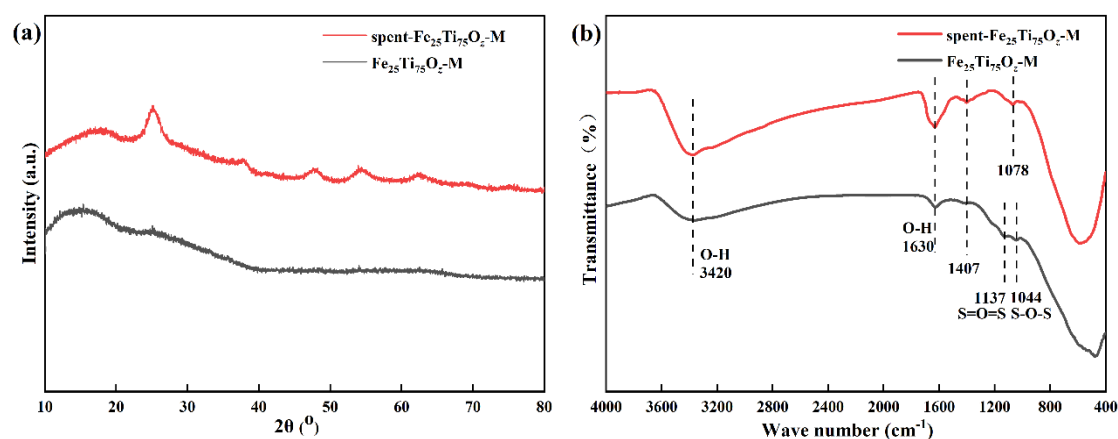


Fig.3. XRD patterns (a) and FT-IR spectra (b) of the fresh $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ and spent $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalysts.

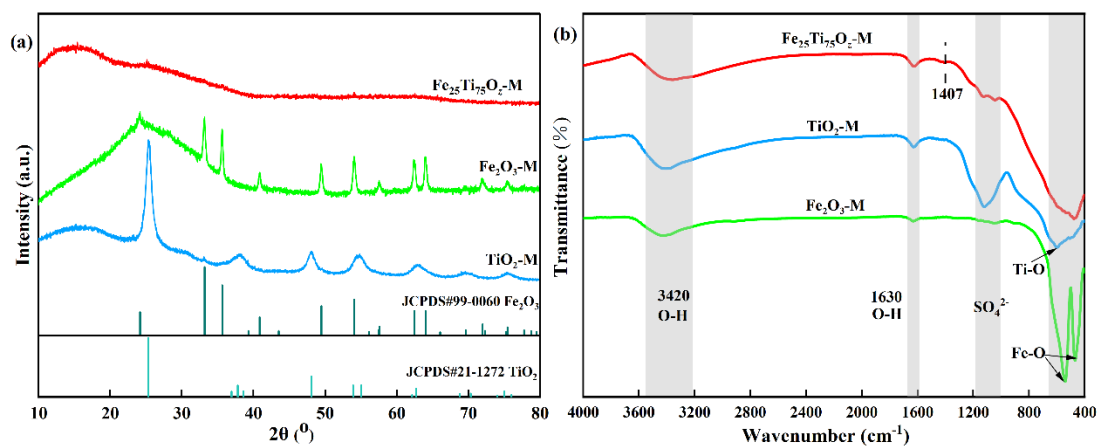
Table 2 Comparison of catalytic activity over various solid acid catalysts for the regeneration of MEA solution

Catalysts	Catalyst loading (wt.%)	Desorption temperature (°C)	Increase in desorbed CO_2 (%)	Relative heat duty (%)	Ref.
NiO-KIT-6	1.25	100	-	75.4	[50]
$\text{SO}_4^{2-}/\text{TiO}_2$	1.5	96	20.0	82.9	[51]
SAPO-34	1.5	96	28.3	75.7	[52]
$\text{SO}_4^{2-}/\text{ZrO}_2$	1.25	96	20	88.9	[52]
$\text{SO}_4^{2-}/\text{SnO}_2$	1.25	96	37.5	75.6	[52]
TiO_2	0.42	90	-	89.7	[53]
MOR	0.42	90	-	88.4	[53]
HZSM-5	0.42	90	-	84.1	[53]
TiP_2O_7	0.1	91	14.2	87.0	[54]
TiO_2	0.1	91	8.8	92.0	[54]
TiC	0.2	91	12.0	90.0	[54]
Al_2O_3	1.25	96	-	79.1	[55]
$\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$	0.4	90	36.0	74.3	This work

3.2 Catalyst Characterization Results

The $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalyst, identified as optimal in terms of Fe:Ti molar ratio, was subjected to detailed characterization, with $\text{TiO}_2\text{-M}$ and $\text{Fe}_2\text{O}_3\text{-M}$ serving as references. Fig. 4(a) shows the XRD patterns of $\text{TiO}_2\text{-M}$, $\text{Fe}_2\text{O}_3\text{-M}$ and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$. $\text{TiO}_2\text{-M}$ and $\text{Fe}_2\text{O}_3\text{-M}$ are both crystalline, matching the standard cards of anatase (JCPDS 21-1272) and hematite (JCPDS 99-0060), respectively. Compared with the sharp reflections of $\text{Fe}_2\text{O}_3\text{-M}$, the peaks of $\text{TiO}_2\text{-M}$ are broader, indicating that sulfation suppresses crystal growth. In contrast, the diffraction profile of the bimetallic $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalyst is essentially amorphous. The similar saturation coordination numbers of Fe and Ti induce severe lattice distortion, preventing the formation of well-defined

278 crystalline phases [39]. Such amorphous structures typically provide high surface areas, large pore
279 volumes, and abundant functional groups, all conducive to accelerated CO₂ desorption from MEA
280 solutions [21].



281

282 Fig.4. XRD patterns (a) and FT-IR spectra (b) of TiO₂-M, Fe₂O₃-M, and Fe₂₅Ti₇₅O₂-M catalysts.

283 The FT-IR spectra in Fig. 4(b) reveals bands at 3420 cm⁻¹ and 1630 cm⁻¹ assigned to the
284 stretching and bending vibrations of surface-adsorbed H₂O (-OH). The feature near 500 cm⁻¹
285 corresponds to Fe-O and Ti-O stretching. Absorptions between 900 cm⁻¹ and 1300 cm⁻¹ are
286 associated with S-O stretching in inorganic chelates. For Fe₂₅Ti₇₅O₂-M, peaks at 1137 cm⁻¹ and 1044
287 cm⁻¹ are ascribed to O=S=O and O-S-O moieties, evidencing both bidentate bridging and chelating
288 sulfate species [39,56]. Sulfate groups are strongly electron-withdrawing, and they extract electron
289 density from adjacent Fe³⁺ and Ti⁴⁺ and simultaneously weaken surface -OH groups, thereby
290 generating abundant Lewis-acid sites and proton-donating -OH groups [39,56]. Additionally, the band
291 at 1407 cm⁻¹ indicates two types of sulfated species on Fe₂₅Ti₇₅O₂-M, which are strongly electron-
292 withdrawing, generating abundant Lewis acid sites and proton-donating -OH groups that facilitate
293 carbamate decomposition, thereby lowering the energy requirement for amine regeneration [39,57].

294 X-ray photoelectron spectroscopy (XPS) was used to verify the surface chemistry and
295 elemental states of TiO₂-M and Fe₂₅Ti₇₅O₂-M. As shown in Fig. 5, Ti, Fe, S and O are detected on
296 Fe₂₅Ti₇₅O₂-M, confirming successful synthesis. Peaks at 464.6 eV (Ti 2p_{1/2}) and 458.8 eV (Ti 2p_{3/2})
297 are assigned to Ti-O-S and Ti-O bonds, respectively, evidencing surface Ti⁴⁺ [39,56]. S 2p_{1/2} (169.8
298 eV) and S 2p_{3/2} (178.7 eV) signals correspond to S⁶⁺, indicating sulfate incorporation [39]. The O 1s
299 peaks at ~530.1 eV and ~530.8 eV are associated with lattice oxygen and lattice hydroxyls, whereas
300 the component near 532 eV reflects oxygen in sulfate bonded to metal atoms [58]. After Fe

301 introduction, the binding energies shift to higher values, implying decreased electron density, likely
 302 due to Fe-Ti interactions. Importantly, Fe incorporation enriches the surface -OH population, which
 303 is crucial for carbamate decomposition. Finally, the Fe 2p envelope can be deconvoluted into eight
 304 sub-peaks: the component at 711.3 eV is assigned to $\text{Fe}^{2+} 2p_{3/2}$ and that at 713.2 eV to $\text{Fe}^{3+} 2p_{3/2}$.
 305 During operation, reversible redox cycling between Fe^{2+} and Fe^{3+} not only ensures catalyst
 306 recyclability but also generates abundant oxygen vacancies that enhance catalytic activity [27].

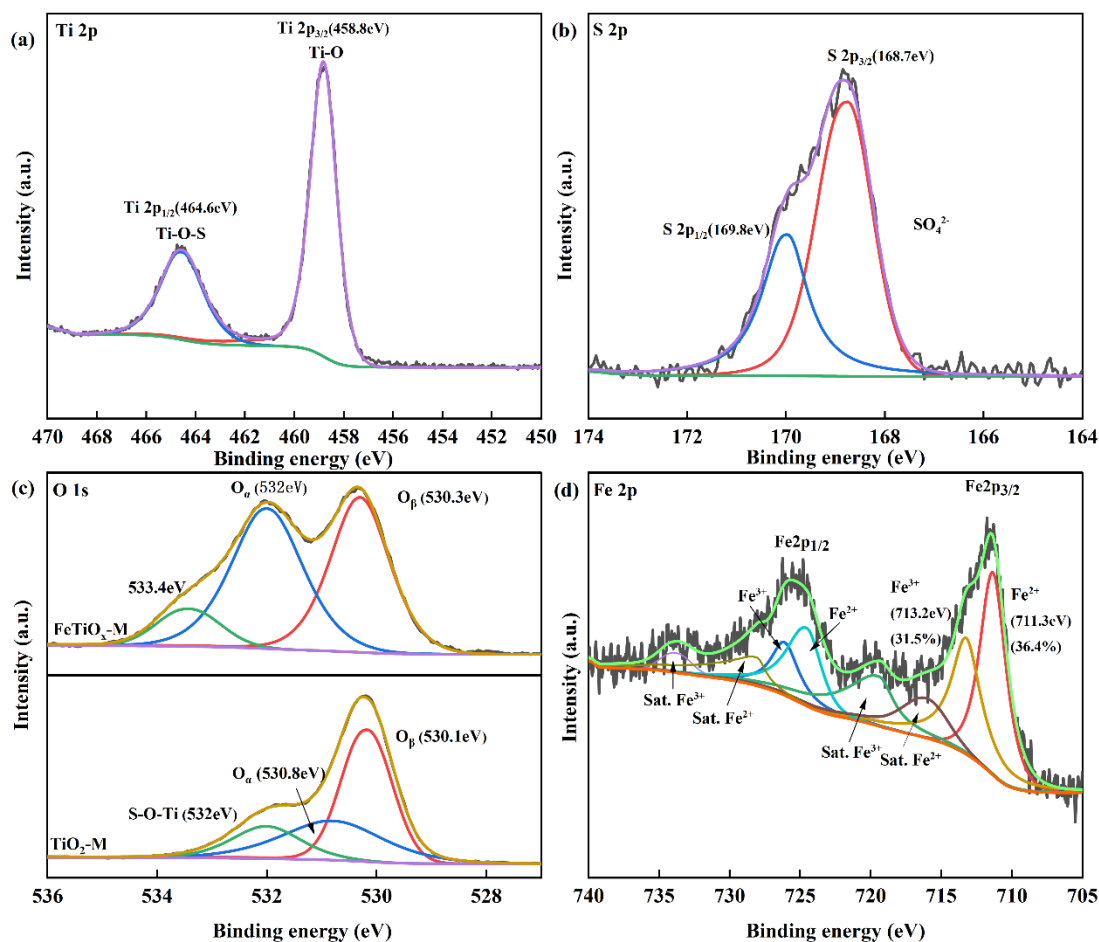
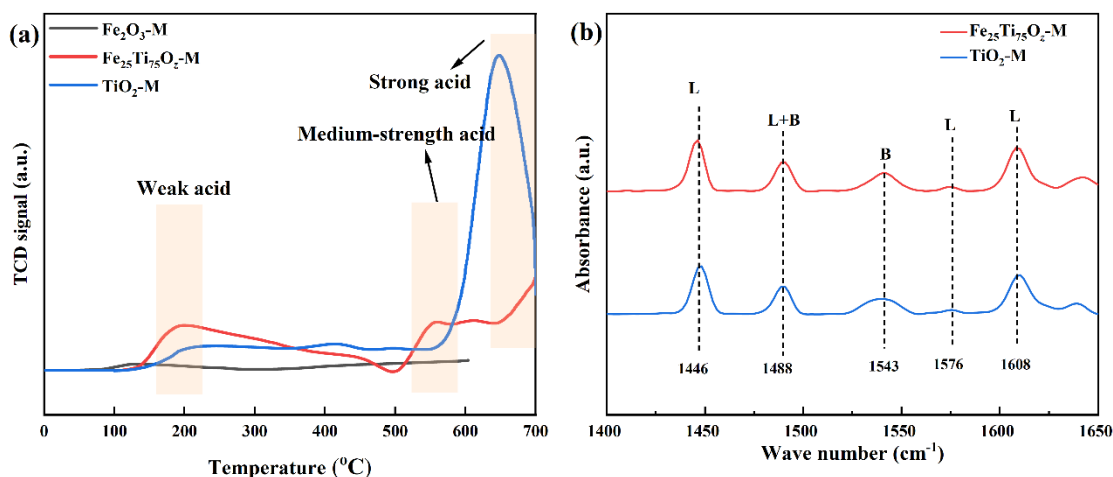


Fig.5. XPS spectra of $\text{TiO}_2\text{-M}$ and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalysts: (a) Ti 2p, (b) S 2p, (c) O 1s, and (d) Fe 2p.

309 The surface acidity of $\text{TiO}_2\text{-M}$, $\text{Fe}_2\text{O}_3\text{-M}$ and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ was quantified by NH_3
 310 temperature-programmed desorption ($\text{NH}_3\text{-TPD}$). Desorption temperatures in the ranges 50-400 °C,
 311 400-600 °C and > 600 °C are assigned to weak, medium and strong acid sites, respectively [39]. As
 312 shown in Fig. 6(a), $\text{Fe}_2\text{O}_3\text{-M}$ exhibits negligible acidity, whereas $\text{TiO}_2\text{-M}$ displays a pronounced
 313 desorption peak above 600 °C, indicating a high density of strong acid sites, which is consistent
 314 with XPS analysis. After Fe is incorporated into the Ti matrix, distinct desorption signals appear in
 315 all three temperature windows, indicating that Fe introduction not only preserves the strong sites

316 but also generates additional weak and medium-strength acid sites. This enrichment is attributed to
 317 the electron-withdrawing effect of sulfate on Lewis-acid centers. The type and abundance of acid
 318 sites are critical for carbamate decomposition. Py-IR spectra of $\text{TiO}_2\text{-M}$ and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ are
 319 compared in Fig. 6(b). Bands at 1446 , 1576 and 1608 cm^{-1} are diagnostic of Lewis (L) acid sites,
 320 while those at 1488 cm^{-1} (L + B) and 1543 cm^{-1} (Brønsted, B) reflect combined Lewis/Brønsted
 321 and pure Brønsted acidity, respectively [49]. Both catalysts contain abundant L and B sites, however,
 322 quantitative analysis of Py-IR spectra at 150 and $250\text{ }^\circ\text{C}$ (Table 3) shows that Fe doping largely
 323 preserves Brønsted acidity while significantly increasing Lewis acidity. This increase in Lewis sites
 324 enhances carbamate decomposition, corroborating the catalytic performance trends as
 325 aforementioned. Therefore, the cooperative interaction of Brønsted and Lewis acid sites is the key
 326 to the superior CO_2 -desorption activity of $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$.



327
 328 Fig.6. (a) NH_3 -TPD profiles of $\text{TiO}_2\text{-M}$, $\text{Fe}_2\text{O}_3\text{-M}$, and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalysts, (b) Py-IR spectra of $\text{TiO}_2\text{-M}$ and
 329 $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$ catalysts.

330 Table 3. Concentration and distribution of Lewis (L) and Brønsted (B) acid sites on $\text{TiO}_2\text{-M}$ and $\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$
 331 catalysts.

Samples	Weak acid (mmol/g)				Medium-strength acid (mmol/g)			
	B	L	B/L	Total	B	L	B/L	Total
$\text{TiO}_2\text{-M}$	0.061	0.077	0.78	0.138	0.04	0.051	0.84	0.089
$\text{Fe}_{25}\text{Ti}_{75}\text{O}_2\text{-M}$	0.063	0.082	0.76	0.145	0.04	0.048	0.79	0.079

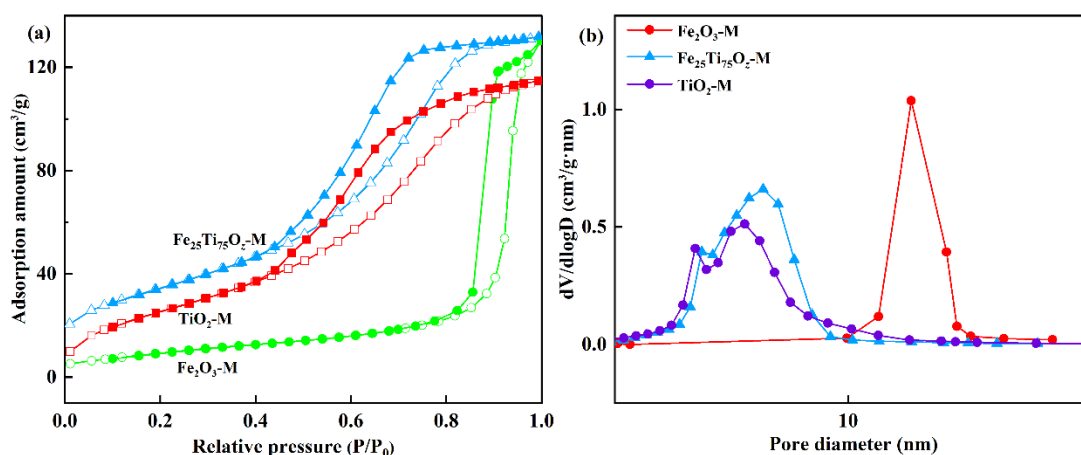


Fig. 7. N₂ adsorption-desorption isotherms and corresponding pore-size distributions of TiO₂-M, Fe₂O₃-M and Fe₂₅Ti₇₅O₂-M catalysts

Fig. 7 presents the N₂ adsorption-desorption isotherms and corresponding pore-size distributions of TiO₂-M, Fe₂O₃-M and Fe₂₅Ti₇₅O₂-M catalysts. According to IUPAC classification, all three isotherms are type IV with well-defined H1 hysteresis loops in the relative pressure range 0.4-1.0, evidencing uniform micro-/mesoporous architectures [21,59]. Compared with Fe₂O₃-M, the low-pressure closure points of TiO₂-M and Fe₂₅Ti₇₅O₂-M shift to lower P/P₀ values. The approximate superimposable isotherms of Fe₂₅Ti₇₅O₂-M and TiO₂-M confirm intensive Fe-Ti lattice intermixing, consistent with XRD and IR results. Textural parameters are summarized in Table 4. Fe₂₅Ti₇₅O₂-M exhibits a slightly larger average pore diameter than TiO₂-M, yet smaller than Fe₂O₃-M, suggesting that Fe incorporation expands the Ti matrix pores and facilitates the carbamate diffusion into the catalyst interior. The mesoporous surface areas follow the order Fe₂₅Ti₇₅O₂-M > TiO₂-M > Fe₂O₃-M, with Fe₂₅Ti₇₅O₂-M displaying the highest BET area (125.6 m² g⁻¹). This increase in surface area and pore volume provides more exposed active sites and greater adsorption capacity for carbamate species, directly enhancing CO₂ desorption and accelerating amine regeneration.

Table 4. Physical structural parameters of TiO₂-M, Fe₂O₃-M and Fe₂₅Ti₇₅O₂-M catalysts.

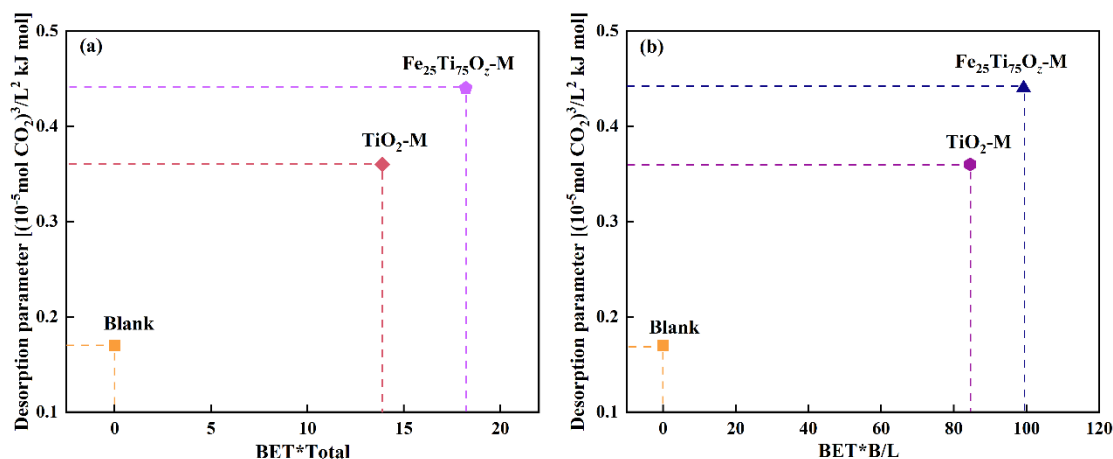
Catalysts	BET surface area ^a (m ² /g)	Pore volume ^b (cm ³ /g)	Pore diameter ^c (nm)
Fe ₂ O ₃ -M	36.1	0.19	16.3
TiO ₂ -M	100.6	0.17	4.5
Fe ₂₅ Ti ₇₅ O ₂ -M	125.6	0.21	4.7

^a BET specific surface area

^b BJH desorption pore volume

^c BJH desorption pore diameter

352 **3.3 Structure-Activity Relationship**



353 Fig.8. Influence of catalyst properties on CO₂ desorption performance: BET*Total (a) and BET*B/L (b).

354 As discussed above, the Fe₂₅Ti₇₅O₂-M catalyst exhibits superior CO₂ desorption performance
 355 compared with TiO₂-M due to its combination of a high BET surface area and abundant weak-acid
 356 sites, even though TiO₂-M possesses a greater total number of medium-strength acid sites and a
 357 larger Brønsted-to-Lewis (B/L) ratio. This indicates that the overall desorption efficiency arises
 358 from the cooperative interplay of multiple physicochemical factors including the total acidity, the
 359 Brønsted (B) sites, Lewis (L) sites, the B/L ratio, and the specific surface area. Fig. 8 illustrates the
 360 correlations between these properties and CO₂ desorption performance. Within it, Fig. 8(a) reveals
 361 a clear positive correlation between the desorption parameter and the BET × Total-acidity product,
 362 demonstrating that surface area and total acidity act synergistically to accelerate CO₂ release. A
 363 larger surface not only accommodates more acid sites but also increases the collision frequency with
 364 carbamate species, thereby raising the reaction rate. More interestingly, Fig. 8(b) shows an equally
 365 pronounced positive correlation between the desorption parameter and BET×B/L, with a sharp
 366 increase from TiO₂-M to Fe₂₅Ti₇₅O₂-M, emphasizing the critical role of Brønsted acidity in
 367 carbamate decomposition. It is explained that Brønsted sites directly donate protons to carbamate
 368 molecules, lowering the activation energy and reducing the heat requirement for amine regeneration.
 369

370 **4. Conclusions**

371 In this study, a novel FeTiO₂-M composite catalyst was successfully developed via a
 372 microwave-assisted co-precipitation method, and the Fe:Ti molar ratio was systematically
 373 optimized to enhance CO₂ catalytic desorption from CO₂-rich MEA solutions. The results
 374 demonstrated that rational design of bimetallic solid-acid catalysts substantially improved both the

375 kinetics and energy efficiency of amine regeneration. At the optimized Fe:Ti ratio of 25:75, the
376 loading of 0.4 wt. % catalyst resulted in the average CO₂ desorption rate increase by 36 % and the
377 relative heat duty decreased by 25.7 %, thereby yielding a relative desorption parameter of 258.8 %
378 with stable performance maintained over five cycles. Therefore, the catalyst not only significantly
379 accelerated CO₂ desorption while reducing regeneration energy, but also exhibited certain cyclic
380 stability over multiple absorption-desorption cycles, and this result laid a foundation for subsequent
381 long-term continuous operation tests to explore its practical application feasibility. Mechanistic
382 investigations revealed that the superior performance arisen from the synergistic interplay of
383 structural and chemical properties, and the formation of an amorphous Fe-Ti solid solution increased
384 mesoporous surface area and pore volume, facilitating carbamate diffusion. Furthermore, the
385 introduction of sulfate groups activated surface hydroxyls and metal acid sites, providing abundant
386 protons for carbamate decomposition, and the balanced distribution of Brønsted and Lewis acid
387 sites ensured efficient CO₂ liberation with minimal energy input. These findings underscore the
388 critical role of tailored surface chemistry and hierarchical porosity in optimizing catalytic
389 performance.

390 **CRedit authorship contribution statement**

391 **Mengyao Wang:** Writing - original draft, Methodology, Validation. **Yanping Du, Haoran Zhang and Junge**
392 **Lv:** Validation and Methodology. **Huancong Shi, Wei Lu and Jing Jin:** Investigation, Supervision, Writing -
393 review & editing, Funding acquisition. **Zhibo Xiong:** Conceptualization, Funding acquisition, Writing - review &
394 editing.

395 **Declaration of Competing Interest**

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

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