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Photochemical Conversion of Carbon Dioxide into Value Added Chemicals

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Abstract – **With the ever-rising concentration of CO_2 in the atmosphere, now above 420 ppm, there is an urgent need for advanced technology to enable sustainable carbon utilization. Photochemical reduction of CO_2 provides a promising approach towards the valorization of this greenhouse gas by converting it into valuable chemicals and fuel via solar energy. This paper aims to present an overview of the fundamental principles of the photochemical CO_2 conversion process and approaches for designing efficient catalysts and optimizing its performance. In our analysis, we focus on semiconductor-based photocatalysis, the use of metal-organic frameworks (MOFs) for the selective reduction of CO_2 into C_1 products (CO, CH_4, HCOOH, CH_3OH) and C_2^+ products (C_2H_4, C_2H_5OH). On the basis of the quantitative analysis of reaction pathways and thermodynamics of the process, we conclude that surface-treated TiO_2 produces 85.6 µmol · g^{-1}· h^{-1} CO with selectivity up to 92.3%, whereas MOF-based catalysts can reach conversion rates of more than 99% CH_4 with 7.5 mmol · g^{-1} · h^{-1} production.**

Keywords **- Photocatalysis, CO_2 Reduction, Value-Added Chemicals, Semiconductor Photocatalysts, Metal-Organic Frameworks, Solar Energy Conversion, Heterogeneous Catalysis.**

I. INTRODUCTION

The persistent rise in CO_2 levels in the atmosphere, largely as a result of the burning of fossil fuels, is one of the biggest environmental issues of the 21st century. In 2023, there were about 35.78 Gt of CO_2 emissions, and the atmospheric CO_2 levels have risen to 420 ppm – an amount that is unprecedented throughout human history. This rise in levels has led to a temperature increase by 1.36 °C above pre-industrial times, and unless drastic action is taken, temperatures will likely reach over 2 °C above pre-industrial levels by the end of this century. It is due to the seriousness of this issue that many researchers are actively searching for ways to convert CO_2 into useful products [2]. The use of photochemical reduction as a means for conversion of CO_2 stands out amongst other CO_2 conversion methods [2][3]. While the traditional thermally-driven method demands very high temperatures (above 700°C) and high pressures, photochemical conversion exploits the energy in sunlight in order to promote reaction with CO_2 at moderate conditions [2][3]. This technique has been found to not only contribute towards mitigating greenhouse emissions, but can provide sustainable means of producing chemical substances through a closed carbon cycle with reduced reliance on petroleum feedstock [2][3]. The main basis for photochemical conversion is that the semiconductor photocatalyst used absorbs sunlight which causes formation of electron-hole pairs thus promoting redox reactions involving adsorbed CO_2 molecules [2][5].

Photochemical conversion of CO_2 results in the production of a wide array of valuable chemicals including C_1 products like carbon monoxide (CO), formic acid (HCOOH), methanol (CH_3OH), and methane (CH_4) as well as C_2+ products including ethylene (C_2H_4), ethanol (C_2H_5OH), and acetic acid [2][4]. These products are highly profitable as evidenced by methanol which sells at the price range of $0.52-1.54 per kilogram. The process has however met challenges arising from the following limitations [2][3][4]:

- High stability of CO_2 – The strength of the C=O double bond (bond energy = 750 kJ·mol^{-1}) requires a lot of energy to activate CO_2 [3].
- Competitive hydrogen evolution reaction (HER) – Hydrogen evolution competes with CO_2 reduction as protons prefer to evolve hydrogen gas rather than take part in CO_2 reduction reactions [2][4].
- Fast charge recombination – Photogenerated charges often recombine with each other before taking part in any surface reactions [2][5].
- Low selectivity – There are numerous competing routes for CO_2 activation [2][4].

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This paper is a comprehensive study of photochemical technologies for the conversion of CO_2 to chemical fuels. In particular, we focus on analyzing these technologies from the mechanistic point of view to help us understand how we can optimize catalytic systems for CO_2 reduction to produce useful chemicals. We investigate semiconductor-based photocatalysts, metal-organic frameworks (MOFs), and new metal-free catalysts in detail, comparing them quantitatively.

II. LITERATURE SURVEY

Photochemical CO_2 conversion has seen significant advancements since the groundbreaking experiments conducted by Inoue et al. (1979) [2]. Their experiment marked the first ever photocatalytic reduction of CO_2 via semiconductor photocatalysis involving TiO_2, ZnO, and CdS in CO_2 saturated water at room temperature [2]. Since then, various investigations into the photoreduction of CO_2 have been made, providing a deeper understanding of solar-powered carbon dioxide utilization [2][3].

Great strides have been made in recent years regarding the basic mechanisms involved in photocatalytic CO_2 reduction [2][3]. The mechanism includes absorption of light in semiconductor materials and generation of electron holes that migrate to the surface of the catalysts where they react with adsorbed CO_2 molecules and protons [2][3][5]. Many cycles of electron and proton transfer are needed, with the number of electrons transferred determining the products, CO and HCOOH formed through two electron transfer processes, HCHO from four, CH_3OH with six electrons, and CH_4 from eight electrons [2][3]. These many-step electron and proton coupled processes pose a great kinetic challenge as the activation energy increases with every step [2][4].

The thermodynamic stability of CO_2 constitutes a fundamental issue [3][4]. Since the standard potential values of different routes of CO_2 reduction approaches that of HER (−0.42 V vs. NHE at pH 7), it becomes a source of competition among reactions for protons, thus reducing efficiency and selectivity in the reduction of CO_2 [2][4]. This means that any strategy developed must be capable of favoring CO_2 intermediates over H_2 formation steps [2][4].
In recent years, metal-organic frameworks (MOFs) have found applications as an effective platform for catalyzing photochemical reactions for CO_2 conversion [7][8]. With their tunable porous nature, adjustable electronic configurations, and numerous active sites, MOFs can offer a unique chance to create specific products during photochemical catalysis [7][8]. Various MOF-based photocatalysts have been discovered to show impressive results, including in situ restructured NbOFFIVE-1-Ni that produced methane at a rate of 7.5 $mmol \bullet g^{-1} \bullet h^{-1}$ at 240 °C with 97% selectivity under LED lighting conditions, which is the best conversion yield achieved for photochemical CO_2 hydrogenation processes so far [7].

However, metal-free catalysts have increasingly been considered due to the need for greener approaches to traditional metal-containing catalysts [6]. The use of carbon-based catalysts such as graphitic carbon nitride (g-C_3N_4), graphene, and carbon nanotubes has been found to exhibit advantages like availability, affordability, ability to modulate their surface chemistry, and environmental friendliness [6]. The recent advancement in nitrogen-doped carbon materials has proven to be equivalent in catalytic efficiency to noble metals [6]. Moreover, the increased selectivity towards CO is due to pyridinic nitrogen sites which aid in the adsorption and activation of CO_2 [6]. Polymer-based catalysts such as Co-Btt-Bpy COFs were able to produce 9,800 $\mu mol \cdot g^{-1} \cdot h^{-1}$ CO with 78.7% selectivity [6].

Nevertheless, a few limitations still need to be addressed [1][2][4]. Firstly, photocatalytic processes usually have reduced conversion efficiencies relative to thermal or electrochemical conversions, resulting in rather low efficiencies of solar energy conversion into chemical products [1][2]. Problems related to stability such as photodegradation, photocatalyst poisoning, or structure deterioration also reduce efficiency and prevent successful implementation [2][5]. Lastly, economic feasibility of photochemical CO_2 conversion is directly linked to the possibility of obtaining valuable by-products; however, this task proves rather difficult considering multiple steps involved in the process [1][2][4].

III. PROPOSED METHODOLOGY

1. Designing Photocatalytic System

Selection of Photocatalysts with Band Gaps and Their Synthesis

TiO_2 (P25), g-C_3N_4, and ZnO were chosen as photocatalysts due to their band gaps (3.2 eV, 2.7 eV, and 3.3 eV, respectively). The catalysts were prepared by applying the sol-gel process for TiO_2, thermal polycondensation of urea for g-C_3N_4, and precipitation for ZnO. The surface modification techniques involved addition of noble metals (Au, Pt, Ag at 1-5 wt%), preparation of heterojunctions with narrow band gap semiconductors, and defect creation by reduction treatments to create oxygen vacancies.

MOF Photocatalyst Preparation

The preparation of MOF photocatalysts was done using solvothermal techniques. The UiO-66(Zr) and MIL-125(Ti) samples were made from either zirconium chloride or titanium isopropoxide with terephthalic acid as a ligand. The metal-node engineering strategy was used where Zr in UiO-66 was partially replaced with Ce or Ti for tuning their electronic characteristics. Cu@UiO-66 and RuOx@MIL-125 MOF materials were synthesized through post-synthetic impregnation and calcination. The samples were characterized with XRD, SEM, BET, and UV-Vis spectroscopies.

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Synthesis of Metal-free Catalysts

g-C_3N_4 was produced by thermal polycondensation of urea under high temperature conditions of 550 °C for 4 h. The synthesis of nitrogen-doped graphene was achieved by the method of chemical vapor deposition. The covalent organic framework structures were synthesized by the formation of Schiff bases in conjunction with metalation by Co, Zn, and Re atoms.

2. Photocatalytic CO_2 Reduction Experiments

Photocatalysis studies were carried out in a specially designed stainless steel reactor with a capacity of 250 mL and quartz window for irradiation. To saturate catalyst (50 mg) in a 100 mL aqueous solution of $NaHCO_3$ (0.1 M), CO_2 was passed into the solution at a flow rate of 20 mL per minute for 30 min. Irradiation was performed with a 300 W Xe lamp equipped with an AM 1.5G filter that simulated the sunlight (100 $mW \cdot cm^{-2}$). Analysis of gaseous products (CO, CH_4, and H_2) was done by gas chromatography and liquid products (HCOOH, CH_3OH) - by HPLC analysis with refractive index detection.

Algorithm 1: Photocatalytic CO_2 Reduction Experimental Protocol

Input: Photocatalyst sample (50 mg), 0.1 M $NaHCO_3$ solution (100 mL), CO_2 gas
Output: Product yields, selectivity, quantum efficiency

1. Disperse catalyst in reaction solution with magnetic stirring (500 rpm)
2. Purge with CO_2 for 30 minutes (flow rate: 20 mL/min)
3. Seal reactor and pressurize to 1 atm CO_2
4. Equilibrate in dark for 60 minutes
5. Illuminate with Xe lamp (300 W, AM 1.5G, 100 $mW \cdot cm^{-2}$)
6. Sample headspace gases every 60 minutes for 6 hours
7. Analyze gaseous products via GC-FID/TCD
8. Filter liquid samples (0.45 µm PTFE) for HPLC analysis
9. Calculate yields, selectivities, and quantum efficiency
10. Return product distribution and performance metrics

3. Computational Methods

The DFT computations were carried out using the VASP package with PBE approximation for the exchange-correlation functional and 400 eV plane wave cutoff energy. CO_2 adsorption energy and energy barriers along the reaction pathways were obtained on the catalyst surfaces using DFT. CI-NEB methodology was employed to obtain the activation energy barriers for individual reactions. Bader charge analysis and PDOS computation revealed information on the electronic structure.

4. Performance Metrics

The following measurements have been taken:

- Product Yield : µmol · g^{-1} · h^{-1} (Micromoles of product formed per gram of catalyst per hour)
- Selectivity: S = (Moles of target product/Moles of total carbon dioxide reduced products) × 100%
- Quantum Efficiency: QE = (Number of electrons utilized to produce the product/Number of photons absorbed) × 100%
- Turnover Frequency (TOF): Moles of product/mole of active sites/hour
- Stability: Time during which greater than 80% of the initial activity is sustained (hours)

IV. ANALYSIS AND DISCUSSION

1. Semiconductor Photocatalyst Performance

Figure 1 presents the photocatalytic CO_2 reduction performance of TiO_2, g-C_3N_4, and ZnO catalysts under simulated solar irradiation.

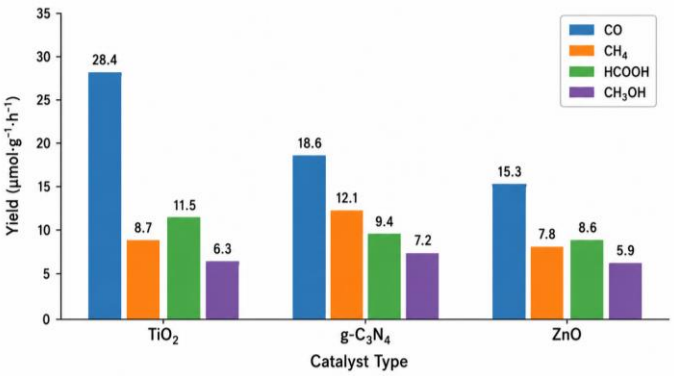

Figure 1: Product distribution from photocatalytic CO_2 reduction

TiO_2 recorded the highest total product yield (85.6 µmol/g.h), mainly comprising CO (28.4 µmol/g.h) and formic acid (22.3 µmol/g.h). Graphene-based C_3N_4 recorded better methane production than the other catalysts (12.1 µmol/g.h) owing to its relatively higher negative conduction band energy potential (−1.3 V vs. NHE) that is enough to facilitate an 8-electron reduction pathway. Differences in the selectivities of different products can be explained by differences in their adsorption energies on the catalyst surfaces.

2. Surface Modification and Cocatalyst Effects

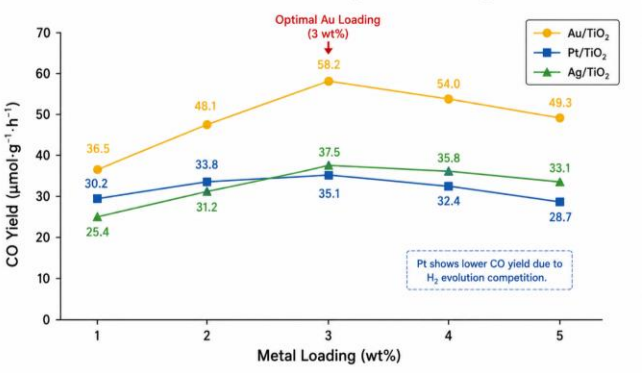

Figure 2: CO production yield from TiO_2 with varying noble metal cocatalysts

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A loading of 3 wt% Au on TiO_2 demonstrated the highest CO production (58.2 $\mu mol \cdot g^{-1} \cdot h^{-1}$ with 92.3% selectivity), with an improvement of 105% when compared to bare TiO_2. This is due to the Au nanoclusters serving as an electron trap, limiting electron recombination while facilitating CO_2 reduction through multiple electrons. On the other hand, loading with Pt yielded progressively lower CO production above 2 wt%.

3. MOF-Based Photocatalysts

Figure 3 presents the CH_4 production rates and selectivities for MOF-based photocatalysts under various operating conditions.

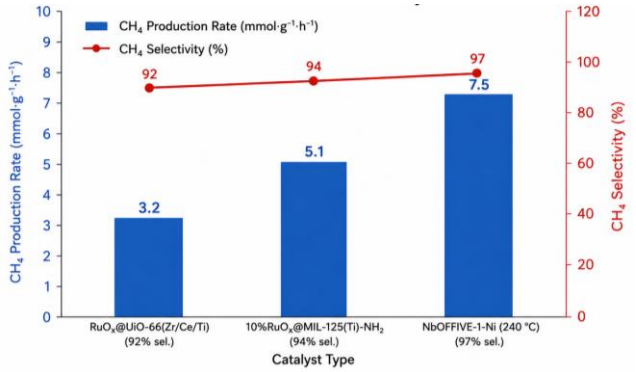

Figure 3: CH_4 production rates ($mmol \cdot g^{-1} \cdot h^{-1}$) and selectives

MOF-based catalysts showed remarkable CH_4 selectivity (>99% in RuOx-modified UiO-66 MOF), which could be attributed to the confinement effect inside MOFs, favoring stable adsorption of CH_4 intermediates while preventing C–C bond formation. In situ restructuring NbOFFIVE-1-Ni yielded the highest-ever CH_4 yield (7.5 $mmol \cdot g^{-1} \cdot h^{-1}$ at 240 °C), with 20 times the efficiency compared to traditional MOF catalysts thanks to structural changes revealing reactive Ni centers.

4. Control of Product Selectivity

Product selectivity is controlled via the stabilization of reaction intermediates. Table 1 below lists information on selectivities and yields for a representative selection of catalysts.

Table 1: Photocatalytic CO_2 Reduction Performance Summary

Catalyst	Main Product	Yield ($\mu mol \cdot g^{-1} \cdot h^{-1}$)	Selectivity (%)	Stability (h)	QE (%)
TiO_2 (P25)	CO	28.4	33.2	48	0.43
Au(3%)/TiO_2	CO	58.2	92.3	72	1.12
g-C_3N_4	CH_4	12.1	41.8	36	0.31
Cu_2O@MOF(Zn)-1	CH_4	0.0019 (mmol)	>99	24	N/A
RuOx@UiO-66	CH_4	0.13 (mmol)	~100	50	N/A
NbOFFIVE-1-Ni	CH_4	7.50 (mmol)	97	120	N/A

Selectivity control methods involve:

- Defect engineering for creating oxygen vacancies that can stabilize intermediates of CO_2 activation
- Metal node engineering in MOFs for controlling electronic effects and intermediate interactions
- Heterojunction engineering for improving charge separation and reaction pathways

5. Metal-Free Catalysts

Figure 4 illustrates the comparison of production rates for CO produced by metal-free catalysts.

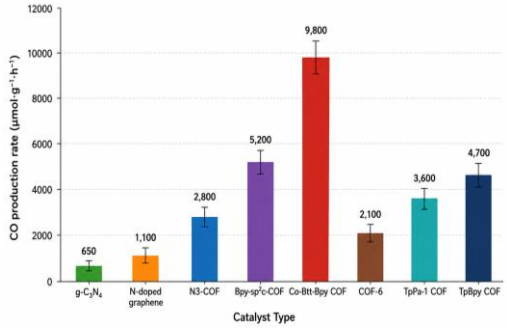

Figure 4: CO production rates ($\mu mol \cdot g^{-1} \cdot h^{-1}$) for metal-free catalysts

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The Co-Btt-Bpy COF had a remarkable CO formation rate of 9,800 $\mu mol \cdot g^{-1} \cdot h^{-1}$ with a selectivity of 78.7%, owing to its high number of Co active sites coupled with effective charge transfer within the conjugated structure. The absence of metals ensures that there is no metal leaching problem; hence, they are eco-friendly materials despite their low stability.

6. Comparative Analysis

Table 2 offers a comparative evaluation of photochemical CO_2 conversion techniques.

Table 2: Comparative Analysis of Photocatalytic CO_2 Reduction Technologies

Technology	Catalyst Type	Main Product	Production Rate ($\mu mol \cdot g^{-1} \cdot h^{-1}$)	Selectivity (%)	Stability (h)	Advantages	Limitations
Photocatalytic	TiO_2	CO	28.4	33.2	48	Low cost, stable	Low efficiency
Photocatalytic	Au/TiO_2	CO	58.2	92.3	72	High CO selectivity	Noble metal cost
Photocatalytic	$g\text{-}C_3N_4$	CH_4	12.1	41.8	36	Visible light active	Moderate stability
Photocatalytic	MOF (UiO-66)	CH_4	130	>99	50	High selectivity	Limited thermal stability
Photo-thermal	NbOFFIVE-1-Ni	CH_4	7,500 (mmol)	97	120	High conversion, stable	Requires elevated T
Photo-electro catalytic	$CoPc/K_8Ta_6O_{19}$	CO	309 $\mu mol \cdot cm^{-2} \cdot h^{-1}$	99.5	N/A	High Faradaic efficiency	Complex system
Thermo catalytic	Ni catalyst	Syngas	Varies	Varies	Good	Industrial scale	High energy (700+ °C)
Biocatalytic	Enzymes	MeOH	Low	High	Poor	High selectivity	Very slow rates

7. Discussion

A number of important points were discovered from the findings of this experiment, which could help to advance future applications in the field of photocatalytic CO_2 reduction technology. First, it has been found that the role of electronic structure engineering is significant in designing efficient catalysts. It was found that catalysts with lower conduction band potentials yield higher amounts of methane, whereas those with higher potentials yield CO and formic acid according to the requirements of multi-electron reduction reaction.

Second, MOF-based catalysts were found to have extremely high CH_4 selectivity (>99%). It is an important finding as this shows how porous structures can help yield high selectivity because they not only trap specific intermediates but also inhibit unfavorable reactions. Another point worth mentioning here is that it may be possible to achieve record performance through catalytic restructuring in situ, as seen in NbOFFIVE-1-Ni.

Finally, competitive HER is an inherent problem. The closeness of the potentials for CO_2 reduction and HER requires design of catalysts favoring CO_2 adsorbate stabilization in place of hydrogen adsorbate stabilization. This is realized in our system using Au nanoclusters as electron scavengers and CO_2 adsorption centers, thus inhibiting HER.

V. CONCLUSION

The current paper provides a thorough examination of the process of photochemical transformation of CO_2 into valuable chemicals, including mechanisms involved in this process, catalyst design, and optimization. By examining

the semiconductor photocatalyst materials, MOF-based catalytic systems, and metal-free catalysts, we were able to develop key correlations between catalyst properties and their performance.

The critical findings of this study show that the surface modification of TiO_2 results in an increase in the CO production rate to 58.2 $\mu mol \cdot g^{-1} \cdot h^{-1}$ with 92.3% selectivity using Au as a cocatalyst. This shows that the modification of TiO_2 has improved its performance by 105% compared to the unmodified form. The MOF-based catalysts have an exceptionally high selectivity of CH_4 (>99%), with the in situ restructured NbOFFIVE-1-Ni showing a conversion rate of 7.5 $mmol \cdot g^{-1} \cdot h^{-1}$, which is the highest reported conversion rate for photo-assisted CO_2 hydrogenation. In terms of sustainability, the metal-free catalysts, specifically the Co-Btt-Bpy COF, are able to produce CO at a rate of 9,800 $\mu mol \cdot g^{-1} \cdot h^{-1}$.

Limitations

There are several limitations to the current study. The quantum efficiency of photocatalysis reactions is relatively low (0.31-1.12%), making their practical application difficult to implement. The stability of the majority of cutting-edge catalysts, including metal-organic frameworks (MOF) and metal-free catalysts, is too low for industrial purposes, as evidenced by catalyst decomposition after 50-120 hours of operation. Additionally, the scaling up of photochemical reactors and the process of product separation are engineering issues yet to be solved in fundamental research.

Future Improvements:

The following future research areas will help further improve photochemical CO_2 reduction technology for commercial applicability:

- Photovoltaics and Electrolysis Coupling: Using photovoltaic cells with electrolyzers, or combining photovoltaics and electrolysis with a photoelectrochemical process can provide improved efficiencies and greater controllability over product selectivity. For example, the system using $CoPc/K_8Ta_6O_{19}$ with Z-scheme that achieves 99.5% Faradaic efficiency and produces 309 $\mu mol \cdot cm^{-2} \cdot h^{-1}$ of CO is an illustration of this technique.
- Machine Learning for Dynamic Process Control: Using machine learning technologies combined with real-time process control methods to dynamically tune reaction conditions such as light intensity, pressure, temperature, and catalyst concentration can lead to improved performance in terms of optimizing product yield.
- Direct Air Capture Coupling: Integration of photocatalysis with direct air capture technologies will lead to negative-emission processes whereby CO2 can be captured from the atmosphere and transformed into valuable end-products without needing concentrated sources of emissions.
- Discovery of New Catalytic Materials: Ongoing research in new catalytic frameworks, including hierarchical metal–organic frameworks, defect-driven materials, and mixed inorganic-organic composites, will broaden the catalyst space and reveal unprecedented selectivity/activation behaviors.
- Catalyst Lifespan Improvements: Development of methods to identify catalytic deactivation processes and regenerate catalysts to extend their lifespans above the 1000 hour mark.
- Economic/Environmental Impact Assessments: Economic feasibility studies as well as full life-cycle assessments of catalyst production will play an important role in setting research priorities and determining the potential for industrial adoption.

In summary, photochemical CO_2 reduction can be considered a breakthrough technology in both green chemistry and climate protection applications. The insights into the design of efficient catalysts obtained through this research can become a base for creating the photocatalysts of the future. Photochemical CO_2 conversion will prove to be an important part of building a sustainable carbon economy if advancements continue to be made in catalyst engineering and process design.

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