IAEA Technical Meeting on Emerging Applications of Plasma Science & Technology

IAEA International Atomic Energy Agency
Atoms for Peace

Non-thermal plasma catalytic dry reforming of methane over Ni-Co_3O_4 supported modified-Titania catalysts: Effect of process conditions on syngas production and DFT analysis

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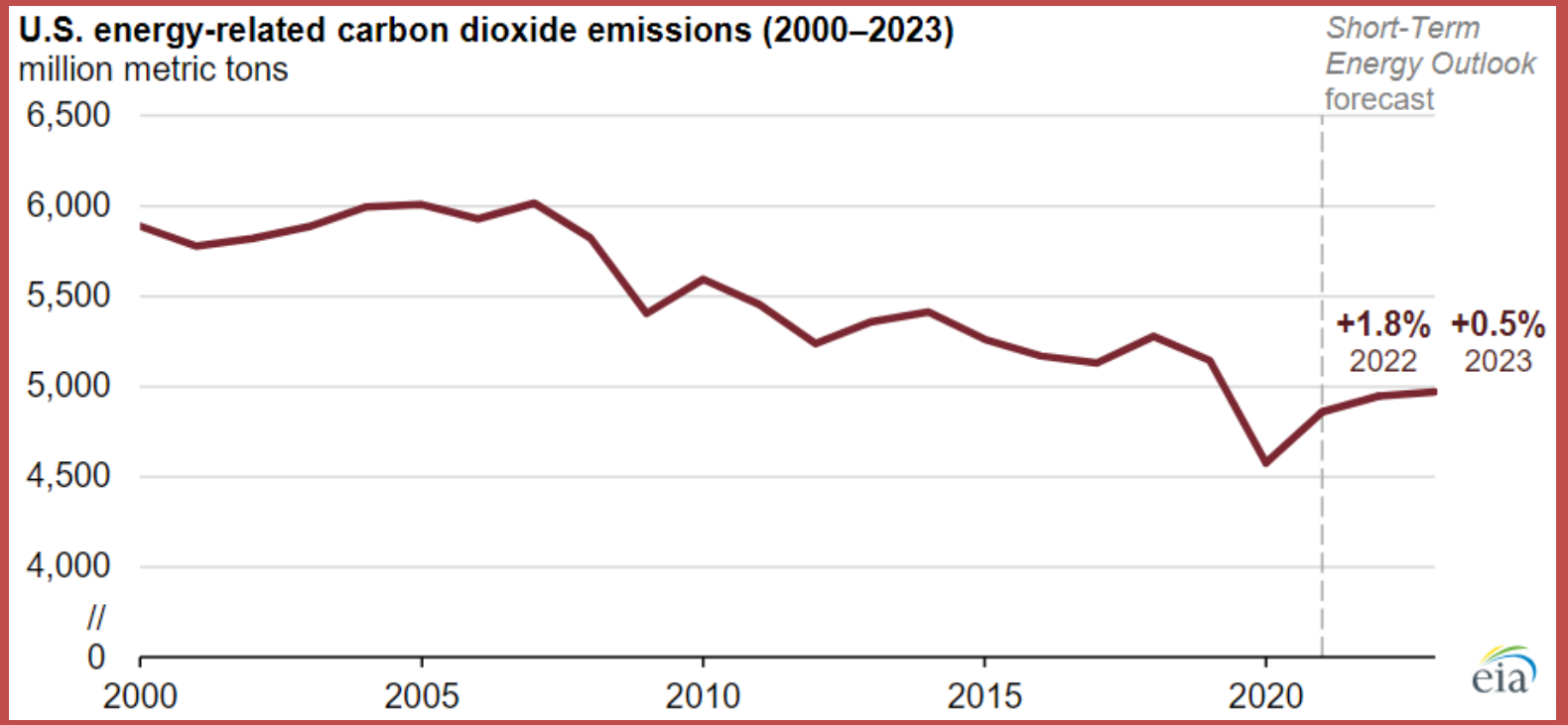
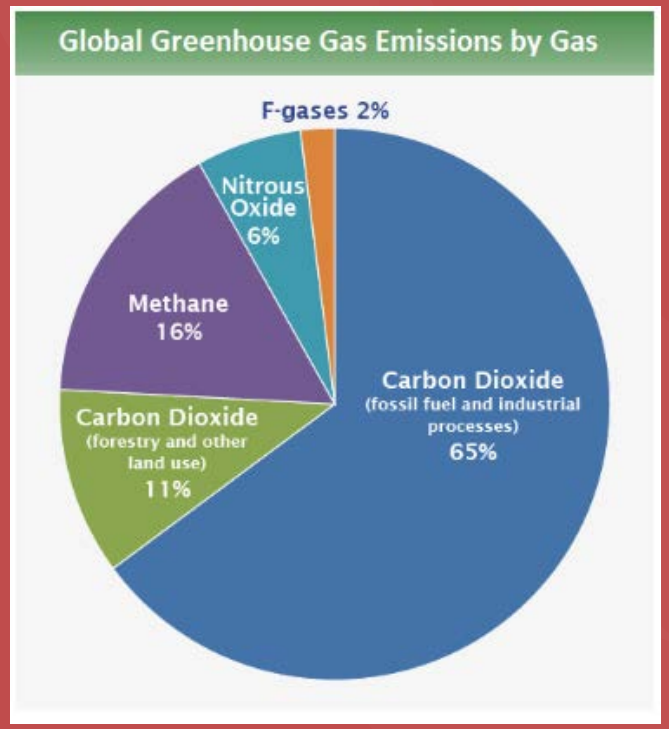
Innovating Solutions

Research Background





Source: U.S. Energy Information Administration, *Short-Term Energy Outlook*

CO_2 emissions increase in 2023

CO_2 & CH_4 remain major constituents of GHG

Fossil fuel consumption is primary source of global GHG emissions

Research Background

Initiatives to tackle greenhouse gases (CO_2-CH_4)

IAEA aims to reduce greenhouse gas emissions

UNDP is committed to 50% carbon footprint reduction by 2030

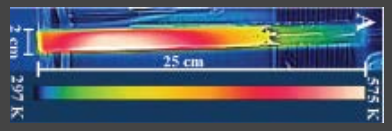
Paris Agreement

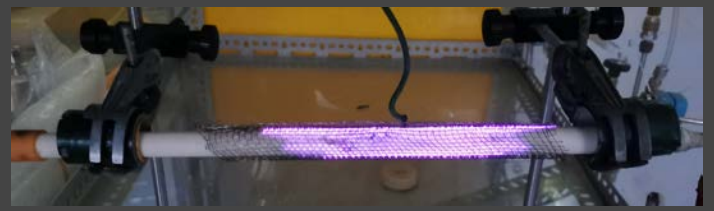
NET ZERO 2050

Solution

Non-Thermal Plasma Technology

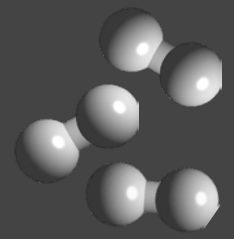
for catalytic dry reforming of CH_4 to produce syngas (H_2-CO)



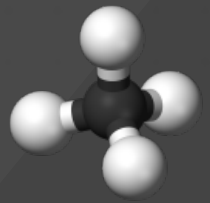


Non-Thermal Plasma Plasma

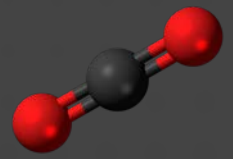
Catalyst



Hydrogen

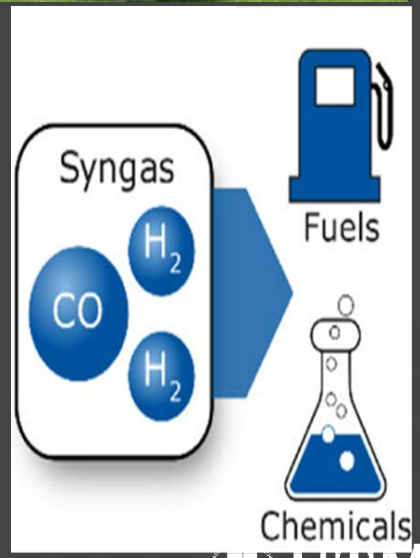


Methane



Carbon dioxide

Carbon monoxide



Global Syngas Demand & Market Size

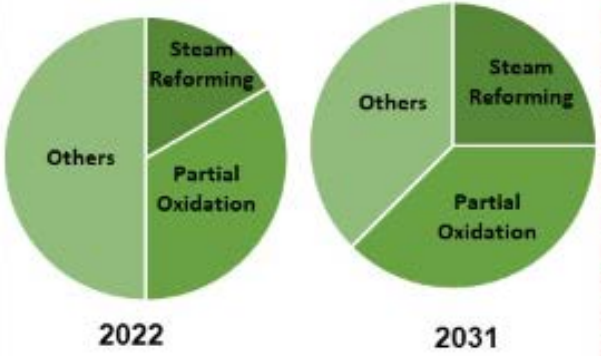
North America

Market Volume:
2,80,630 MWth (2022)

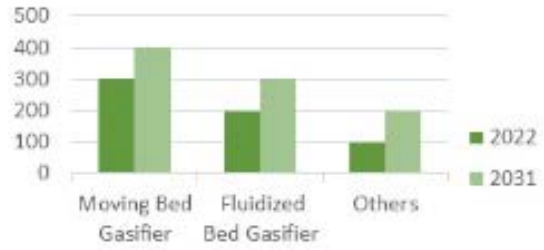
CAGR
(2023-2031)

11.6%

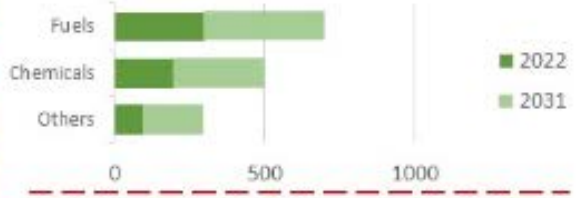
By Production Technology:



By Gasifier Type:



By Application:



Key Players:

BASF We create chemistry
Linde
KBR
SIEMENS
AIR PRODUCTS
DOW

Market size value in 2022	USD 2,80,630 MWth
Volume forecast in 2031	USD 7,38,662 MWth
Growth rate CAGR	CAGR of 11.6% from 2023 to 2031

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Global Market & Expected Economic Analysis

Estimated Lab Scale Yearly Total Revenue

$15 M
CO

CO Production Yearly = 96 kg

Output/operation hrs:
3000ml/hour, 8h/day, 20 days/month
Total CO per month: 9.6 l
Parameter: 60 sccm FFR, 1.5 cm ID & 1 kW power
Price per 50 liter = $ 6586.39

Hydrogen Production Yearly = 407.7 kg

Output/operation hrs:
3000ml/hour, 8h/day, 20 days/month
Total H_2 per month in liter = 480 l
Total H_2 per year in liter = 5760 l
Price per kg = $ 21.28

$8,676
Hydrogen

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Literature Review

Feed	Catalyst and Parameters	Product	Reference
Steam–CH_4, CO_2 gas mixture	Catalyst = Cu/ZnO/Al_2O_3/MgO Plasma power = 20 W, Feed flow: Steam = 25 ml/min, S/C = 4.5, catalyst = 200 mg	H_2 = 88 % CO = 25 % CO2 = 65 %	(Geng et al., 2022)
Water–CH_4 gas mixture	Catalyst = Cu/CeO2 Plasma SIE = 19.8 J/L, Feed flow: Steam = 0.5 ml/min, CH_4 = 50 ml/min, catalyst = 1 g	H2 = 248.7 CO = 11.25 ($\mu molg^{-1}h^{-1}$)	(Bajpai et al., 2023)
CH_4, CO_2 – Ar gas mixture	Catalyst = Ni/Al_2O_3 Plasma power = 3.9 W, Feed flow = 60 ml/min, CH_4/CO_2 = 1, catalyst = 12 pallets	H_2 = 42 % CO = 34 %	(Stanley et al., 2023)
CH_4 - CO_2 gas mixture	Catalyst = Ni/CeO2/C Plasma power = 40 W, Feed flow: Feed flow = 50 ml/min, CH_4/CO_2 = 1	H_2 = 50.0 % CO = 53.2 %	(Wang et al., 2020)
CH_4 - CO_2 gas mixture	Catalyst = Ni/$CeZrO_2$ Plasma power = 200 W, Feed flow: Feed flow = 50 ml/min, CH_4/CO_2 = 1, catalyst = 0.5 g	H2/CO = 0.98	(Dai et al., 2021)

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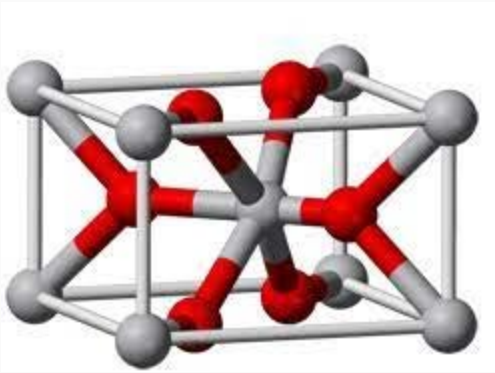
Why Ni-Co_3O_4/TiO_2?

Why TiO_2 as Catalyst Support

- Environmental-friendly, nontoxic and inexpensive semiconductor material .
- Mainly used in photo-catalysis reaction. However, lack of studies using TiO_2 as catalyst support in other applications including Dry reforming of methane (DRM).
- Advantages of TiO_2 as a catalyst support:
  - Acts as active metal support
  - Also acts as the reducible oxide (catalyst).
  - Provides unique electronic interactions between the metal & support.



Why Ni active metal and Cobalt as Catalyst Promoter

- Nickel (Ni) → abundant, commercially used catalyst & active metal in thermal/nonthermal applications
- However, Ni suffers from instability and deactivation.
- To tackle these problems, noble metals like Pt, La, Au, and Ag can be used as catalyst promoters, but these expensive and rare metals makes the catalyst commercially infeasible.
- Cobalt (Co) → as catalyst promoter, Co can increase stability and activity of Ni.

  → Inexpensive as compared to noble and rare earth metals

Methodology

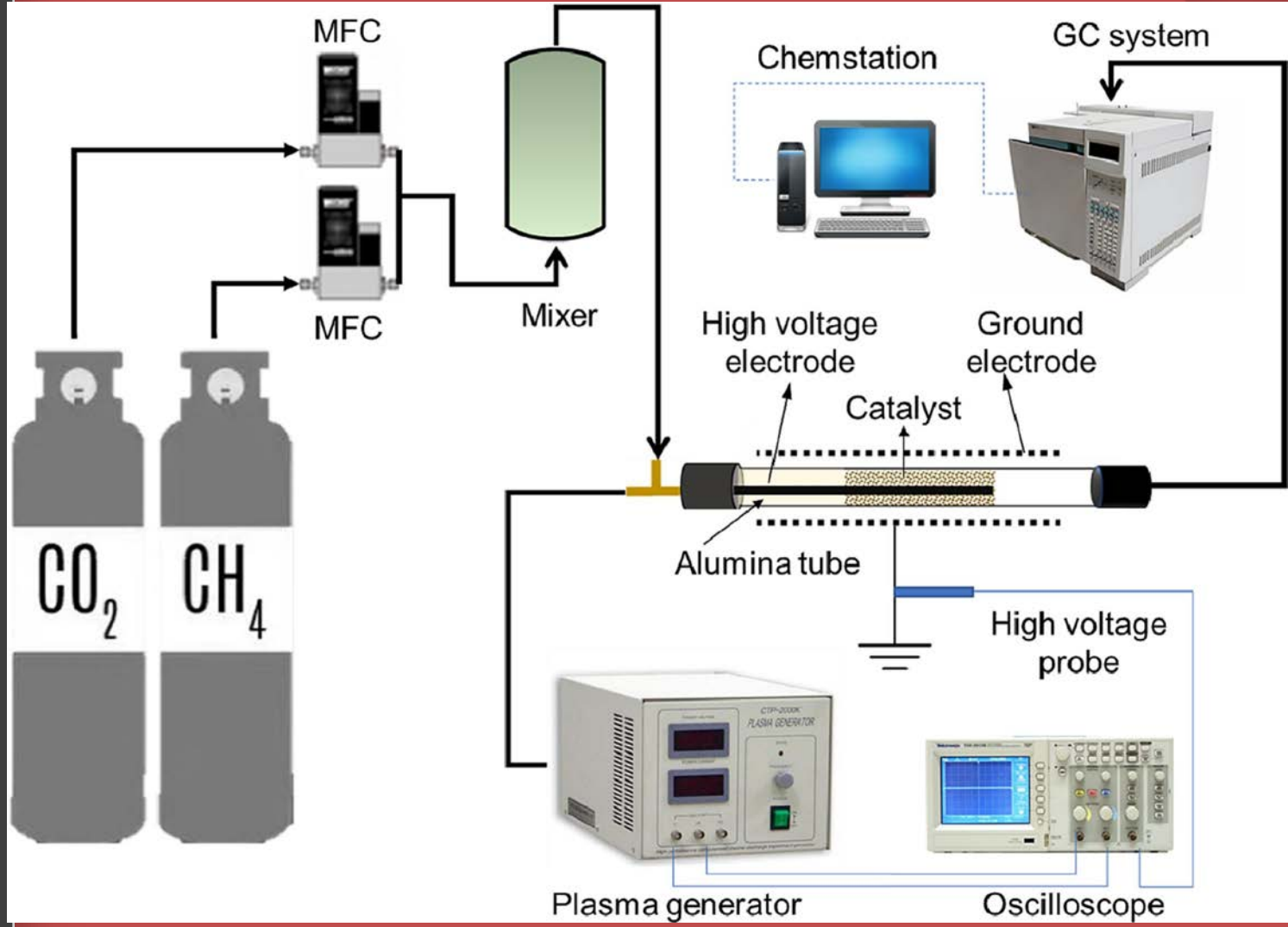
Alumina tube packed-bed DBD dimensions: Length = 40 cm, ID = 10 cm, OD = 12 cm

High voltage (HV) positive electrode: steel tube = 4 mm diameter

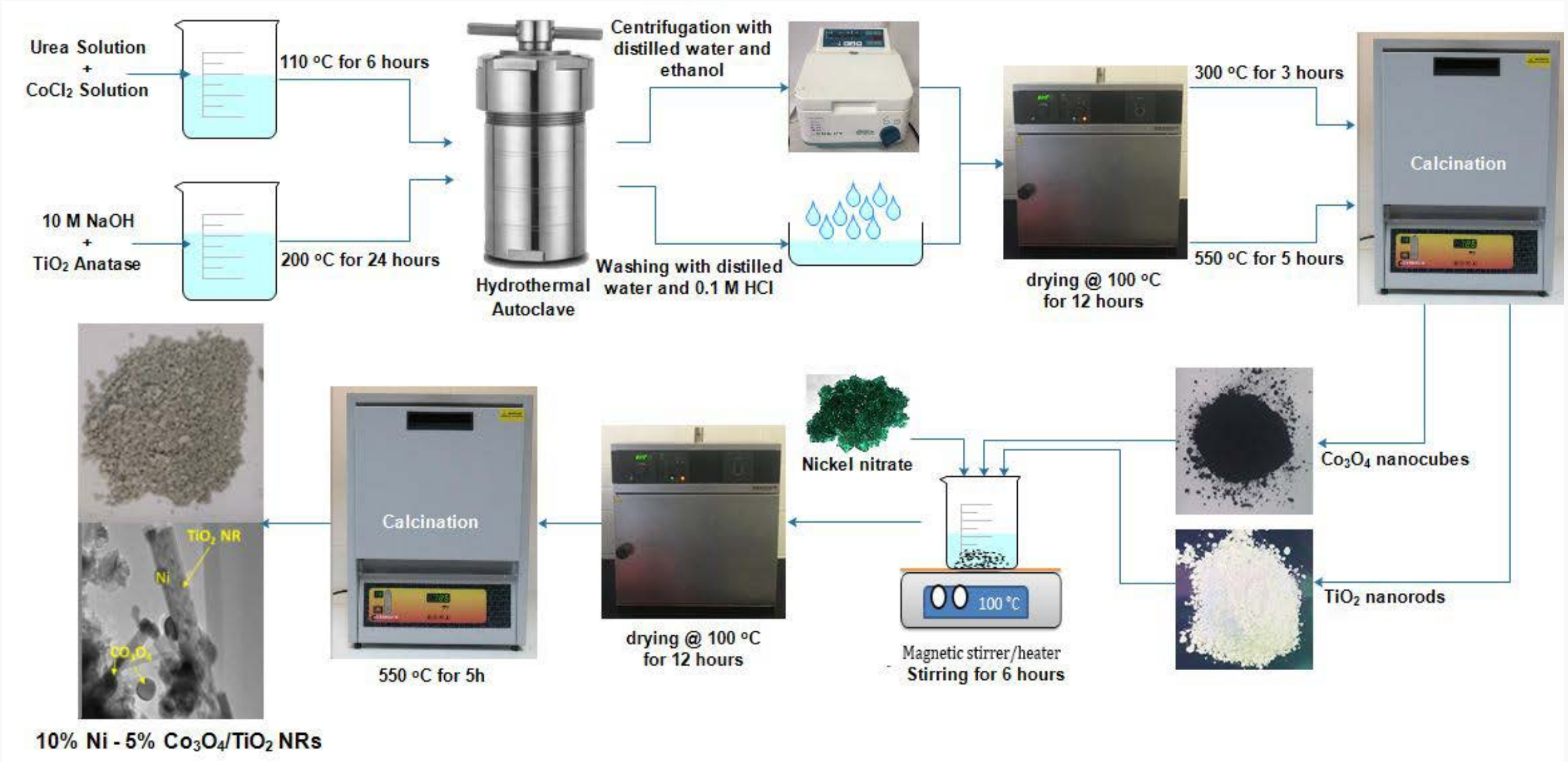
Ground electrode: stainless-steel mesh = 20 cm

Reaction Conditions: catalyst loading (0.3 g), total feed flow rate (20 ml min-1), CH_4/CO_2 feed ratio (1/1), GHSV (1200h-1) and SIE (300 J ml-1)

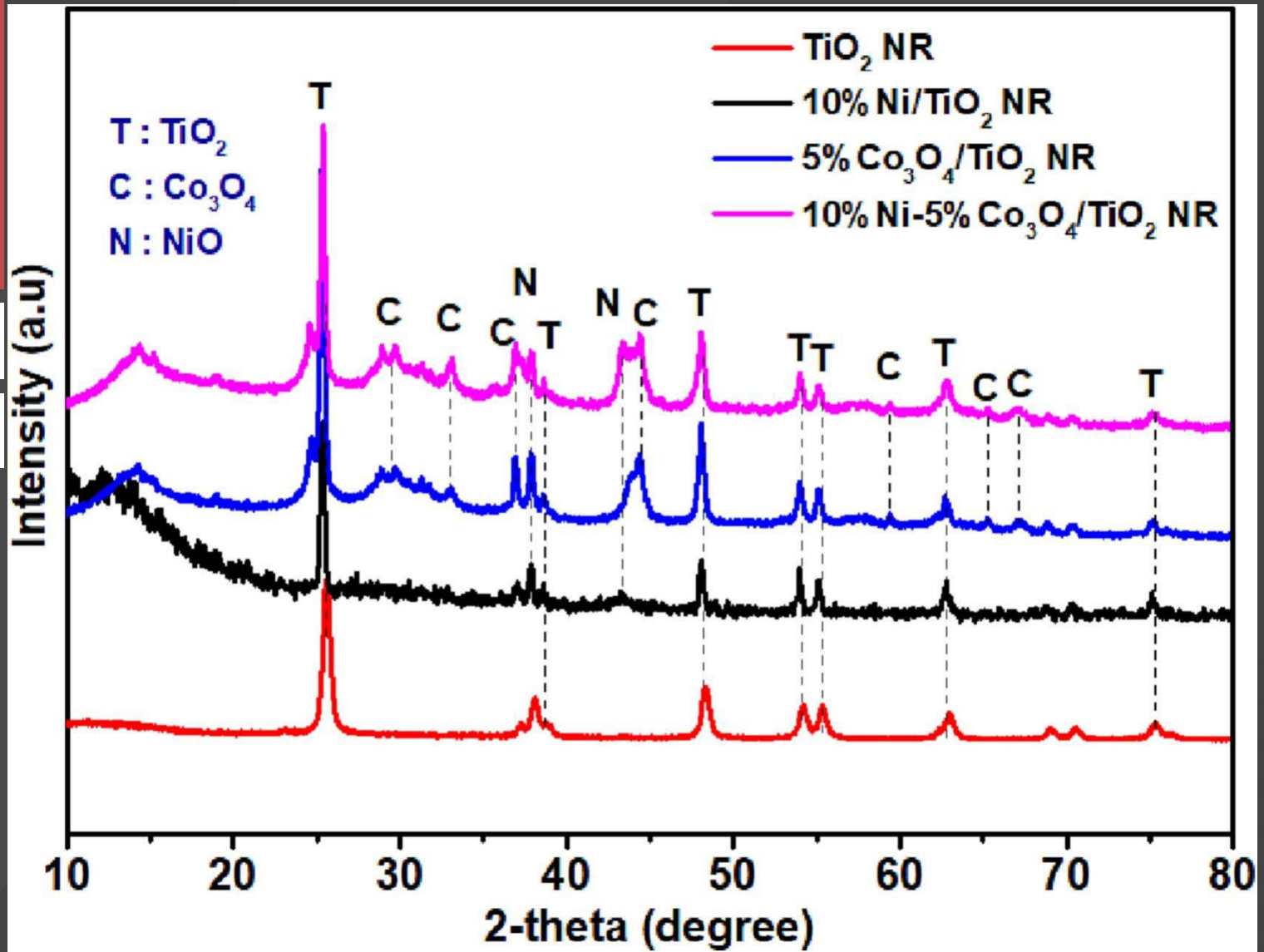
Experimental Setup



Schematics of Catalyst Preparation



Catalyst Characterization



XRD profiles of various catalysts:

- TiO_2 NR catalyst has diffraction peaks at 2θ = 25.4° (101), 38.0° (004), 48.1° (200), 53.0° (105), 55.2° (211) & 62.9° (204), corresponds to **tetragonal TiO_2** in pure **anatase** phase
- **Co_3O_4 nanocubes** detected at 2θ = 30.0° (220), 36.9° (222), 44.4° (400), 59.4° (511) & 65.4° (440)
- **Rhombohedral NiO** phase was detected for both 10%Ni/TiO_2 NR & 10%Ni-5%Co_3O_4/TiO_2 catalysts at 2θ = 37.2° (101) & 43.3° (012)

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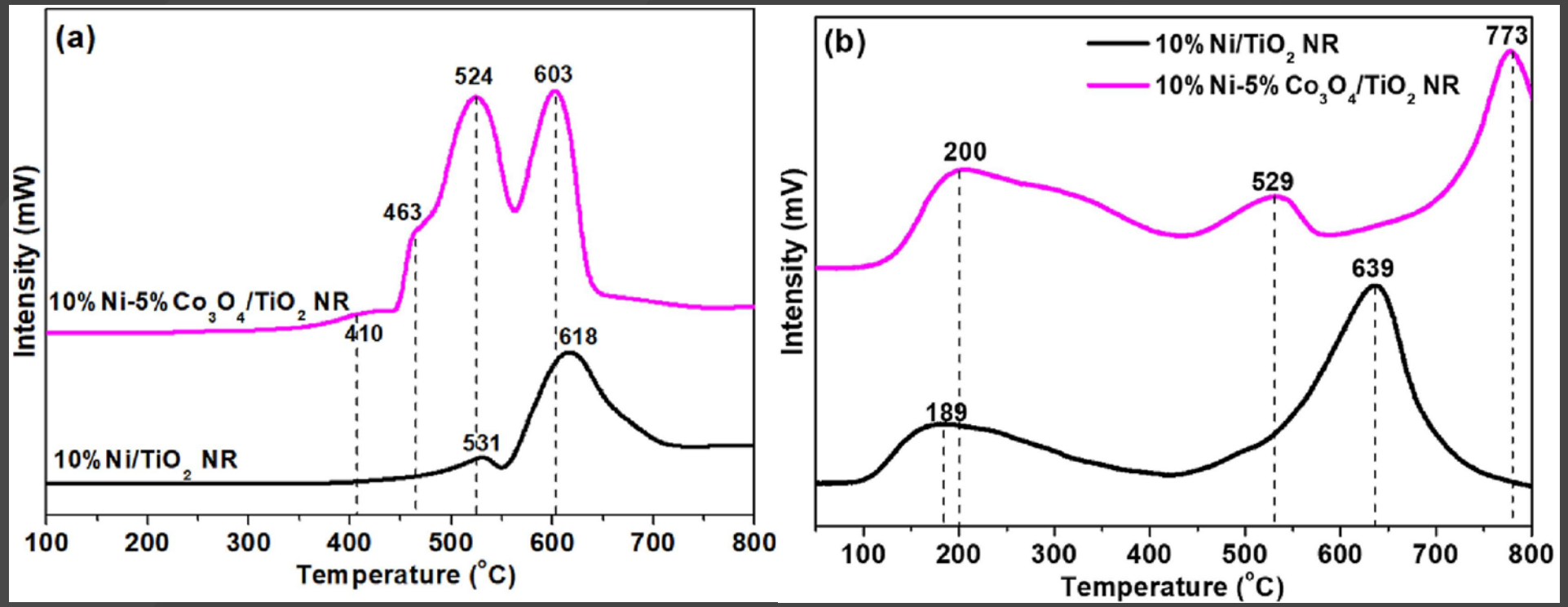
Catalyst Characterization

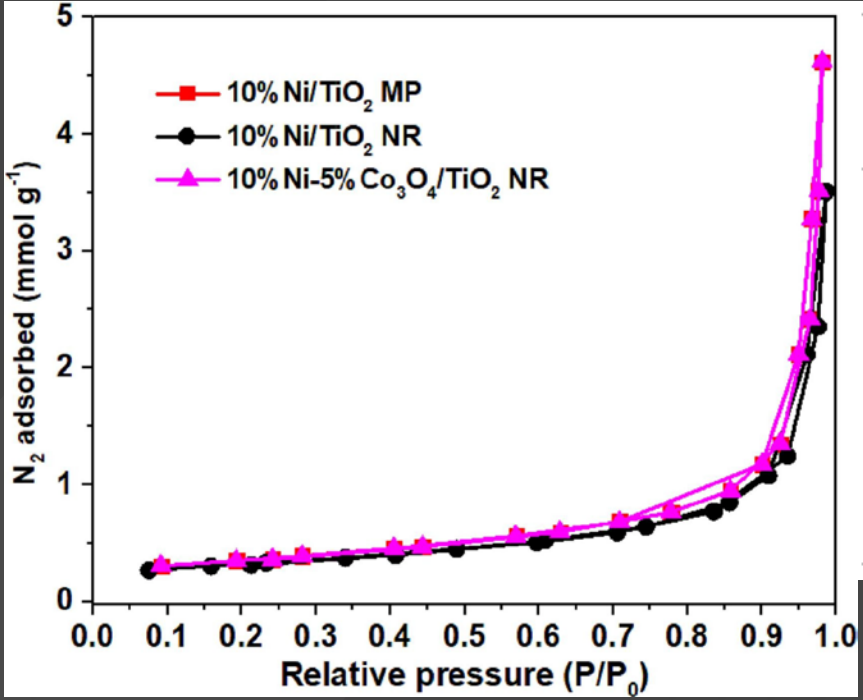


(a) H_2-TPR; and (b) CO_2-TPD profiles; for 10%Ni/TiO_2 NR and 10%Ni-5%Co_3O_4/TiO_2 NR catalysts:

- 10%Ni-5%Co_3O_4/TiO_2 NR displays higher H_2 consumption of **2.288 mmol g^{-1}** than the 10%Ni/TiO_2 NR **(1.269 mmol g^{-1})** catalyst.
- Higher basic sites of **243.8 µmol g^{-1}** are acquired for 10%Ni-5%Co_3O_4/TiO_2 NR than the 10%Ni/TiO_2 which confirms the enhanced basicity by synergistic effect of Ni-Co_3O_4 supported TiO_2 NR catalyst.

Results and discussion

Catalyst characterization



Catalysts	S_{BET} (m^2g^{-1})	V_t (cm^3g^{-1})	V_{2-50}, BJH (cm^3g^{-1})	$V_{0.5-2.0}$, t-plot (cm^3g^{-1})	$V_{0.2-0.5}$, MP (cm^3g^{-1})	APD (nm)
10%Ni/TiO_2 MP	8.3	0.079	0.074	0.004	0.002	38.0
10%Ni/TiO_2 NR	25.4	0.160	0.145	0.012	–	25.2
10%Ni-5%Co_3O_4-TiO_2 NR	23.1	0.122	0.111	0.014	0.003	21.0

BET analysis of surface properties of Ni/Co supported TiO_2 NR or MP catalysts.

N_2 adsorption-desorption isotherm plots for 10%Ni/TiO_2 NR, 10%Ni/TiO_2 MP & 10%Ni-5%Co_3O_4/TiO_2 NR catalysts:

- All catalysts exhibit **mesoporous material** with Type III and H3 hysteresis loop isotherm ascribed to the steep increase at high relative pressure (P/P_0 > 0.90) with capillary condensation steps at P/P_0 range of 0.7–0.9 indicating the presence of **mesopores**.

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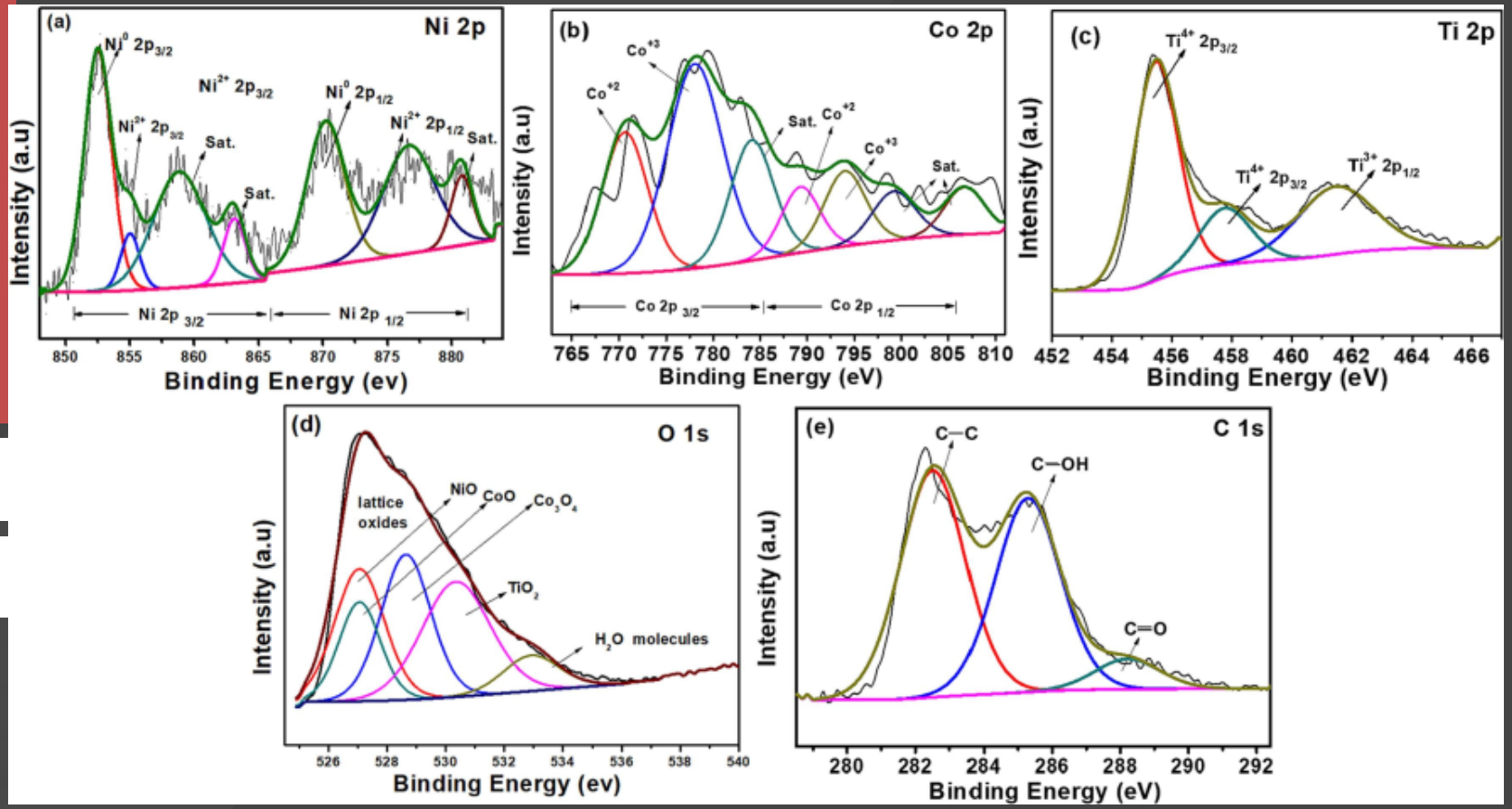
Catalyst characterization



XPS analysis of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst:

- X-ray spectrum shows 2 valence states of Ni i.e., **Ni^{2+} & Ni^0**.
- Co spectrum reveals 2 oxidation states corresponding to **Co^{2+} & Co^{3+} .**
- Ti peak confirms that the main valence state **is Ti^{4+}.**

XPS analysis of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst; High resolution spectrum of (a) Ni 2p; (b) Co 2p; (c) Ti 2p; (d) O 1 s; and (e) C 1 s.:

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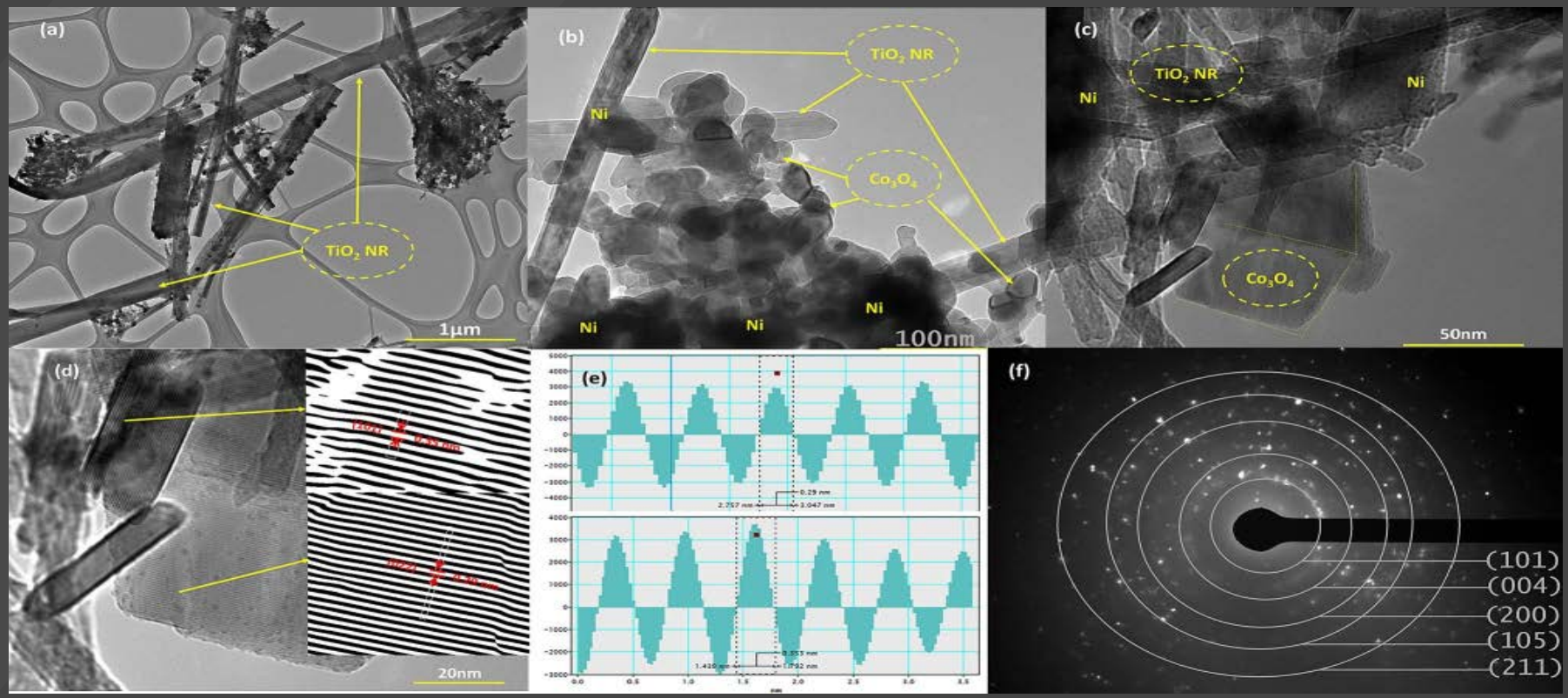
Results and discussion

Catalyst characterization



(a-e) HRTEM micrographs with different magnifications and their respective d-spacing; and (f) selected area (electron) diffraction (SAED) pattern, for the 10%Ni-5%Co_3O_4/TiO_2 NR:

- Composite catalyst exhibit NR morphology, could be ascribed to TiO_2 covered with **Ni nanoparticles** & **Co_3O_4 nanocubes**.
- SAED pattern establishes presence of **Co_3O_4 nanocubes**.

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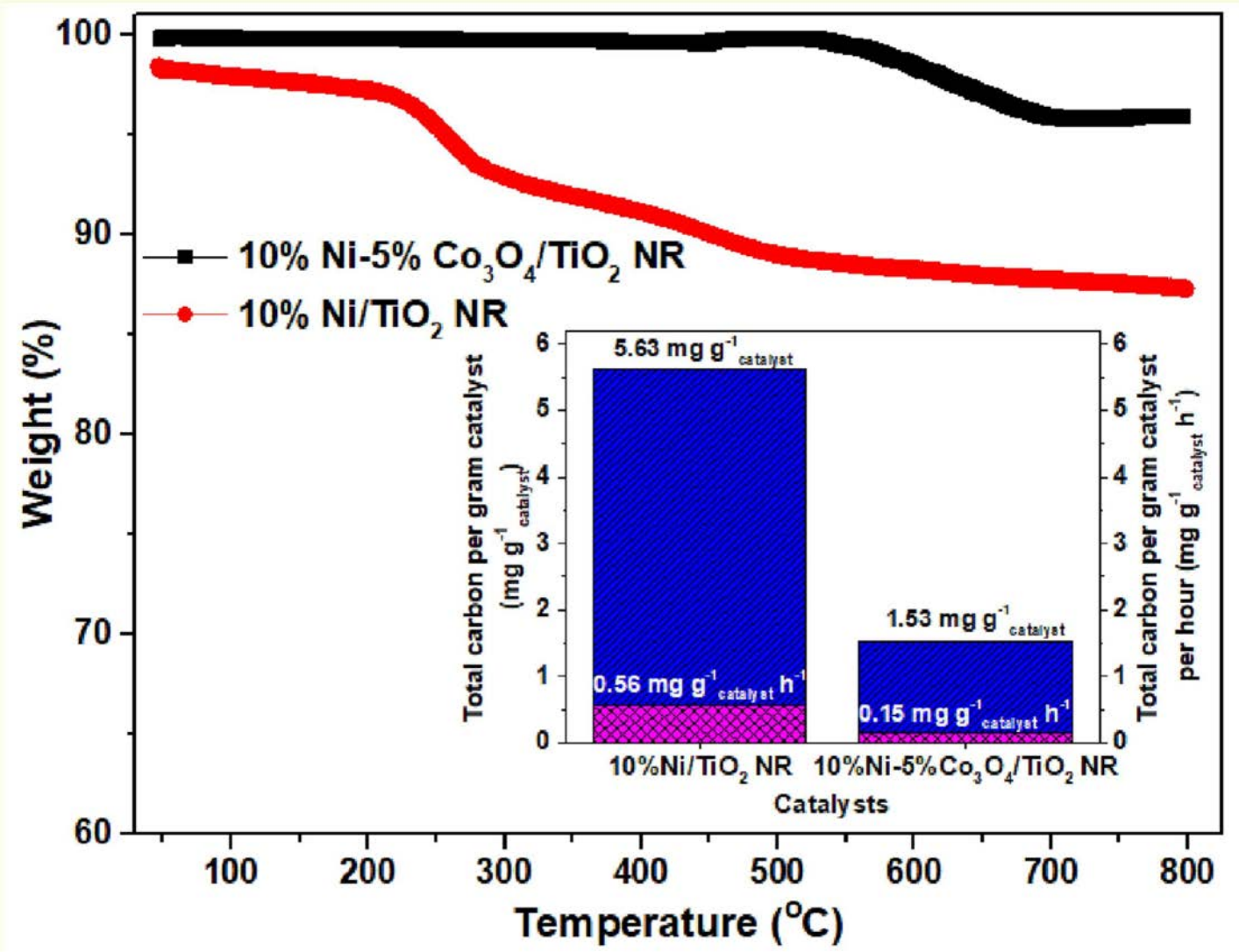
Results and discussion

Catalyst characterization



TGA results of 10%Ni/TiO_2 NR & 10%Ni-5%Co_3O_4/TiO_2 NR spent catalysts; (inset) carbon deposition after DBD plasma DRM experiment.:

- **Higher amount** of total weight loss was recorded for the spent catalyst of 10%Ni/TiO_2 NR as compared to the 10%Ni-5%Co_3O_4/TiO_2 NR.
- **Lower carbon** deposition detected for spent 10%Ni-5%Co_3O_4/TiO_2 NR with **1.53 mg g^{-1} catalyst** inferred the high resistance behavior towards carbon formation.

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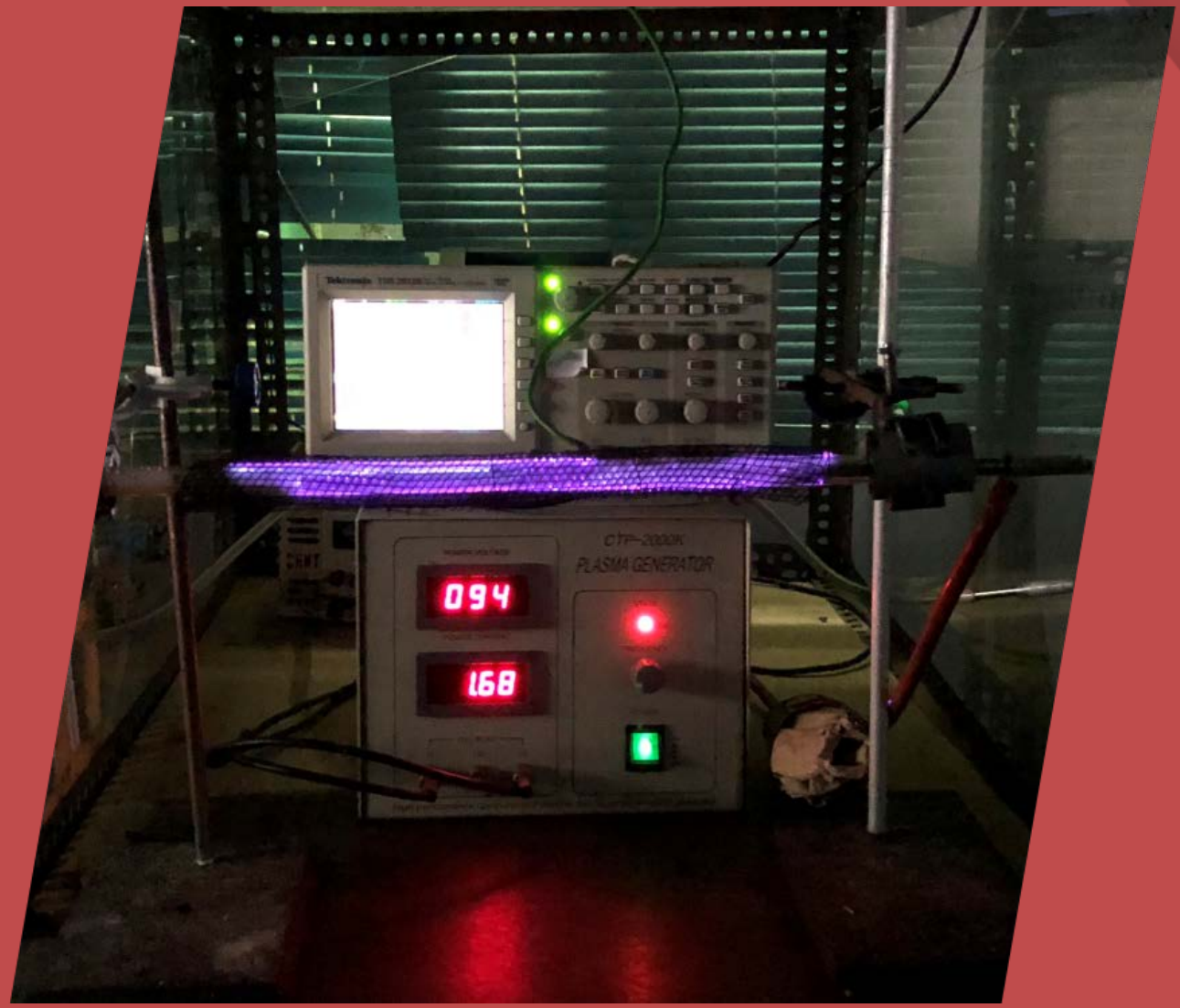
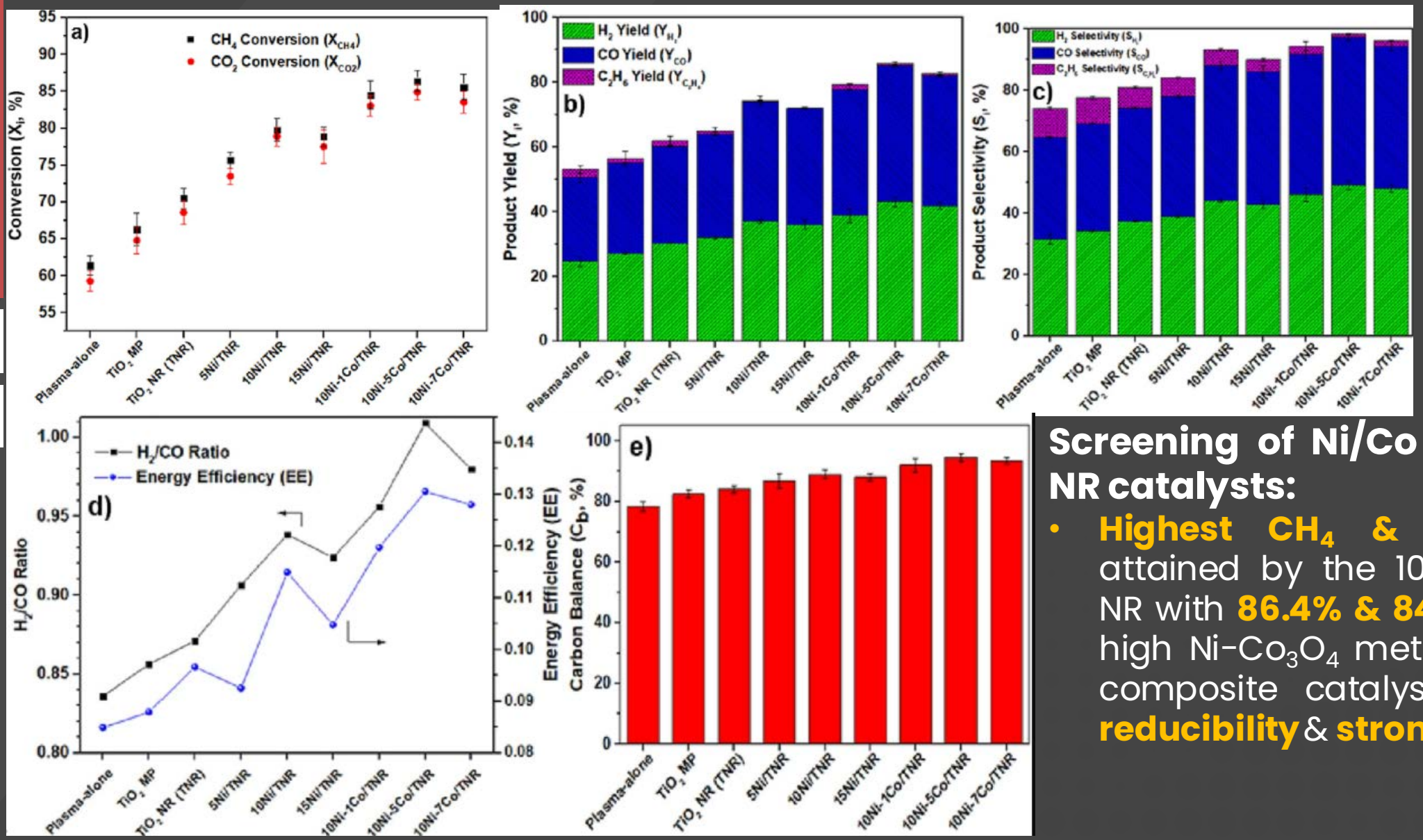
Screening of Ni/Co supported TiO_2 NR/MP catalysts



Results and discussion



Screening of Ni/Co supported TiO_2 NR catalysts:

- **Highest CH_4 & CO_2 conversion** attained by the 10%Ni-5%Co_3O_4/TiO_2 NR with **86.4% & 84.9%,** attributed to high Ni-Co_3O_4 metal dispersion over composite catalyst with **improved reducibility** & **strong basicity.**

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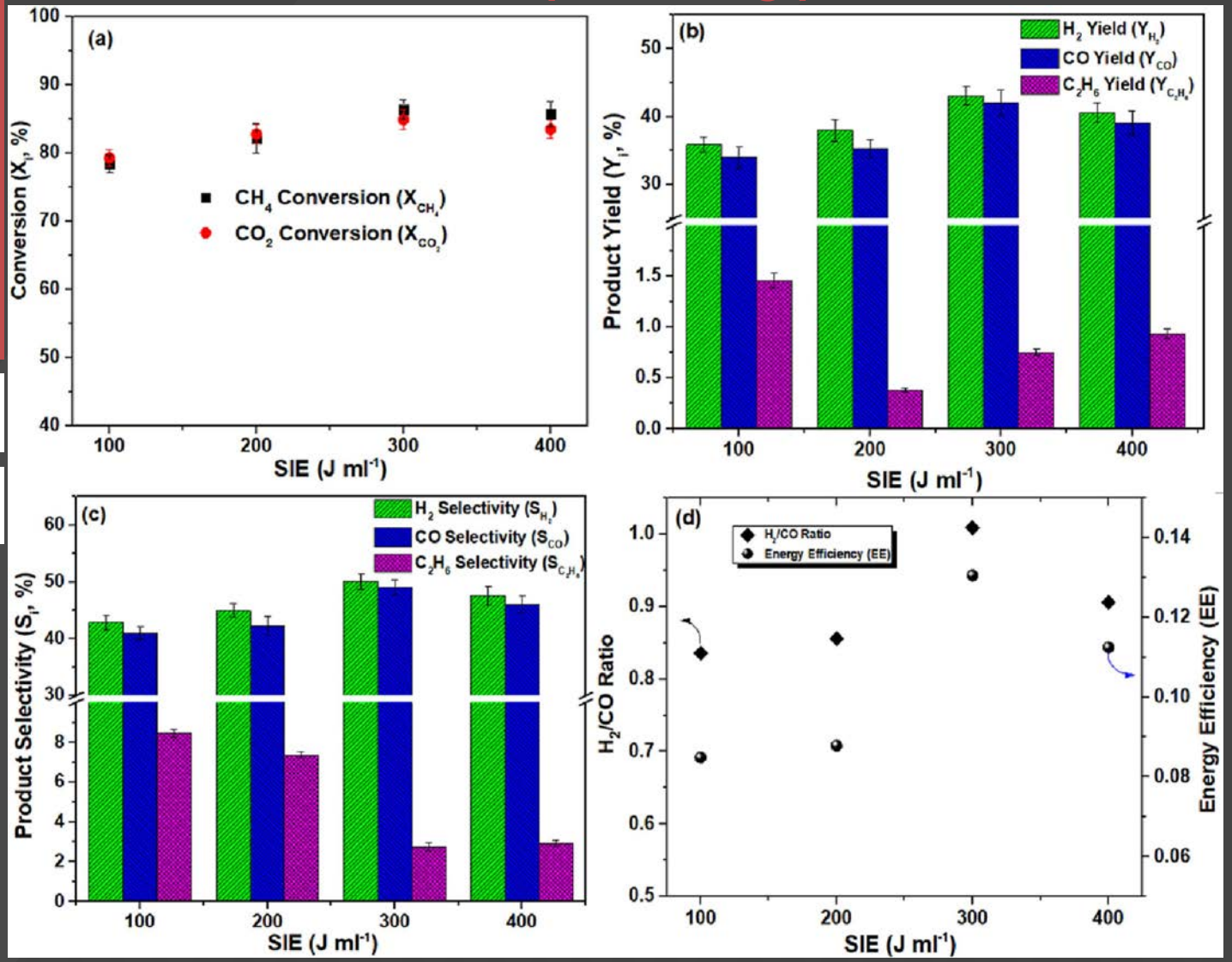
Results and discussion

Effect of different operating parameters



Effect of specific input energy:

- Increasing SIE from 100 J ml^{-1} to 300 J ml^{-1} leads to higher conversion of reactants attributed to the increment of electric field & plasma induced **energetic electron density** in the plasma discharge zone.

- X_{CH4} is relatively higher than X_{CO2} due to the **lesser energy** required for C - H bond cleavage & average threshold energy for bond partition of CH_4 at **4.5 eV** & **10 eV**, respectively, compared to C - O bond cleavage & average threshold energy for CO_2 **of 5.5 eV** & **11.9 eV**

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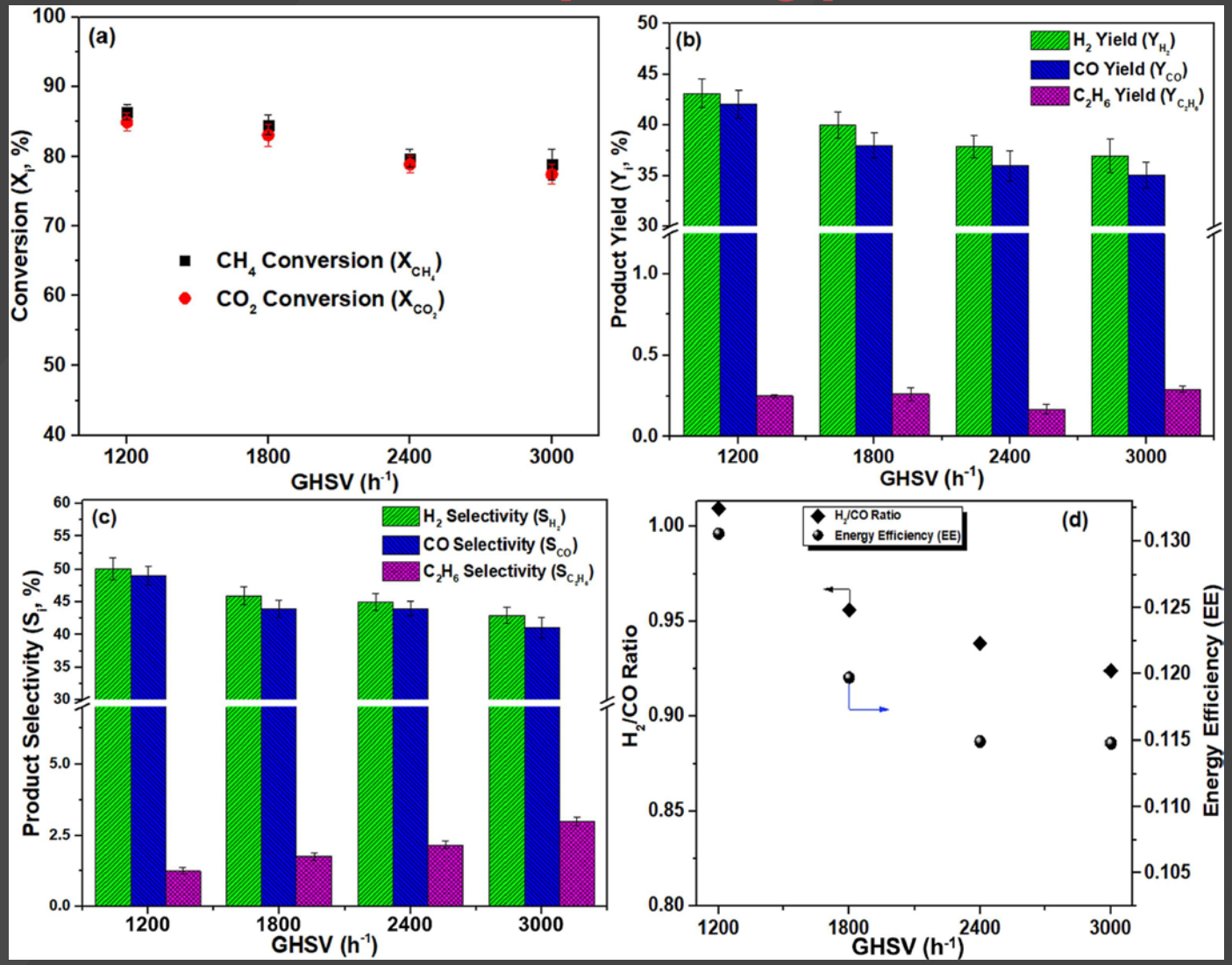
Results and discussion

Effect of different operating parameters



Effect of GHSV:

- Drop in X_{CH4} & X_{CO2} as the GHSV increases indicates the effect of the **reduced contact time** inducing lesser interaction between the active species & reactants.

- Highest X_{CH4} & X_{CO2} attained at **lower** GHSV of 1200 h^{-1} with 86.4% & 84.9%, respectively, with the **highest** H_2 & CO yield obtained at **43.1%** & **42.0%**, respectively.

- H_2/CO ratio near unity with the highest energy efficiency of **0.131 mmol kJ^{-1}** achieved at the **lower GHSV**.

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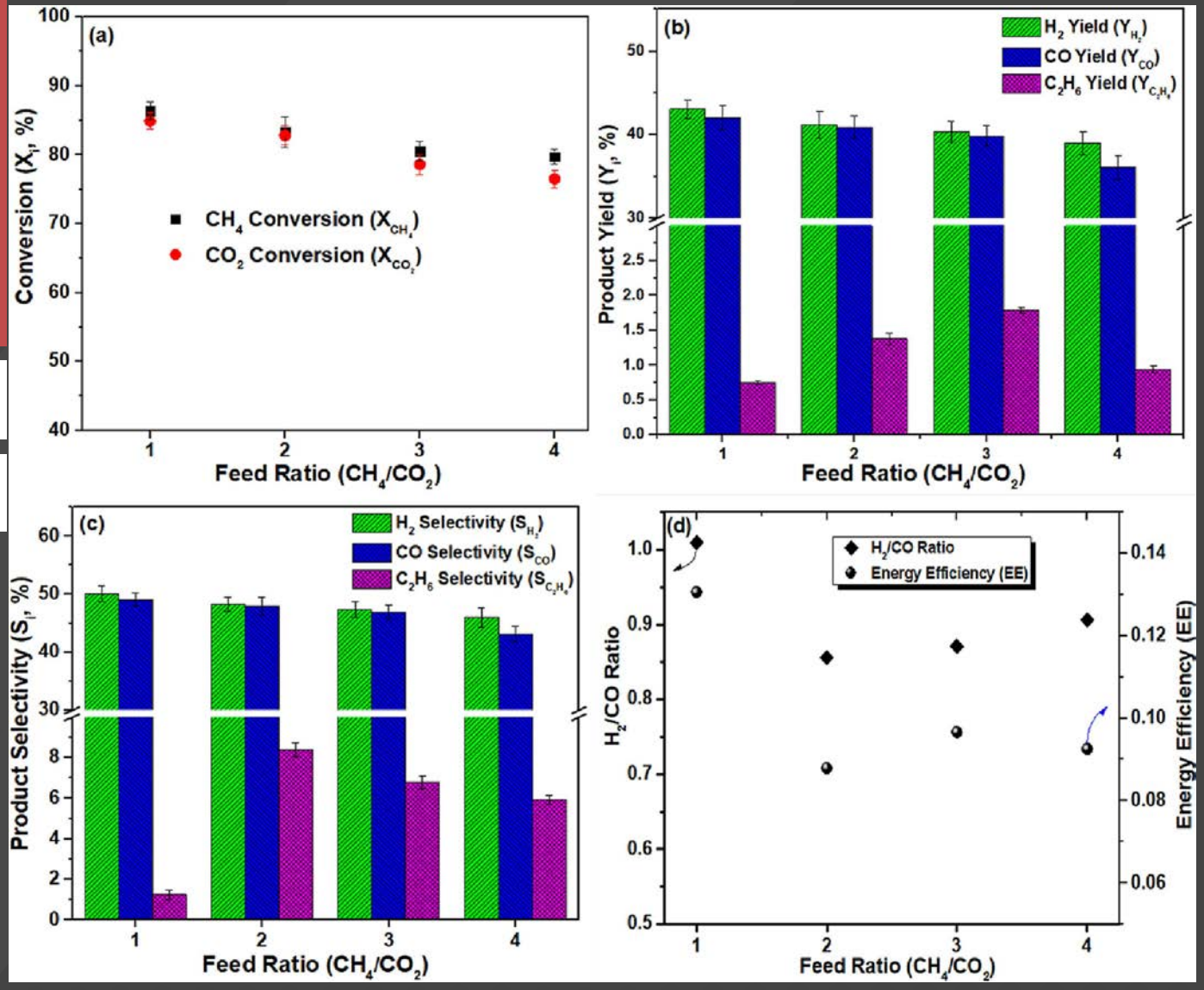
Results and discussion

Effect of different operating parameters



Effect of feed ratio:

- X_{CH4} & X_{CO2} **decrease** as the feed ratio increase.
- Highest X_{CO2} (85%) & X_{CH4} (86.4%) is recorded at a feed ratio of **one.**

- Syngas ratio of **1.01 & EE** of **0.13** mmol kJ^{-1} is observed the highest at a feed ratio of one.

- In summary the plasma DRM efficiency can be optimized by manipulating the **CH_4/CO_2 ratio** in the feed stream.

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Results and discussion

Comparison of catalytic performance

Catalysts	Loading (g)	Power (W)	Flow rate (ml min^{-1})	SIE (J ml^{-1})	X_{CO2} (%)	X_{CH4} (%)	S_{CO} (%)	S_{H2} (%)	H_2/CO ratio	EE (mmol kJ^{-1})
10%Ni-5%Co_3O_4/TiO_2 NR (This work)	0.3	100	20	300	85	86	49.	50.1	1.01	0.131
10%Ni/Al_2O_3-MgO (Khoja *et al.*, 2018)	0.5	100	20	300	73	74	48	47	0.98	0.117
Ni–Fe/SiO_2 (Zheng *et al.*, 2015a)	0.2	160	40	-	68.6	60.5	86.7	74.3	0.91	0.120
Ni-La_2O_3/SiO_2 (Zheng *et al.*, 2015b)		160	50	-	56.8	66.8	83.1	72.9	-	0.144
15%Ni/TiO_2 (Ray *et al.*, 2019)	-	24	30	200	10	20	53	44	1.08	-
15Ni/Al_2O_3 (Ray *et al.*, 2019)	-	24	30	200	11	25	39	61	0.89	-
12%Cu-Ni/Al_2O_3 (Zhang *et al.*, 2010)	0.1	60	60	60	30	56	52	31	0.6	-
26%Ni/Al_2O_3 (Tu *et al.*, 2011)	1.8	97	50	166	30.2	56.4	52.4	31	0.59	-
15Ni/ZSM (Ray *et al.*, 2019)	-	24	30	200	11	18	49	41	1.02	-

- Data not available

Comparison of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst for DBD plasma-catalytic DRM with previous literature:

- **Reactant conversion** in this study has significantly improved as compared to other catalyst packings cited from previous literature.
- Enhanced product selectivity & H_2/CO **ratio unity** in this study can also be observed as compared to previous literature.
- **EE** of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst is also recorded better & comparable with previous reports.

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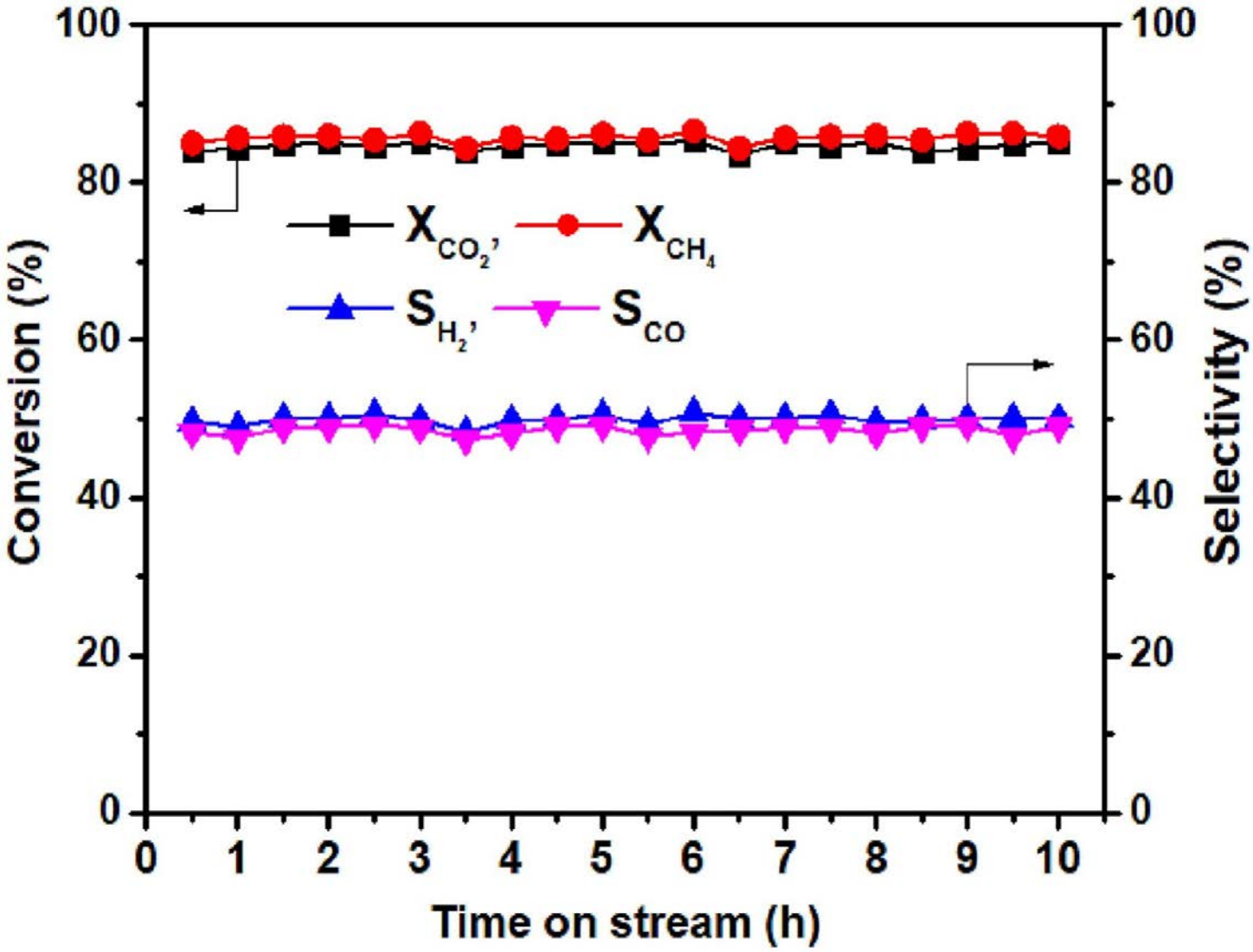
Results and discussion

Catalyst Stability Test



Stability test of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst in 10 h time-on-stream:

- High catalytic stability was observed with **85% X_{CO2} & 85.9 X_{CH4}**.
- Selectivity of syngas remained constant at **~49% CO** & **~ 50% H_2.**
- Stability experiment of 10%Ni-5%Co_3O_4/TiO_2 NR in agreement with characterization outcomes of XPS & H2-TPR for the role of **reduced active metal** & **strong metal-support interaction**.

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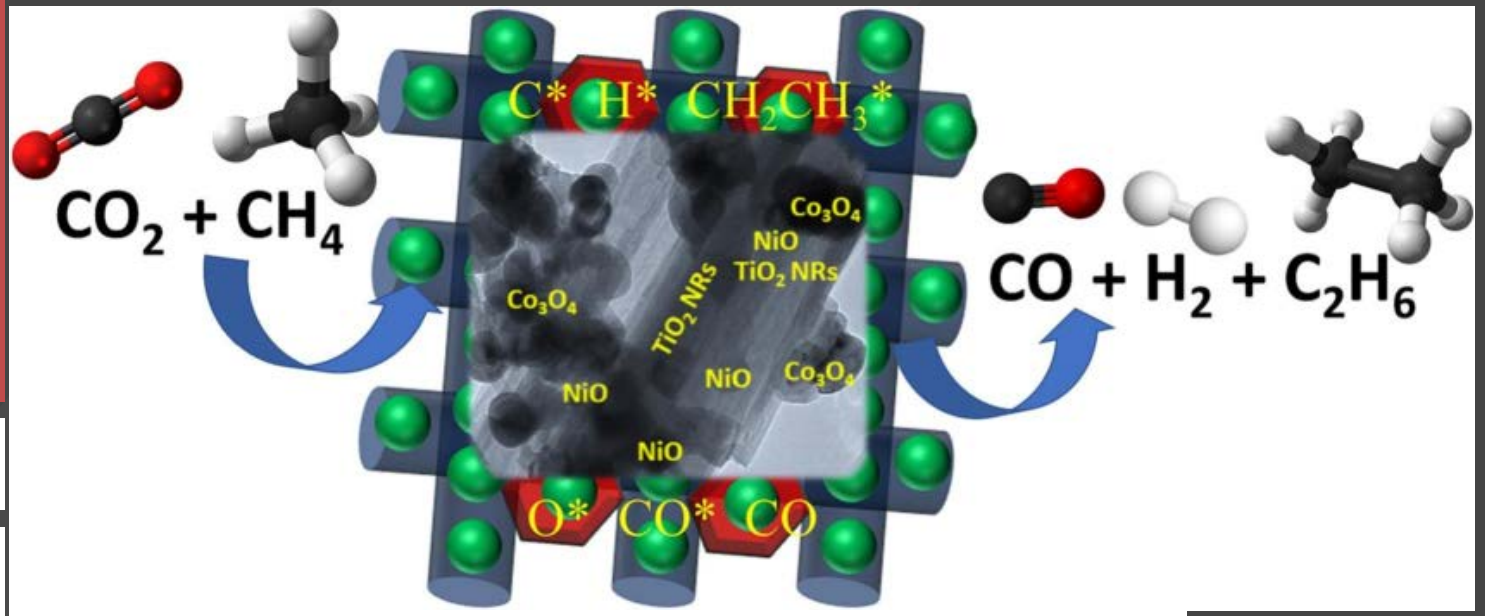
Results and discussion

Reaction mechanism



CH_4 reforming	CO_2 reforming
$e^- + CH_4 + 2S \rightarrow CH_3^*.S + H^*.S + e^-$	$e^- + CO_2 \rightarrow CO + O^* + e^-$
$e^- + CH_4^*.S \rightarrow CH_4^* + e^- + S$	$e^- + CO_2 + S \rightarrow CO_2^*.S$
$CH_4^* + S \rightarrow C^*.S + 2H_2$	$CO_2^*.S \rightarrow C^* + O_2^* + S$
$H^* + H^*.S \rightarrow H_2 + S$	$2O_2^*.S \rightarrow O^* + O_3 + S$
$CH_3^*.S + CH_3^*.S \rightarrow C_2H_6 + 2S$	$C^*.S + O^* \rightarrow CO + S$

S – Surface active sites

Possible reactions on plasma-assisted catalyst surface for DRM:

- Input energy is provided by the highly oscillating charge particles **(electrons) & radicals** produced under plasma conditions.
- The CO* and H* radicals react with excited plasma-generated electrons to produce **syngas**.
- CH_2* and CH_3* radical species react with excited electrons under the presence of plasma & catalyst to higher hydrocarbons such as **C_2H_6.**

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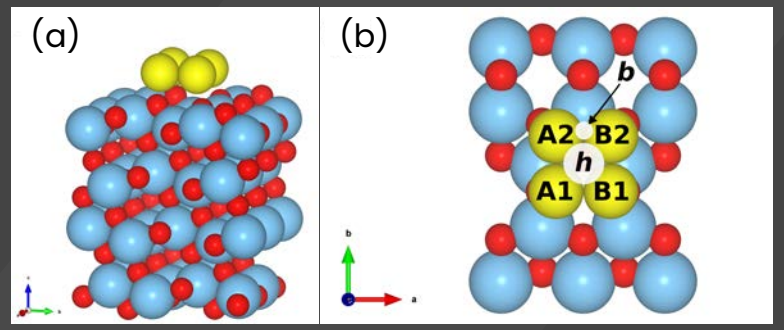
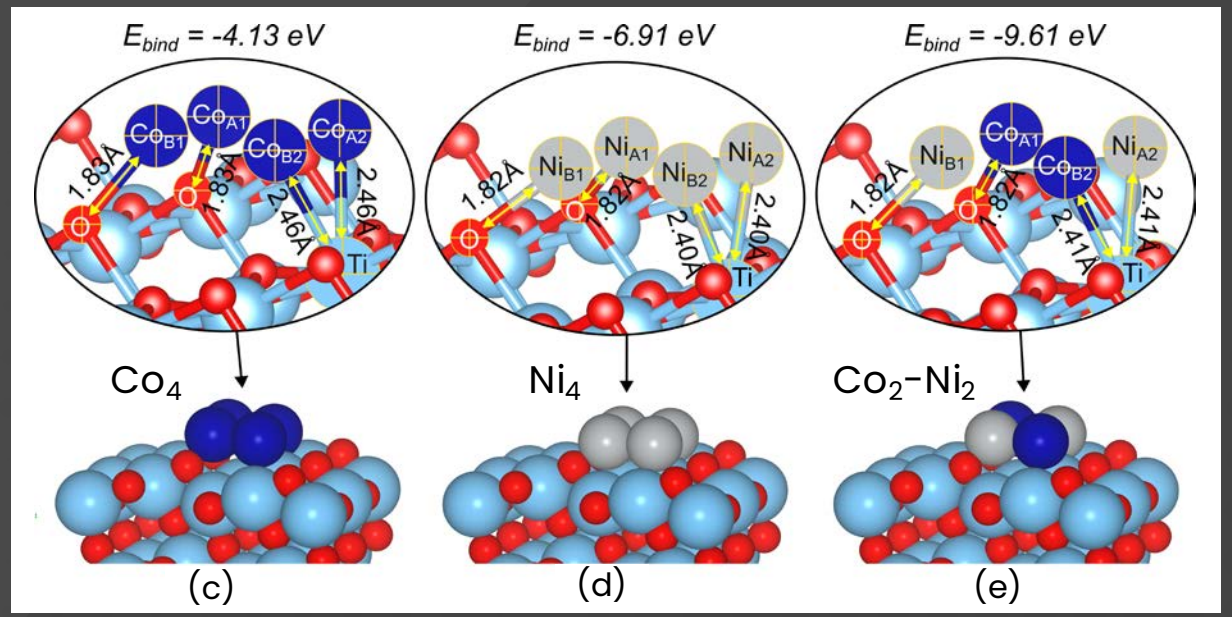
Results and discussion

H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT





Structural stability in interaction between Co_4, Ni_4, and Co_2-Ni_2 cluster on Anatase-TiO_2 surface.

- Initial configuration of metal cluster (MC) placement on a-TiO_2 surface illustrated in Figure (a) - side view and (b) – top view.
- Optimized geometries, binding energy, and bond lengths illustrated in Figure (c)-(e).
  - Stable MC configuration: A1/B1 binds to surface oxygen, A2/B2 to surface Ti. (Consistent with Prior Metal Cluster Studies on Metal Oxides).
  - MC stability: Co_2-Ni_2/a-TiO_2 exhibits highest binding energy. (E_{bind} = -9.61 eV)
  - Enhanced Co_2-Ni_2 cluster stability suggests potential for prolonged catalytic activity through Co-Ni synergy.

Ti Co Ni O

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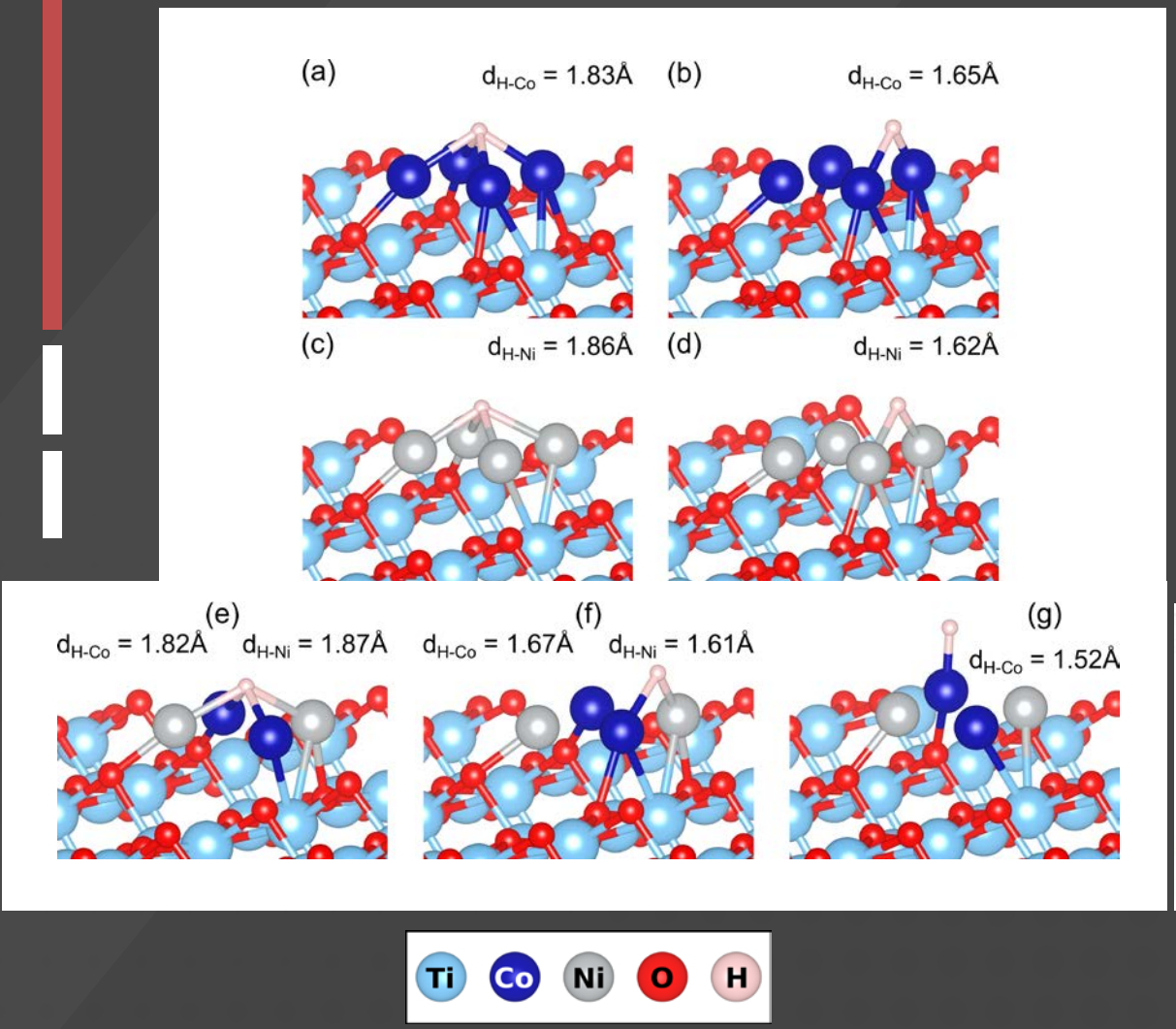
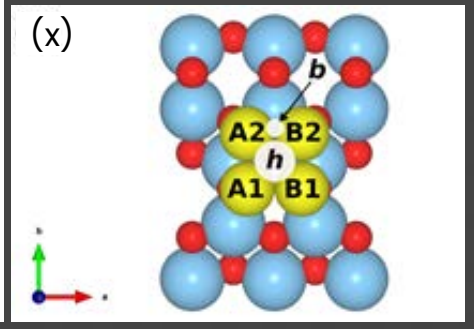


Single H atom adsorption and stability on MC/a-TiO_2

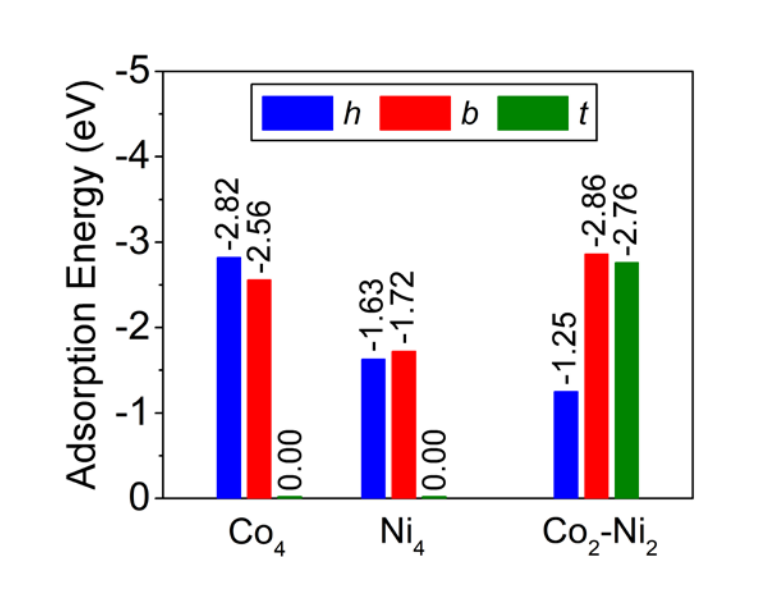
- Four H adsorption sites examined on MC/a-TiO_2 surface: top sites of A1 and A2 (*t*), hollow sites (*h*), and bridge sites (*b*) – see Figure (x).
- Most stable adsorption site illustrated in Figure (a) – (g)
- Co_4/a-TiO_2: H prefers *h* sites (Figure a) and *b sites* (Figure b).



- Ni_4/a-TiO_2: H favors *h* sites (Figure c) and *b* sites (Figure d).
- Co_2-Ni_2/a-TiO_2: H prefers on *h* sites (Figure e), *b* sites (Figure f) and *t* sites (Figure g).

Results and discussion

H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT



Single H atom adsorption and stability on MC/a-TiO_2

- Adsorption energy of H atom on Co_4, Ni_4, and Co_2-Ni_2 supported on a-TiO_2 surface at preferable active sites (*h*, *b*, or *t*) - see figure.
- H atom thermodynamically most stable on Co_2-Ni_2/a-TiO_2 at the *b* Site (-2.86 eV).
- Co_2-Ni_2/a-TiO_2 catalyst offers more active sites for H atom adsorption (*h*, *b*, and *t* sites) .

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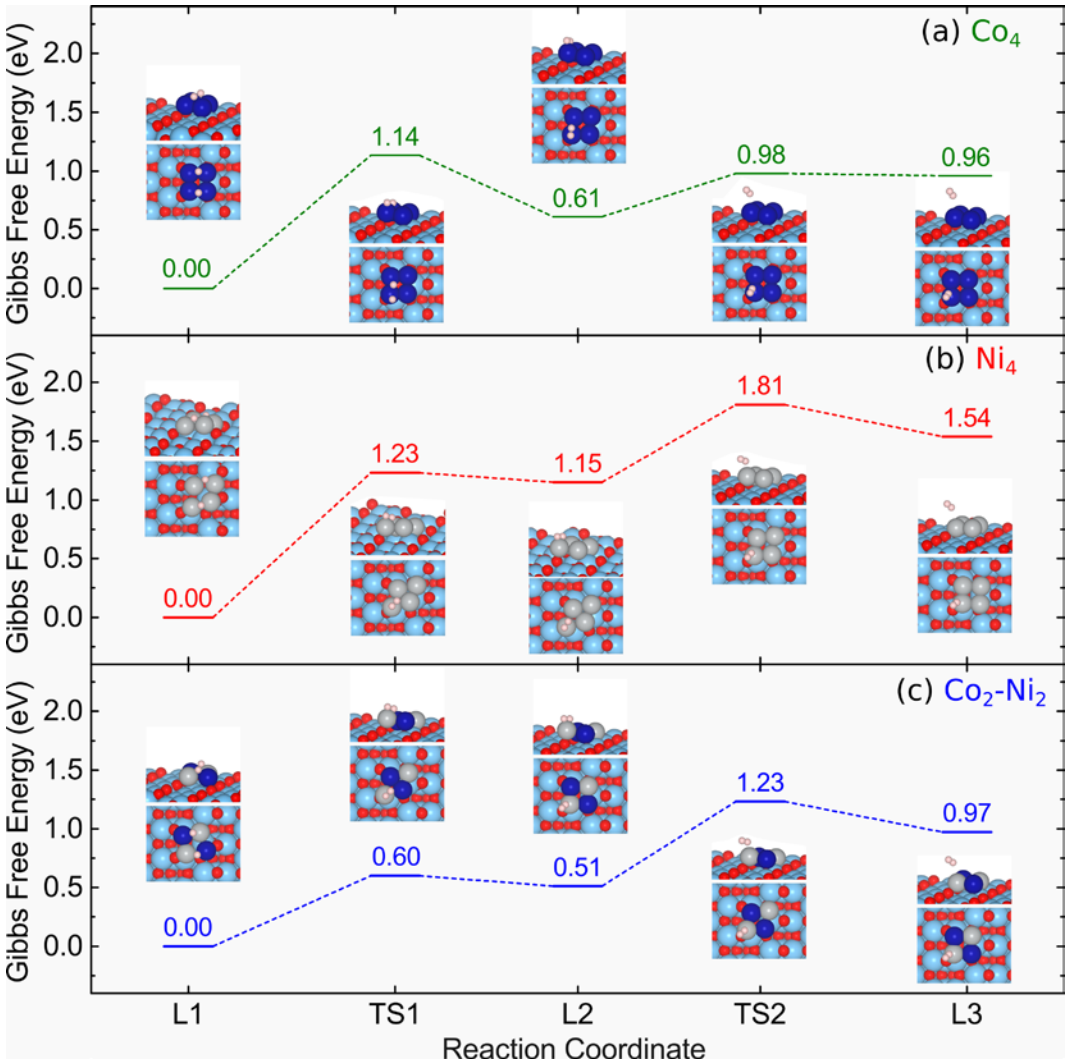
H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT



Reaction pathway for the associative desorption of H_2 on MC/a-TiO_2 catalysts :

- Proposed two-step H_2 desorption pathway reaction – see figure.
- Initial placement of two preabsorbed H atoms at adjacent bridge sites.
- 1st-step reaction (L1-L2): These H atoms diffuse and form an H_2^* radical on surface.
- 2nd-step reaction (L2-L3): These adsorbed H_2 undergoes desorption.

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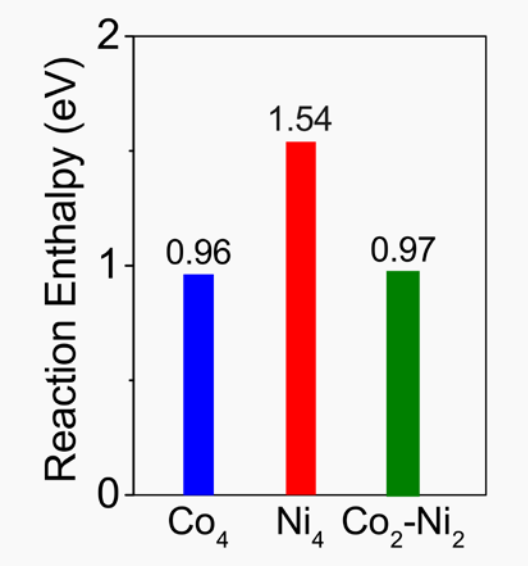
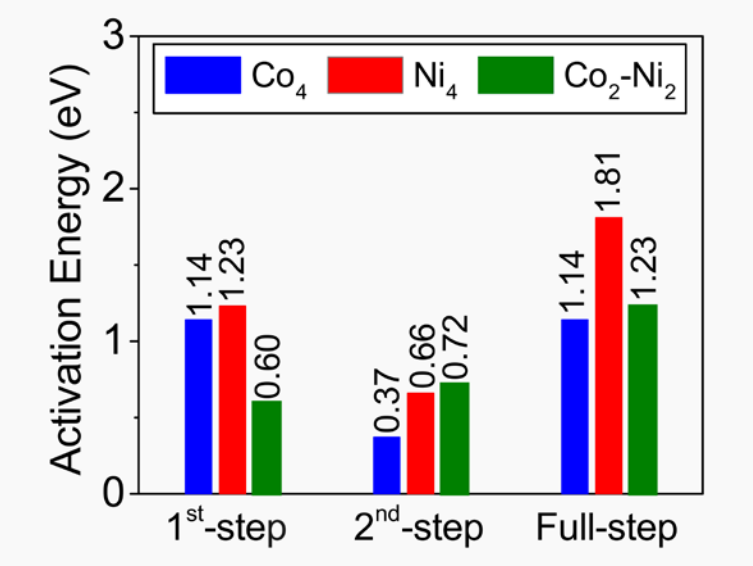
Results and discussion

H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT





Reaction pathway for the associative desorption of H_2 on MC/a-TiO_2 catalysts :

- Enthalpy of reaction is endothermic for all MC/a-TiO_2 -see figure (left).
- E_a for 1st-step, 2nd-step, overall reaction is summarized – see figure (right)
- 1st-step reaction (L1-L2): Co_2-Ni_2/a-TiO_2 offering lowest E_a(0.60 eV).
- 2nd-step reaction (L2-L3): Co_2-Ni_2/a-TiO_2 exhibits highest E_a (0.72 eV)
- Co_4/a-TiO_2 has the lowest overall E_a for associative H_2 desorption. (E_a =1.14 eV)

Results and discussion

H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT

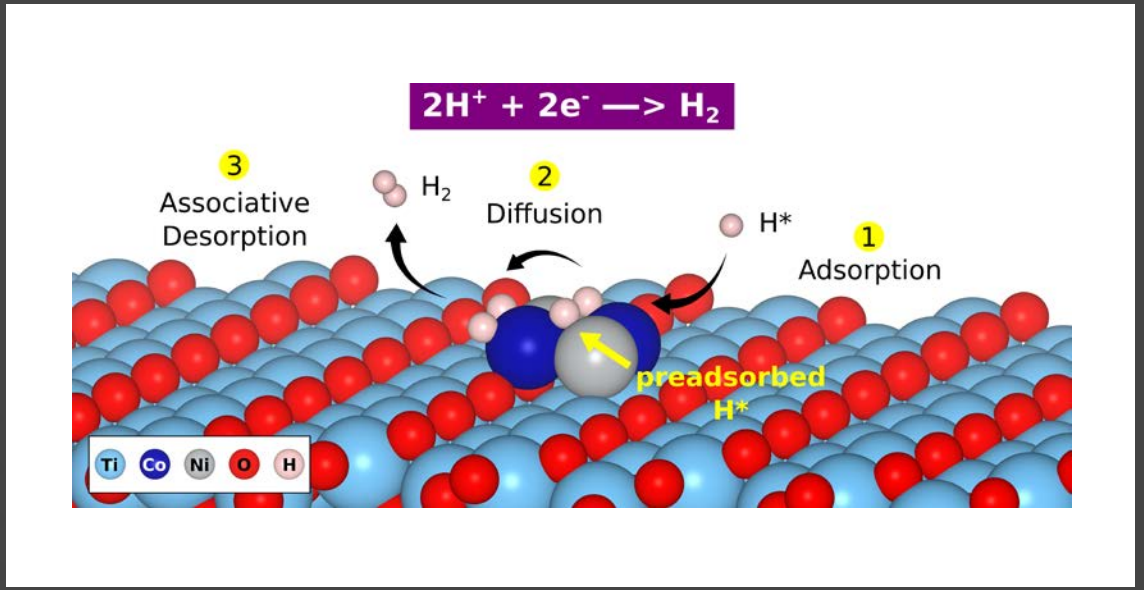
Reaction pathway for the associative desorption of H_2 on MC/a-TiO_2 catalysts :

- However, Co_2-Ni_2/a-TiO_2 significantly enhances H_2 production activity:
  1) This enhancement is attributed to a minor 0.09 eV difference in E_a between Co_2-Ni_2/a-TiO_2 and Co_2/a-TiO_2.
  2) Supported by improved stability between Co_2-Ni_2 cluster and a-TiO_2 surface, prolonging catalytic activity.
- Co-Ni clusters synergistically enhance and extend catalytic activity in H_2 production

Results and discussion

H Adsorption & Desorption on (Ni_4, Co_4, Co_2-Ni_2) Supported on the Anatase-TiO_2 (101) Surface: DFT

Proposed reaction mechanism of adsorption and associative desorption of H_2 on a Co_2-Ni_2/a-TiO_2 surface.



Conclusions

- Ni-Co_3O_4/TiO_2 NR catalysts are successfully synthesized & investigated in the packed bed DBD non-thermal plasma-catalytic DRM reaction for syngas production.
- Catalytic screening for different metal loading confirms superior performance of 10%Ni-5%Co_3O_4/TiO_2 NR catalyst with the highest CH_4 & CO_2 conversion of 86.4% & 84.9%, H_2/CO ratio unity, highest EE of 0.131 mmol kJ^{-1} & 94.5% carbon balance.
- The enhancement in activity is ascribed to successful impregnation of NiO nanoparticles and Co_3O_4 nanocubes on TiO_2 nanorods with homogeneous dispersion for the 10%Ni-5%Co_3O4/TiO_2 NR composite catalyst as evident in XRD and HRTEM.
- High reducibility, excellent basicity and strong metal-support interaction promote progressive adsorption of CO_2 and other plasma-generating energetic species and boost CO_2 conversion in DRM reaction.
- The optimum reaction condition has been found with the highest conversion and product yield at SIE 300 J ml^{-1}, GHSV 1200 h^{-1} and CH_4/CO_2 feed ratio of 1.
- DFT analysis shows that the introduction of Co & Ni supported on the a-TiO_2 catalyst have a positive impact on hydrogen production activity & migration on the surface.

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