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Urea Oxidation Reaction as an Alternative Anodic Pathway for Energy-Efficient Hydrogen Generation

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ABSTRACT

Water splitting is a key route to green hydrogen but is limited by the sluggish oxygen evolution reaction (OER), which requires high overpotentials and leads to substantial energy losses. Replacing OER with more thermodynamically favorable anodic reactions can lower the cell voltage while valorizing waste streams. Among these, the urea oxidation reaction (UOR) is especially attractive because urea is abundant in fertilizers, industrial effluents, and human/animal urine, and its electrooxidation is much easier than OER: the equilibrium potential of UOR coupled with cathodic hydrogen evolution is ~0.37 V vs RHE (and as low as ~0.07 V based on recent thermodynamic analyses), compared to 1.23 V for OER. Over the past decade, significant progress has been made in elucidating UOR mechanisms and developing high-performance electrocatalysts particularly Ni-based (oxy)hydroxides, layered double hydroxides, chalcogenides, phosphides, and phosphate-based materials, often supported on carbon, MXenes, or other conductive scaffolds which can drive UOR at technologically relevant current densities with markedly reduced cell voltages while simultaneously removing urea from wastewater or urine. This review examines UOR as a viable anodic process for hydrogen production, covering thermodynamic and mechanistic fundamentals, design principles and major catalyst families (with emphasis on structure activity relationships), electrolyzer architectures that couple UOR with hydrogen evolution (alkaline urea electrolysis, direct urine electrolysis, and solar-biased systems), and recent operando and theoretical insights into active sites and reaction pathways. Finally, techno-economic and environmental aspects are assessed, remaining challenges including catalyst stability, selectivity, poisoning, and carbonate scaling are identified, and future directions toward practical urea-assisted hydrogen technologies are proposed.

Introduction

The transition to a low-carbon energy system has placed hydrogen at the center of many national decarbonization strategies, particularly as a clean fuel and feedstock for ammonia, steel, and chemical production. Conventional hydrogen production is still dominated by steam methane reforming and coal gasification, which are energy intensive and CO_2-intensive. In contrast, water electrolysis powered by renewable electricity offers a pathway to "green" hydrogen with near-zero direct emissions [1].

However, the overall efficiency of water electrolysis is limited by the anode. The OER is a complex four-electron, four-proton process involving multiple oxygenated intermediates and strong O-O bond formation, which leads to large kinetic barriers and high over potentials even on state-of-the-art Ir- or Ru-based catalysts. In practical alkaline or acidic electrolyzes, cell voltages of 1.8-2.0 V are typically required at 200-500 mA cm^{-2}, far exceeding the thermodynamic minimum of 1.23 V at 25 $^{\circ}$C [1,2]. These energy losses translate directly into higher electricity consumption and operating costs.

To overcome this bottleneck, there has been growing interest in replacing OER with thermodynamically more favorable anodic organic oxidation reactions that can, in principle, lower the anode potential while co-producing valuable chemicals or remediating pollutants. Representative

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examples include the oxidation of small organics (glycerol, ethanol, ethylene glycol), hydrazine oxidation (HzOR), and the urea oxidation reaction (UOR) [3]. Among these, UOR is particularly appealing because urea is cheap, widely available, and present at high concentrations in industrial and municipal waste streams, making it both a pollutant and a potential resource.

The overall UOR in alkaline media can be expressed as:

$$CO(NH_2)_2 + 6OH^- \rightarrow N_2 + CO_2 + 5H_2O + 6e^- \quad \text{(Eq. 1)}$$

with CO_2 rapidly converted to carbonate/bicarbonate under alkaline conditions. When coupled with cathodic hydrogen evolution, urea electrolysis has long been cited as having a theoretical equilibrium cell voltage of ~0.37 V, much smaller than 1.23 V for conventional water splitting [4,5]. Recent thermodynamic work by Protsenko revisited this value and argued that, for the typical reaction pathway considered in urea-assisted hydrogen production, the open-circuit voltage is closer to 0.07 V, further strengthening the case for UOR-assisted hydrogen generation from an energy perspective [6,7]. Regardless of the exact value, there is broad consensus that UOR is significantly less energy-demanding than OER and thus can reduce the required cell voltage and electricity consumption.

Beyond energy savings, UOR-based systems offer two additional advantages. First, they enable the simultaneous removal of urea from wastewater and urine, mitigating eutrophication and nitrogen pollution. Second, urea is a relatively safe, non-toxic, and non-volatile compound that can act as a hydrogen carrier, as emphasized in early perspective work on “urea as a hydrogen carrier” [8]. These attributes make urea-assisted electrolysis attractive for decentralized hydrogen production, integrated wastewater treatment, and even closed-loop sanitary systems. Several comprehensive reviews have appeared recently on urea electro catalysis and catalytic urea oxidation for energy and environmental applications [9-11]. Compared with these broader treatments, the present article narrows the focus explicitly to UOR as an anodic process for energy-efficient hydrogen generation. We emphasize (i) how thermodynamics and reaction pathways translate into potential energy savings at the device level, (ii) how electro catalyst structure and composition influence UOR activity and stability at practical current densities, and (iii) what challenges must be overcome to realize industrially relevant urea-assisted hydrogen systems.

In modern electrochemical research, experimental characterization alone is often insufficient for resolving the complex interplay between surface chemistry, reaction energetics, mass transport, and degradation pathways. As a result, computational and simulation-based methodologies have become indispensable tools for deepening mechanistic insight and guiding rational materials design. Depending on the system under investigation, approaches such as density functional theory (DFT), molecular dynamics (MD), Monte Carlo simulations, and finite-element modeling (FEM), and polymer-physics simulations enable quantitative evaluation of adsorption energetics, charge distribution, molecular interactions, inhibitor–surface affinity, ion transport, and current distribution. Recent studies across diverse corrosion-, coating-, polymer-, and protection-related engineering fields have demonstrated how the integration of simulation with experimental work yields more accurate predictions, clarifies rate-determining steps, and enhances structure–property understanding [12-20]. The collective message from these works is clear: high-fidelity simulation frameworks are essential complements to experiment, particularly in electrochemical systems where multiple coupled phenomena govern performance. A simplified schematic of a two-electrode urea assisted electrolyzer, illustrating the UORHER coupling and the resulting low-voltage hydrogen production pathway, is presented in Figure 1.

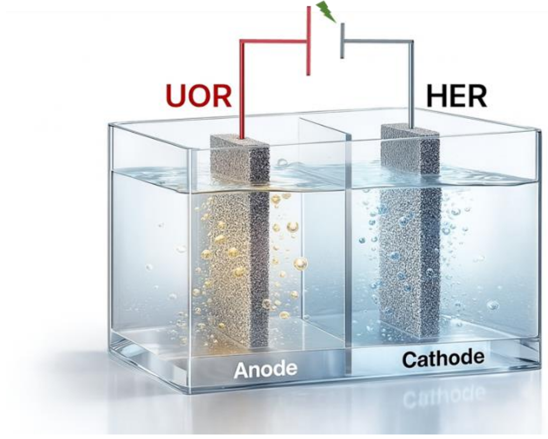

Figure 1. Schematic representation of a two-electrode electrolyzer coupling the urea oxidation reaction (UOR) at the anode with the hydrogen evolution reaction (HER) at the cathode. The assisted pathway lowers the required cell voltage and enables energy-efficient hydrogen production in alkaline media

Fundamentals of the Urea Oxidation Reaction

Thermodynamic landscape and comparison with OER: The thermodynamic driving force for UOR can be assessed by combining standard Gibbs free energy data for urea, nitrogen, carbonate, and water under alkaline conditions. Classical analyses, treating the overall urea-assisted hydrogen production reaction

$$CO(NH_2)_2 + H_2O \rightarrow N_2 + CO_2 + 3H_2 \quad \text{(Eq. 2)}$$

yield a theoretical cell voltage of about 0.37 V at 298 K, corresponding to a standard potential of ~0.37 V vs. RHE for UOR when paired with HER [1,5]. This figure has been widely used to motivate urea-assisted water electrolysis as a low-voltage alternative to OER-based water splitting.

However, a more detailed thermodynamic re-evaluation by Protsenko showed that the commonly quoted 0.37 V value arises from inconsistent reaction stoichiometries and that, when the appropriate half-reactions and activities are used, the true open-circuit voltage of the relevant urea oxidation pathway is closer to 0.07 V [6,7]. While this correction does not change the qualitative conclusion UOR is far more favorable than OER it suggests that there is theoretically even more room for reducing the cell voltage than previously recognized.

In practical alkaline electrolytes (e.g., 0.33-1.0 m urea in 1.0 m KOH or NaOH), kinetic limitations, mass transport, and ohmic losses mean that measurable UOR currents are typically obtained only at potentials ≥1.3-1.4 V vs. RHE on conventional Ni-based catalysts, and industrially relevant hydrogen production rates usually require cell voltages in the range of 1.35-1.6 V rather than the theoretical limit [1,9]. Nevertheless, this still represents a significant reduction compared with OER-based water electrolysis at the same current density.

Thermodynamic and green-metrics analyses indicate that urea oxidation pathways leading to N_2 and carbonate with minimal side products exhibit particularly favorable "electricity economy" defined as the minimum electrical work required per mole of desired product. Protsenko's green chemistry evaluation showed that the oxidation of urea to gaseous N_2 and carbonate ions has the most advantageous combination of low electricity consumption and acceptable atom economy among several possible urea oxidation routes [7]. These considerations strongly support targeting highly selective UOR pathways to maximize energy and environmental benefits.

Mechanistic pathways and intermediates: Despite its thermodynamic favorability, UOR is mechanistically complex. It involves six-electron transfer, multiple dehydrogenation steps on the urea molecule, C-N bond cleavage, N-N coupling, and C-O bond formation, occurring on dynamically reconstructed catalyst surfaces that often transform into high-valent (oxy)hydroxide phases under operating conditions [10,21]. Several mechanistic frameworks have been proposed to rationalize UOR kinetics:

- ✓ **Adsorbate evolution mechanism (AEM):** In analogy to OER, UOR is described in terms of sequential adsorption and dehydrogenation of urea-derived intermediates ($CO(NH_2)_2$*, CONH*, CO*, NH_x*) on active metal sites, coupled with OH^- adsorption and electron transfer. Rate-determining steps often involve C-N bond cleavage or the dehydrogenation of adsorbed amide species.
- ✓ **Lattice oxygen-involved mechanisms (LOM):** For certain oxides and ox hydroxides, lattice oxygen may actively participate in forming C-O bonds or in oxidizing nitrogen intermediates, with concomitant formation of oxygen vacancies and re-oxidation by OH^- from the electrolyte.
- ✓ **Chemical electrochemical (CE) pathways:** Some studies suggest that the initial dehydrogenation of urea can proceed via chemical steps on higher-valent NiOOH or CoOOH surfaces, followed by electrochemical regeneration of the oxidized surface states, akin to "redox mediator" behavior.

In situ/operando Raman, FTIR, X-ray absorption spectroscopy (XAS), and differential electrochemical mass spectrometry (DEMS) have identified key intermediates such as adsorbed cyanate (CNO^-), carbamate, carbonate, and various NH_x* species [9,21]. While N_2 is often considered the desired nitrogen product, recent work has highlighted that competing oxidation paths can lead to NO_x^-/NO_3^- species, especially at high anodic potentials or on particular catalyst compositions. Highly selective UOR catalysts that favor N_2 formation over nitrate/nitrite are therefore crucial to minimize nitrogen pollution and maximize atom efficiency [9,11].

Kinetic descriptors and performance metrics: Typical electrochemical metrics used to evaluate UOR electro catalysts include:

- ✓ Onset potential and potential at fixed current densities (e.g., η at 10, 50, 100, or 500 mA cm^{-2}), often reported vs. RHE (three-electrode measurements) or as full-cell voltages in two-electrode configurations.
- ✓ Tafel slopes, reflecting the rate-determining step and overall kinetics.

- ✓ Turnover frequency (TOF) normalized by electrochemically active surface area (ECSA) or the number of active metal sites.
- ✓ Charge transfer resistance from electrochemical impedance spectroscopy (EIS).
- ✓ Long-term chronoamperometric or Chrono potentiometric stability, including tolerance to carbonate buildup and poisoning species.
- ✓ Faradaic efficiency for hydrogen at the cathode and for nitrogen-containing products at the anode.

Recent structure activity correlation studies have revealed that high UOR activity is typically associated with catalysts that can: (i) stabilize high-valent Ni^{3+}/Ni^{4+} or analogous oxidized states at relatively low potentials; (ii) exhibit optimized adsorption energies for urea and key intermediates; and (iii) provide abundant oxyphilic sites and favorable proton-coupled electron transfer pathways [21,22]. Such descriptors guide rational catalyst design.

Electro catalyst Landscape for UOR-Assisted Hydrogen Generation

General design principles: Modern UOR electro catalyst design builds on three overarching ideas:

- ✓ **Electronic structure engineering:** Tuning the d-band center, oxidation state distribution, and metal oxygen covalence to favor the formation of highly active, high-valent species (e.g., γ-NiOOH-like phases) at lower potentials while maintaining sufficient conductivity [1, 11].
- ✓ **Nano structuring and interface design:** Creating 3D porous architectures, core shell morphologies, and heterostructures that maximize active surface area, expose edge/defect sites, and facilitate reactant/product transport, especially in concentrated urea/KOH mixtures.
- ✓ **Support and hybridization strategies:** Integrating active phases with conductive scaffolds such as Ni foam, carbon fibers, doped carbons, or MXenes to reduce charge transport resistance and improve mechanical robustness.

Within this framework, Ni-based catalysts remain the workhorse for UOR due to their favorable adsorption properties for urea and OH^-, suitable redox potentials for $Ni^{2+}/Ni^{3+}/Ni^{4+}$, and moderate cost. However, a wide range of material classes has now been explored. Representative studies on self-supported Fe-Co-Ni nanostructures grown on copper foam for alkaline HER, hierarchical three-phase Ni/Co/Fe selenide networks anchored on 2D NiCo-MXene for seawater electrolysis and hydrazine oxidation, and ultrafast-synthesized NiCo-MXene nanocomposites for coupled HER and UOR in alkaline and seawater media collectively show that properly engineered multicomponent Ni/Co/Fe-based transition-metal architectures, especially when integrated with MXene supports, can deliver high activity and durability for low-voltage hydrogen production using only earth-abundant elements [23-25].

Ni-based hydroxides, ox hydroxides and LDHs: Pure $Ni(OH)_2/NiOOH$ systems on Ni foam or Ni mesh are historically the earliest and most studied UOR catalysts. Under anodic polarization in alkaline urea solutions, $Ni(OH)_2$ is transformed into γ-NiOOH, which is widely regarded as the true active phase for UOR, analogous to its role in OER [4,10]. On such electrodes, UOR onset typically occurs near the Ni^{2+}/Ni^{3+} redox potential (~1.4 V vs. RHE), with current densities of 10-50 mA cm^{-2} reached around 1.45-1.50 V vs. RHE in 0.33-0.5 m urea +1.0 m KOH.

Bimetallic LDHs (e.g., NiFe-, NiCo-, NiMn-LDH) supported on Ni foam, carbon cloth, or other conductive substrates show systematically improved UOR performance compared with simple $Ni(OH)_2$ [9,11]. In these materials, the second metal cation (Fe, Co, Mn, etc.) modulates the electronic structure of the Ni centers, introduces cation disorder, and often enhances the density of active high-valent sites. Oxygen vacancies and defect-rich LDHs further facilitate urea adsorption and OH^- activation. For example, oxygen-deficient NiAl-LDH Nano sheets on Ni foam have been reported to achieve ~10 mA cm^{-2} at ~1.42 V vs. RHE in 1 m KOH + 0.33 m urea, with small Tafel slopes indicative of favorable kinetics [26].

Recent reviews by Yang et al. and Ge et al. provide exhaustive catalogs of Ni-based LDHs and reconstructed NiOOH catalysts for UOR, outlining correlations between composition, defect chemistry, and activity trends across hundreds of compositions [10,27]. These works underline that subtle changes such as Fe doping level, Co/Ni ratio, or introduction of high-entropy multination LDHs can significantly shift UOR onset potentials and durability. To place the above trends on a more quantitative footing, several representative Ni-based UOR electro catalysts are compiled in Table 1. As seen from these data, defect-rich $Ni(OH)_2/NiOOH$ and NiFe- or NiCo-based LDHs grown on conductive three-dimensional substrates such as Ni foam or carbon cloth typically reach 10-50 mA cm^{-2} at ≈1.36-1.45 V vs. RHE in alkaline urea electrolytes, in line with the general picture drawn above. Compositional modulation (e.g., incorporation of Fe, Co, Mn or high-valent dopants such as Hf) and the use of nitride or oxide–nitride pre-catalysts further lower the operating potential and enable high current densities at relatively small over potentials. Overall, the comparative performance in Table 1 reinforces that electronic-structure engineering combined with hierarchical 3D architectures is central to optimizing Ni-based hydroxides, ox hydroxides and LDHs as practical anodes for urea-assisted hydrogen generation.

Table 1. Representative Ni-based hydroxide/ox hydroxide and LDH electro catalysts for the urea oxidation reaction.

Catalyst	Structure	Electrolyte	UOR	Notes	Ref.
NiO@ESM-C	NiO nanoparticles nested in hierarchically porous carbon	1.0 M KOH + 0.33 M urea	1.36 V vs RHE @ 10 mA cm^{-2}; 1.46 V @ 25 mA cm^{-2}	Synergistic NiO–porous carbon interface; fast charge transfer and bubble release.	[28]
NiFeO-LDH/NF	H_2O_2-treated NiFe LDH on Ni foam (NiFeO-LDH)	1.0 M KOH + 0.33 M urea (alkaline UOR)	1.37 V vs RHE @ 10 mA cm^{-2}	H_2O_2 treatment tailors surface oxygen ligands, promotes formation of highly active NiOOH.	[29]
$MnCO_3/Ni(OH)_2$@CC	$MnCO_3/Ni(OH)_2$ heterostructured Nano flower arrays on carbon cloth	1.0 M KOH + 0.33 M urea	η ≈ 90 mV @ 10 mA cm^{-2}; η ≈ 167 mV @ 200 mA cm^{-2}	Strong interfacial coupling; Mn species optimize adsorption/activation of urea.	[30]
Hf–NiFe-LDH/NF	Hf-doped NiFe LDH on Ni foam	1.0 M KOH + 0.33 M urea	1.37 V vs RHE @ 100 mA cm^{-2} (UOR), ~118 mV lower than OER at same j	Multielectronic synergetic effect of Hf dopant; facilitates Ni^{2+}/Ni^{3+} redox and NiOOH formation.	[31]
NCMO–Ar	Co-doped $NiMoO_4$ porous Nano sheets	Flow cell, alkaline KOH + urea	1.342 V @ 10 mA cm^{-2}; no attenuation at 100 mA cm^{-2} for 200 h	Co doping + oxygen vacancies in $NiMoO_4$ boost conductivity and UOR kinetics.	[32]

Nickel chalcogenides and nitrides: Nickel sulfides, selenides, and nitrides are often considered “pre-catalysts” that transform under anodic UOR conditions into Ni-(oxy)hydroxide shells on chalcogenide/nitride cores. This core–shell architecture can enhance activity by combining a highly conductive inner core with a catalytically active outer NiOOH layer.

NiS and Ni_3S_2 nanostructures on Ni foam or carbon supports, including honeycomb-like Ni-Mo-S architectures, have shown high functional activity for HER and UOR, enabling low overall cell voltages for urea electrolysis (e.g., ~1.47 V at 10 mA cm^{-2}) [33, 34]. Long-term stability, however, can be compromised by sulfur leaching and surface reconstruction.

Similarly, $NiSe_2$, Ni_3Se_2 and bimetallic CoNiSe or FeNiCoSe phases exhibit strong UOR activity, benefiting from improved conductivity and tuned electron density around Ni [11,35]. Under operating potentials, these selenides tend to develop Ni/Co ox hydroxide shells enriched in high-valent species. Incorporating urea-active selenides into hierarchical 3D architectures (e.g., Nano flowers, Nano sheets, or nanotubes) further improves mass transport and bubble removal.

Nickel nitrides (Ni_3N, Ni_2N) have also been investigated as UOR pre-catalysts; their higher intrinsic conductivity and favorable Ni-N covalence can facilitate electron transfer, while in situ oxidation produces NiOOH-like surfaces [9].

Phosphides and phosphate-based catalysts: Transition-metal phosphides (Ni_2P, Ni_5P_4, CoP, FeP, and bimetallic phosphides) represent another major class of UOR pre-catalysts. Their metallic or semi-metallic conductivity and tunable P-M covalence are advantageous for both HER and UOR, making them promising functional electrodes for overall urea electrolysis.

Ni_2P Nano flowers on Ni foam have been shown to drive UOR at low over potentials (e.g., <1.45 V vs. RHE at 10 mA cm^{-2}) while also serving as efficient HER catalysts, enabling full urea electrolyzes operating at ≲1.5 V for moderate current densities [9,11]. Recent work on cerium-doped Ni_5P_4 highlights how aliovalent doping can modulate the electronic structure, promote surface reconstruction to NiOOH-like phases, and enhance UOR kinetics at industrial current densities [11].

Complementary to phosphides, phosphate and biphosphate materials such as Ni, Co, Cu, or bimetallic Ni-Co phosphates have been explored as UOR catalysts. Their robust polynomic frameworks and strong metal-oxygen-phosphate interactions confer good structural stability and can tune the acidity/basicity of surface sites. A recent review by Abd El-Lateef and co-workers systematically summarized phosphorus-based catalysts for UOR, emphasizing their potential to couple urea electro oxidation with sustainable waste-to-energy conversion [11].

To provide a quantitative perspective on these materials, representative phosphide and phosphate-based UOR electro catalysts and their key performance metrics are compiled in Table 2.

Table 2. Summary of selected P-based UOR electro catalysts.

Catalyst	Structure	Electrolyte	UOR metrics (E vs RHE or cell voltage)	Key remarks	Ref.
NiP/CNTs/NF	Amorphous NiP deposited on CNT-decorated Ni foam	1.0 M KOH + urea (alkaline)	j ≈ 400 mA cm^{-2} at high potential (exact E not stated in summary)	High j with fast kinetics; high apparent rate constant (1.1×10^{6} cm^{3} mol^{-1} s^{-1}) | In situ reconstruction to $Ni(OH)_2$/NiOOH active phase during UOR; dual N,P-doping in CNTs tailors electron density.	[36]
c-$CoNiP_x$/a-P-MnO_y composite	Hierarchical Co–Ni phosphide + amorphous P-MnO_y	1.0 M KOH + urea	1.24 V @ 10 mA cm^{-2}; 1.35 V @ 100 mA cm^{-2}	Co incorporation accelerates Ni^{2+}/Ni^{3+} oxidation to NiOOH; P-MnO_y shell enhances adsorption and stability.	[37]
Co-MnP/MnO@NiP/NF	MnP/MnO@NiP core–shell on Ni foam	1.0 M KOH+ urea	1.24 V @ 10 mA cm^{-2} (UOR); full urea electrolysis: 1.52 V cell @ 10 mA cm^{-2}	Protective MnO_x layer and multi-component interfaces improve charge transfer and corrosion resistance.	[38]
NiP@PNC/NF	NiP shell on P,N co-doped CNT network on Ni foam	1.0 M KOH + 0.33 M urea	1.34 V @ 50 mA cm^{-2}; 1.43 V @ 100 mA cm^{-2} (vs RHE)	Hierarchical NiP–PNC architecture gives large ECSA and high C_{dl}; strong NiP–CNT interaction stabilizes active sites.	[39]
Ni_2P@Ni-MOF/NF	Ni_2P nanocrystals embedded in Ni-MOF Nano sheets on Ni foam	1.0 M NaOH + 0.33 M urea	1.41 V vs RHE @ 100 mA cm^{-2} (UOR, 3-electrode)	MOF-derived porous framework tunes microenvironment of Ni_2P; fast bubble release and high conductivity.	[40]
Ni_2P@NiO/NiF	Ni_2P nanoparticles inserted in porous NiO nanosheet network on Ni foam	1.0 M KOH + urea	50 mA cm^{-2} at 1.31 V vs RHE; low onset UOR potential (≈1.31 V)	Synergistic Ni_2P/NiO heterostructure on Ni foam significantly enhances UOR response vs single-component Ni_2P or NiO.	[41]

Non-nickel transition-metal catalysts: While Ni dominates UOR research, there is growing interest in Ni-free catalysts for specific applications or to mitigate potential Ni leaching. Co-based oxides and hydroxides, Fe-rich perovskites, and Mn-containing mixed oxides have been investigated as UOR anodes, sometimes in combination with Ni but also as stand-alone catalysts. A recent article on Co_3O_4-based materials for UOR, for example, demonstrated that appropriate Nano structuring and doping can yield respectable UOR activity even without Ni, although achieving the same performance as optimized Ni-based systems remains challenging [9]. Metal-oxide UOR catalysts more broadly including doped spinels, perovskites, and MOF-derived oxides have been reviewed by Phule et al., who highlight strategies such as heteroatom doping, defect creation, and integration with carbon supports for improving charge transport and urea adsorption. These Ni-free or Low-Ni systems may be particularly attractive in environments where Ni dissolution is problematic or where regulation restricts Ni usage.

MXene- and carbon-based hybrid catalysts: Two-dimensional carbides and nitrides (MXenes) provide highly conductive, hydrophilic, and surface-tunable supports for UOR catalysts. Their large surface area, abundant surface terminations (-O, -OH, -F), and flexible stacking behavior facilitate intimate contact with active nanoparticles or (oxy)hydroxide layers.

Recent studies have demonstrated that decorating MXenes such as $Ti_3C_2T_x$, $Mo_2TiC_2T_x$ or $V_4C_3T_x$ with Ni-based LDHs, chalcogenides, or phosphides yields hybrid electrodes with enhanced UOR activity and stability [42-45]. The MXene support enhances electron transport, suppresses particle agglomeration, and may also engage in synergistic interactions with the active phase, modulating adsorption energies of urea and intermediates.

For instance, NiCr-LDH grown on V_4C_3 MXene showed markedly improved UOR activity compared with NiCr-LDH alone, attributed to enhanced conductivity and optimized OH^-/urea adsorption at the LDH-MXene interface [42]. Similarly, hierarchical $NiCo_2O_4$-$FeCo_2S_4$/$Mo_2TiC_2T_x$ composites leveraged triple junctions between spinel oxide, sulfide, and MXene to provide multiple synergistic active sites for both UOR and HER [43]. Carbon-based hybrids including N-doped carbon, carbon nanotubes, graphene, and 3D carbon foams play a broadly analogous role: they provide high conductivity, mechanical stability, and tunable surface chemistry for anchoring Ni-based nanoparticles or LDH Nano sheets. Numerous studies on Ni/NiO, Ni-B, Ni-C, and Ni-doped carbon dots supported on doped carbons or biomass-derived carbons have reported improved UOR kinetics and long-term stability relative to unsupported catalysts [9,34].

To highlight these MXene-supported UOR architectures in a more quantitative manner, representative MXene-based electro catalysts and their key performance figures are summarized in Table 3.

Table 3. Selected MXene-based electrodes for UOR / urea-assisted electrolysis.

Catalyst	MXene type & architecture	Electrolyte	UOR / cell metrics	Mechanistic role of MXene	Ref.
NiCr-LDH/V_4C_3 MXene	NiCr layered double hydroxide grown on V_4C_3 MXene	1.0 M KOH + 0.33 M urea	~1.36 V vs RHE @ 10 mA cm^{-2} (UOR, 3-electrode)	V_4C_3 provides 2D conductive scaffold; interfacial charge transfer to NiOOH boosted by Cr and MXene.	[42]
Ni-MOF/Ti_3C_2 MXene	Ni-MOF nanosheets anchored on Ti_3C_2 MXene	Alkaline KOH + urea (e.g. 1 M KOH + 0.33 M urea)	j ≈ 161 mA cm^{-2} at 0.8 V vs Hg/HgO in urea-containing electrolyte	Ti_3C_2 increases conductivity and exposure of MOF-derived Ni sites; porous Ni-MOF/Ti_3C_2 network aids mass transport.	[46]
Co_3O_4/Ti_3C_2 MXene	Co_3O_4 nanostructures on Ti_3C_2 MXene	1.0 M KOH ± urea	Shows much lower onset potential and higher current for UOR vs bare Ti_3C_2; exact E@10 mA not reported in summary	MXene under layer improves electron transport and mechanical integrity; Co_3O_4 is reconstructed to CoOOH during UOR.	[44]

Noble-metal and dual-atom catalysts: While noble metals such as Pt, Pd, Ru, and Rh generally exhibit good intrinsic activity toward urea oxidation, they suffer from high cost and susceptibility to poisoning by CO-like intermediates, limiting their large-scale deployment. Nonetheless, noble-metal-containing systems remain important as model catalysts and for certain niche applications (e.g., urea sensors or direct urea fuel cells).

Recent work on dual-atom and single-atom catalysts for example, Ni dual-atom sites on N-doped carbon has opened a complementary design space. Carefully engineered dual-atom catalysts can create well-defined active ensembles that exhibit high turnover frequencies for UOR while minimizing noble-metal loading or even avoiding noble metals entirely [9,21]. These systems also offer ideal platforms for theory–experiment correlation, since their active sites are structurally well defined.

Electrolyzer Configurations for Urea-Assisted Hydrogen Generation

Alkaline urea electrolysis cells: The most common configuration for UOR-assisted hydrogen production is an alkaline electrolyzer in which UOR occurs at the anode and HER at the cathode, typically using urea-containing KOH or NaOH solution. Ni foam, stainless steel, or other metallic substrates coated with UOR catalysts serve as the anode, while Ni, NiMo, or other HER catalysts serve as the cathode.

In such cells, full-cell voltages as low as 1.33-1.45 V have been reported at 10 mA cm^{-2}, with some advanced functional Ni-based catalysts enabling 100 mA cm^{-2} at ≈1.5-1.6 V in 1.0 m KOH +0.33-0.5 m urea [1,9]. These voltages are substantially lower than those required for conventional OER-based water electrolysis at the same current densities, directly translating into electrical energy savings per kilogram of hydrogen produced.

Functional electrodes that are active for both UOR and HER (e.g., Ni_2P, Ni-Mo-S, Ni-LDH-derived structures) simplify cell design and allow “mirror” configurations where identical electrodes serve as both anode and cathode, simply biased at different potentials [11,33].

Urine electrolysis and real wastewater treatment: Replacing synthetic urea solutions with real human or animal urine introduces additional complexity: the presence of chloride, phosphate, ammonium, creatinine, and various organic/ inorganic contaminants affects conductivity, catalysis, and corrosion. Nonetheless, direct urine electrolysis is highly attractive because it simultaneously addresses wastewater treatment and hydrogen production.

Recent work has demonstrated that appropriately designed Ni-based catalysts can sustain high current densities in fresh or hydrolyzed urine, although performance is typically inferior to that in pure urea solution due to fouling and competing side reactions [9]. Very recent strategies combine selective UOR catalysts with robust cathodes and optimized cell configurations to achieve efficient hydrogen generation while removing a substantial fraction of urea and organic contaminants from urine [47].

Photo electrochemical and solar-driven systems: Integrating UOR with photo electrochemical (PEC) devices or photovoltaic-biased electrolyzes offers a pathway toward solar-driven hydrogen production with reduced anode bias. For example, solar-assisted urea electrolysis configurations have used semiconductor photo anodes (e.g., $BiVO_4$, Fe_2O_3, or perovskite absorbers) coupled with UOR catalysts, exploiting the lower UOR potential to reduce the required photo voltage and enable overall urea splitting under sunlight [9, 48]. Carefully designed tandem or Z-scheme devices can further push the system toward unbiased operation, although this remains challenging in the presence of complex urea/urine chemistries.

Direct urea fuel cells (DUFCs): Although the main focus of this review is hydrogen production, it is worth noting that many of the same UOR catalysts can be used in direct urea fuel cells, where UOR is coupled with oxygen reduction (ORR) at the cathode to generate electrical power directly. DUFC developments have been reviewed in detail elsewhere [11]; lessons from DUFC anode design particularly regarding poisoning, crossover, and mass transport in urea-containing media are highly relevant to urea-assisted electrolyzes.

Mechanistic Insights from Operando Characterization and Theory

Surface reconstruction and active phases: A recurring theme across UOR research is that the as-synthesized catalyst is often a “pre-catalyst” that reconstructs under anodic UOR conditions. Ni phosphides, sulfides, selenides, and nitrides all tend to oxidize to NiOOH-like surface layers, while the underlying bulk retains its original phase and serves as an electron-conducting scaffold [1,11]. Operando XAS and X-ray photoelectron spectroscopy (XPS) have revealed dynamic changes in metal oxidation states and local coordination during UOR, with the formation of high-valent Ni^{3+}/Ni^{4+} and analogous Co/Fe species correlating strongly with activity. The degree and reversibility of this reconstruction depend on the initial phase, dopant elements, and applied potential; understanding and controlling it is crucial for designing durable catalysts.

Identification of intermediates and rate-determining steps: In situ Raman and FTIR spectroscopies have been used to detect surface-bound intermediates, including CNO^{-}*, carbamate, and carbonate, as well as changes in the vibrational bands associated with high-valent Ni-O or Co-O moieties. These measurements, coupled with kinetic isotope experiments and pH-dependent studies, support a mechanism where initial dehydrogenation of urea and NH_2 groups is followed by C-N bond scission and N-N coupling, with OH^- playing a central role in both dehydrogenation and reoxidation of the catalyst surface [9,10]. Differential electrochemical mass spectrometry and online gas chromatography have enabled quantification of N_2, CO_2 (or carbonate), and possible NO_x or N_2O by-products, providing insights into product selectivity. Recent highly selective UOR catalysts have been engineered to favor N_2 formation, minimizing NO_x production even at relatively high over potentials [9, 11].

Theoretical modeling and activity descriptors: Density functional theory (DFT) calculations play a key role in understanding UOR at the atomic level. Computational studies on NiOOH, NiFe-LDH, and various doped ox hydroxides have identified adsorption energies of key intermediates (e.g., $CO(NH_2)_2$*, CONH*, CO*, OH*) and free-energy diagrams for competing pathways, suggesting that an optimal balance between urea and OH^- adsorption is necessary to maximize activity and suppress poisoning by strongly bound carbonaceous species [21,22].

DFT has also been employed to evaluate how dopants (Fe, Co, Mn, Cr, high-entropy mixtures) and supports (MXenes, doped carbons) modulate metal oxygen covalence, band structure, and charge redistribution, providing microscopic explanations for experimentally observed activity trends. Emerging approaches, including machine-learning-assisted screening and micro kinetic modeling, may further accelerate the discovery of next-generation UOR catalysts.

Techno-Economic and Environmental Considerations

Energy savings and system efficiency: From a systems perspective, the primary advantage of replacing OER with UOR is the reduced anode potential and, consequently, lower overall cell voltage. For a given current density, the difference between a 1.8-2.0 V OER-based electrolyzer and a 1.4-1.6 V UOR-based electrolyzer corresponds to a 10-30% reduction in electrical energy consumption per kilogram of hydrogen, depending on operating conditions and ohmic losses [1,9]. These savings directly affect the levelized cost of hydrogen, especially in regions with expensive electricity.

Thermodynamic metrics such as "electricity economy" introduced by Protsenko quantify the theoretical minimum electrical work per mole of hydrogen for different urea oxidation pathways and confirm that UOR can outperform standard water electrolysis from an energy standpoint, provided that over potentials and internal resistances are kept under control [7].

Wastewater remediation and nitrogen management: Simultaneously, UOR-assisted systems can remove urea from wastewater or urine, offering environmental co-benefits. Integrating urea electro oxidation into decentralized sanitation or industrial effluent treatment could reduce nitrogen loading in receiving waters and mitigate eutrophication [9]. The key is to ensure that nitrogen is predominantly converted to benign N_2 rather than nitrate or nitrite, which themselves are regulated pollutants.

Life-cycle assessments and comparative exposure studies are beginning to examine the environmental implications of using urine-derived urea as a feedstock for hydrogen production and fertilizer recycling, highlighting both opportunities and challenges related to trace contaminants and public acceptance [8].

Practical challenges: scaling, fouling and durability: Despite rapid progress at the laboratory scale, several practical hurdles must be addressed before UOR-based hydrogen production can be deployed widely:

- ✓ **Carbonate scaling and precipitation:** CO_2 generated at the anode reacts with OH^- to form carbonate/bicarbonate, which can precipitate with metal cations and foul electrodes or separators, particularly at high current densities and elevated temperatures.
- ✓ **Impurities in real streams:** Chloride, organics, and other contaminants in real wastewater or urine can cause competing reactions (e.g., chlorine evolution), electrode corrosion, or membrane degradation.
- ✓ **Catalyst stability:** Long-term operation requires resistance to dissolution, phase segregation, and structural collapse. Some Ni-based pre-catalysts undergo irreversible leaching of dopants or halogens, gradually losing activity.
- ✓ **System integration:** Optimizing flow fields, gas management (bubble removal), and electrolyte composition for large-area cells remains non-trivial, particularly when treating variable-composition waste streams.

Beyond catalyst activity and cell design, the long-term durability of steel frames, piping, and auxiliary components in UOR-assisted systems will also depend on the availability of robust passive coatings. Recent advances in nanostructured passive coatings further demonstrate how small loadings of inorganic nanoparticles can drastically improve barrier performance and adhesion under extreme conditions [49]. Addressing these issues will require parallel advances in materials, reactor design, and operational strategies.

Outlook and Future Directions

Urea oxidation has evolved from a niche electrochemical curiosity into one of the most promising alternative anodic reactions for energy-efficient hydrogen production. In the past five years, a rich body of work has established robust mechanistic frameworks, identified high-performance Ni-based and non-Ni catalysts, and demonstrated energy-saving urea-assisted electrolyzes under realistic conditions. Comprehensive reviews on catalytic urea oxidation and UOR electro catalysts now provide invaluable maps of the field [9-11].

Looking ahead, several research directions appear particularly impactful:

- ✓ **Selectivity engineering:** Designing catalysts and operating regimes that favor N_2 and carbonate while suppressing nitrate/nitrite formation, even at high current densities and in real wastewater/urine matrices.
- ✓ **Robust pre-catalyst architectures:** Developing chalcogenide, phosphide, nitride, and high-entropy systems that reconstruct into stable, highly active ox hydroxide shells without rapid leaching or structural degradation.
- ✓ **MXene and 2D/3D hybrid platforms:** Exploiting MXenes and advanced carbon architectures as multifunctional supports in rationally designed heterostructures that couple excellent conductivity, high ECSA, and tunable interfacial chemistry.
- ✓ **Integrated techno-economic and life-cycle analysis:** Combining green-metrics thermodynamic analysis with realistic cost models, life-cycle assessment, and risk

analysis for urea/urine-based hydrogen plants.

- ✓ **Scale-up and system demos:** Moving from coin-cell-scale experiments to pilot-scale demonstrators that operate continuously on real waste streams, validating performance, durability, and maintenance requirements over thousands of hours.

If these challenges can be addressed, UOR-assisted hydrogen production could play a significant role in a future hydrogen economy by turning a ubiquitous nitrogen-containing waste into a valuable co-reactant, reducing both the energy cost of hydrogen and the environmental burden of urea-rich effluents.

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